STRAIN AGEING IN ULTRA-HIGH STRENGTH DRAWN PEARLITIC STEELS

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DECLARATION

I declare that this dissertation is my own unaided work, except where due acknowledgement of the assistance of others has been made. It is being submitted for the degree of Master of Science in Engineering in the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree or examination in any other University.

Nicholas Paul Widdrington Davies

10th day of APRIL 1987

ABSTRACT

Ultra-high strength steel wire, with a tensile strength greater than than 2000 MPa, has been used for a number of years in ropes used for hoisting applications by the mining industry. Recently, problems have arisen in certain ropes, due to a measured decrease in the rope breaking force after storage, and the phenomenon has been attributed to strain ageing. This study was initiated in an attempt to determine the operative mechanisms and to recommend possible practical solutions to the problem.

The approach used was to investigate the effect of various parameters on the rate of ageing in conventional wire, measured by the changes in the tensile proof stress and the wire ductility. These parameters included the carbon and nitrogen contents; the drawing strain; the drafting schedule; the drawing speed; and the pearlite interlamellar spacing. The effects of silicon and aluminium additions were also determined.

The reaction rate for ageing was found to be described in all cases by an Arrhenius-type rate equation, with an activation energy of about 117 $kJmol^{-1}$. The rate exponent, as defined by the Johnson-Mehl equation, was found to be about 0,25 to 0,30. The ageing kinetics were found to be increased by an high nitrogen content, a high level of strain, and a low pearlite interlamellar spacing. An increased drawing speed, and a low final reduction in the drafting schedule appeared to reduce the rate of deterioration of the wire ductility. High levels of silicon (above about 1%) were also found to be beneficial, although lower levels had the opposite effect.

The predominant ageing mechanism could not be isolated from these results, but they tended to confirm some work described in the literature, which ascribed the rate-controlling step to cementite dissolution to provide carbon atoms for traditional "Cottrell" ageing. The situation, however, appears to be more complex, due to the significant effect of both the drafting schedule (and hence probably the residual stress level) and the nitrogen content. An alternative mechanism which may account for these effects, whereby some refinement of the dislocation substructure occurs during ageing, is proposed.

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TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

Page No

LIST OF FIGURES

Figure CHAPTER TWO

Page No

Figure CHAPTER FOUR

LIST OF TABLES

LIST OF PLATES

CHAPTER ONE : INTRODUCTION

Ultra-high strength steels are generally considered to have tensile strengths in excess of 2000 MPa. Such values are readily attainable in cold-drawn plain carbon pearlitic steels by a combination of optimising the heat treatment prior to drawing and by drawing to sufficient reductions.

Such steel wire has found extensive application in ultra-high tensile (U.H.T.) ropes, used for hoisting by the mining industry. The requirements of the mining industry for increased shaft depths and increased payloads has motivated research into the manufacture of roping wire with ever higher tensile strengths (e.g. Benson, 1984), and such work has largely been successful.

However, one of the most important qualities of roping wire is its ductility, since the wires must be able to accommodate high cross-over forces from adjacent wires in the rope while in service. Although the ductility of high strength roping wires is usually satisfactory after manufacture, it has been observed that this property sometimes deteriorates with time, leading ultimately to a reduction of the breaking force of the rope. This phenomenon has been attributed to strain ageing.

Much work has been performed on the ageing phenomenon in drawn high carbon steel wires, and practical improvements to the "problem" of ageing have been made with some success over recent years; notably by paying attention to efficient cooling in the drawing process. With current strength levels of plain carbon U.H.T. ropes, it is probable that the problem has been overcome satisfactorily. However, the continuing requirements for ropes of higher strength and longer life have motivated research into establishing fundamental solutions to the ageing phenomenon.

The present study was proposed to contribute to this research, and the basic objectives of this project were thus as follows:

1. to determination the operative mechanisms of the ageing process; and
2. to develop a solution to the problem of ageing.

The ageing process occurs on an atomic scale in steel wire, and even with the most sophisticated techniques available, reliable information on the ageing mechanism in heavily drawn steel wire has not been achieved. Therefore, the process can only be inferred from other experiments.

The basic approach was to investigate those parameters which, on consideration, were thought to be significant to the ageing process. These were studied by drawing wire under controlled conditions, and testing its response to artificial ageing treatments by mechanical testing. The activation energy for ageing and a kinetic parameter were derived, which would, it was hoped, supply useful information regarding the operating mechanism(s).

The study was largely restricted to plain carbon eutectoid steels, since data on normal commercial materials were required before possible alloying additions could be recommended. However, the effect of small additions of silicon was also investigated.

Initially a traditional approach to the experimentation was taken, but it was eventually found that the number of influencing parameters was too large to investigate them in any detail. A statistical approach was then used to examine a group of parameters in a single factorial experimental design.

CHAPTER TWO : STRAIN AGEING IN ULTRA-HIGH STRENGTH DRAWN PEARLITIC STEELS - LITERATURE REVIEW

2.1 INTRODUCTION

The term strain ageing has been applied to a wide variety of effects in which some ageing phenomenon takes place both during or after plastic strain. The subject has been widely investigated for many years, and a great deal of information has been published.

The vast majority of work on strain ageing has been performed on low carbon steels (containing less than about 0,2% carbon), and subjected to low levels of strain (of the order of a few percent). Relatively little work has been performed on high carbon steels, especially where very high levels of plastic strain (e.g. true strains greater than 0,5) have been applied.

For the present study, the strain ageing characteristics of high carbon steels, cold drawn to true strains of greater than about 2,0 are of particular interest. The following literature review will, therefore, concentrate on the extent of current knowledge of strain ageing, especially as far as it pertains to drawn steel wire.

Subsequent sections will consider general aspects of strain ageing, the production methods and properties of carbon steel wire, the influence of strain ageing on these properties, and methods by which the strain ageing susceptibility of steel wire can be measured. Finally, means by which strain ageing can be controlled in finished wires will be discussed.

2.2 THE STRAIN AGEING OF STEELS - AN OVERVIEW

2.2.1 The Ageing Mechanism

The general features of strain ageing are well documented, and have been described in a number of reviews (e.g. Kenyon and Burns, 1940; Low and Gensamer, 1944; Paxton, 1959; and Baird, 1963). Commonly, strain ageing is revealed by the return of the yield point in tensile stress/strain curves after plastic straining and subsequent ageing, as shown by Figure 2.1. All the mechanical properties, however, may be affected, including tensile strength, yield stress, fracture toughness, impact transition temperature, and ductility. Strain ageing also influences physical properties such as damping capacity and electrical resistivity.

It has been shown (Low and Gensamer, 1944) that if carbon and nitrogen are removed from steel, then the initial discontinuous yielding characteristic of annealed mild steels and strain ageing are eliminated. From this and other work Cottrell and Bilby (1949) developed the generally accepted theory of strain ageing in steels.

According to this theory, the yield point in steel is caused by the segregation of carbon and/or nitrogen atoms to the many dislocations present in the iron lattice. These interstitial solute atoms tend to relieve the stresses around the dislocations. From energy considerations, it is possible to show that a higher stress is required to cause plastic flow (i.e. the movement of dislocations away from their surrounding atmospheres of carbon and nitrogen atoms). Once the dislocations have been torn away, the force required to maintain dislocation movement is reduced, and the stress falls to the lower yield point.

Strain ageing can also be explained using the same model. During ageing, carbon and/or nitrogen atoms migrate through the strained iron lattice to the new dislocation positions, locking them in place as before. The extent of strain ageing with time after straining will be dependent on the rate of diffusion of the interstitial solute atoms, on the number of interstitial atoms in solution, and on the temperature.

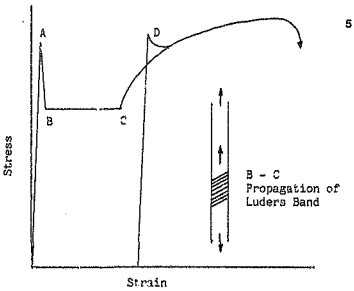


Figure 2.1 : Schematic of Yield Phenomenon as Exhibited by the Tensile Test. After Honeycombe (1981).

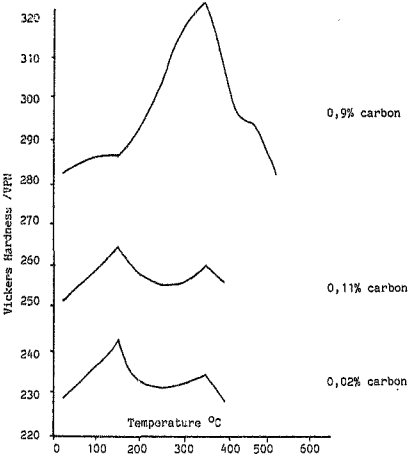


Figure 2.2 : The Effect of Carbon Content on the Hardness of 55% Cold-Rolled Steel after a low temperature heat treatment. After Varo (1964).

Honeycombe (1981) has quoted an alternative theory to that described above, which was attributed to Gilman and Johnson. This theory proposes that once the carbon atom atmospheres have pinned the dislocations, these remain locked and the return in the yield point arises from the sudden generation and movement of newly-formed dislocations. Thus it would be expected that dynamic strain ageing (in which carbon atom atmospheres form continuously on newly generated dislocations) would require a higher dislocation density to complete the deformation.

Electron microscopy studies have demonstrated that, in steels deformed at 200°C, dislocation densities are an order of magnitude greater than in specimens similarly deformed at room temperature. Gilman and Johnson's model also accounts for the increased work hardening rates obtained during warm straining, when compared to straining at room temperature.

A detailed survey by Nishino and Takahashi (1962) of the changes occurring during the low temperature heat treatment of various steels in the range 0,1% to 0,9% carbon used hardness measurements to follow the progress of ageing. The results showed a double peak in the hardness vs temperature response curve, which was most clearly evident in the lower carbon steels. The primary peak, in particular, became more pronounced as the carbon content decreased (Figure 2.2) (see also Varo, 1964).

It can be argued that, during deformation, active dislocations will pile-up at obstacles such as grain boundaries or cementite particles, giving rise to a back stress. At elevated temperature these dislocations will tend to move under the influence of this stress, but in so doing, encounter other groups of dislocations. The interaction between these groups of dislocations renders them sessile. The annihilation of active dislocations can account for the observed hardening effect.

2.2.2 The Effect of Strain Ageing on Mechanical Properties

The effects of strain ageing on the mechanical properties of steels can be considered on the basis of a two-stage ageing process proposed by Hundy (1956), and Wilson and Russell (1960).

The first stage of ageing corresponds to interstitial solute atom migration to dislocation sites to form "Cottrell atmospheres", as described previously. In terms of tensile properties, this should merely increase the yield stress, while tensile strength and ductility (as measured by elongation to fracture) are unaffected. With very low solute contents only the first stage is possible.

The second stage of ageing corresponds to the formation of precipitates on the dislocations. This increases both the yield stress and the ultimate tensile stress, while ductility is adversely affected. In addition, the work hardening rate increases. Overageing, due to coarsening of the precipitates may eventually occur, leading to a small reversal of the above property changes.

Other property changes which occur are as follows. Hardness, as expected, tends to follow the same trends as tensile strength.

There is some evidence (Levy and Kanitkar, 1961; and Lippitt and Horne, 1957) to suggest that the fatigue endurance limit, especially in low stress-high cycle fatigue, is due to strain ageing effects, since no comparable limit is obtained in iron from which all carbon and nitrogen have been removed.

The impact transition temperature is particularly sensitive to first stage ageing, although this parameter does rise continuously throughout the ageing process (von Kockritz, 1930), while the upper shelf energy decreases.

Electrical resistivity, damping capacity and residual magnetism all decrease during strain ageing (Wilson and Russell, 1960; Cottrell and Churchman, 1949; Dahle and Lucke, 1954; and Koster, 1930).

The rate of ageing is temperature dependent, but the maximum property changes are not apparently greatly affected by the temperature of ageing. Overageing, however, is only pronounced at temperatures above about 250°C (Baird, 1963).

2.2.3 The Kinetics of Ageing

The availability of carbon and nitrogen in ferrite for dislocation locking derives from the inevitable supersaturation of these elements, even after slow cooling. Dislocations are known to be very effective nucleation sites for precipitation and Honeycombe (1981) quotes the value for the binding energy of a carbon atom to a dislocation to be 0,5 eV.

Cottrell and Bilby (1949) analysed the kinetics of solute atmosphere formation, while making the simplifying assumption that the only important effect would be the "drift force" pulling solute atoms in towards the dislocation core.

This drift force arises from the decrease in the dilatational energy of a solute atom in sites near to the tension side of an edge dislocation. The following expression was obtained for the number of solute atoms, N(t), that would reach a unit length of a dislocation in time t:

$$N(t) = n_o.3\left(\frac{\pi}{2}\right)^{1/3}.\left(\frac{A.D.t}{k.T}\right)^{2/3} \quad \ldots\ldots\ldots\ldots\ldots (2.1)$$

where : n_o is the average concentration of solute atoms (per unit volume);

D is the diffusion coefficient (m^2s^{-1});

A is a parameter defining the magnitude of the interaction between the solute atom and the dislocations (A is approximately $3x10^{-29}$ Nm for carbon and nitrogen in alpha-iron);

k is Planck's constant, $1,38x10^{-23}$ JK^{-1};

and T is the temperature (K).

Cottrell and Bilby estimated that a dislocation would become saturated with carbon when 1-2 atoms of carbon per atom plane of dislocation had segregated to it. However, Equation 2.1 will only hold for atmosphere densities considerably lower than this saturation level. As saturation is approached, back diffusion from the core region will tend to counterbalance inward flow due to the drift force, and, in addition, relief of the dislocation stresses by segregated atoms may reduce the drift force.

Harper's equation (1951) allows for the fall in solute concentration in the matrix surrounding dislocations as strain ageing proceeds:

$$W = 1 - \exp\left(-3r\left(\frac{\pi}{2}\right)^{1/3}\left(\frac{A.D.t}{k.T}\right)^{2/3}\right) \quad \ldots\ldots\ldots\ldots(2.2)$$

where W = fraction of atoms collecting on dislocations in time t and r = dislocation density.

Hundy (1954) included data from Wert (1950a, 1950b) on the diffusivities of carbon and nitrogen in alpha-iron in Harper's equation, and derived an equation relating the time of strain ageing at room temperature to that at a higher temperature:

$$\log\left(\frac{t_r}{t}\right) = k\left(\frac{1}{T_r} - \frac{1}{T}\right) - \log\left(\frac{T}{T_r}\right) \quad \ldots\ldots\ldots\ldots(2.3)$$

where the value of k depends on whether carbon or nitrogen causes strain ageing. Thus k = 4400 for carbon and 4000 for nitrogen. Here, t is the strain ageing time at temperature T, while the subscript r denotes room temperature values.

Hundy's equation does not, however, fully characterise the ageing process, for three main reasons. Firstly, the equation does not account for the plateau in the ageing curve (Figure 2.3) which is frequently observed, especially when the change in yield point is used to detect ageing (Tardif and Ball, 1956). Secondly, the equation does not allow for the change in solubility of the interstitial atoms with temperature. Finally the equation cannot explain the strain ageing of commercial materials in which nitrogen is often combined with a nitride-former such as aluminium.

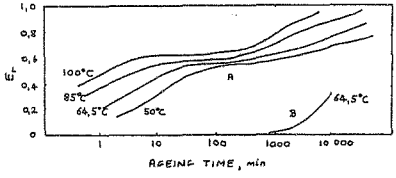


Figure 2.3 : Ageing Curves for 0,035% Carbon, Deep Drawing Rimmed Steel, Prestrained 5% in Tension.
(a) direction of straining the same before and after ageing;
(b) direction of straining after ageing transverse to pre-strain direction.
E_L= Strain aged lower yield extension - initial lower yield extension. After Tardif and Ball (1956).

The small difference in the value of k for carbon and nitrogen in Equation 2.3 reflects the small difference in their diffusivities in b.c.c. iron. The diffusion coefficients for carbon (D_C) and nitrogen (D_N) in ferrite (due to Wert, 1949; and Nabarro, 1948; respectively) are:

$$D_C = 2{,}0 \times 10^{-6}.\exp\left(\frac{-84\ 100}{R.T}\right)\ m^2s^{-1} \quad \ldots\ldots\ldots(2.4)$$

$$D_N = 6{,}6 \times 10^{-7}.\exp\left(\frac{-77\ 800}{R.T}\right)\ m^2s^{-1} \quad \ldots\ldots\ldots(2.5)$$

where R is the gas constant, 8,314 $Jmol^{-1}K^{-1}$;
and T is the absolute temperature (K).

Authors (e.g. Harper, 1951; Wert, 1949; and Polder, 1945) who have studied the strain precipitation of carbon in very low (0,01%) carbon irons using, for instance, internal friction techniques, have derived activation energies for the process which are, generally, close to 84 $kJmol^{-1}$. This agrees with the activation energy for diffusion of carbon in alpha-iron (Wert, 1949; Stanley, 1949). Similarly, the activation energy for strain ageing due to nitrogen has been determined to be 72 $kJmol^{-1}$ (Harper, 1951), a figure also in reasonable agreement with that for diffusion of nitrogen in ferrite (Leslie, 1959).

The activation energy for the diffusion of carbon in ferrite does, however, increase with the carbon content of the steel, according to Gul and Babich (1980). For example, the activation energy for dynamic strain ageing due to carbon increases by 50% (from 136 to 205 $kJmol^{-1}$) on increasing the carbon content from 0,15% to 0,80%.

The kinetics of strain ageing are usually followed by measuring the change in the concentration of interstitial solute remaining in the ferrite lattice, the assumption being made that all solute leaving the lattice segregates to dislocations. If the dislocation density is known or can be deduced, the atmosphere density at any stage of the process can be calculated.

The concentration of carbon and nitrogen in solution in the ferrite has generally been measured either by measuring the height of the corresponding Snoek internal friction peak (Snoek, 1941), or by measuring electrical resistivity (Dijkstra, 1947).

The former method is noted to provide the more accurate results, since it distinguishes more clearly between interstitial solute atoms in normal lattice positions, and those segregated to dislocations.

Opinions vary as to the number of solute (carbon) atoms required to sufficiently lock dislocations, but it has been shown that, in quenched mild steels, between 10 and 100 solute atoms per atom plane were attributed to each dislocation during strain ageing (e.g. Thomas and Leak, 1955; and Dahle and Lucke, 1954).

It would appear, therefore, that sufficient atoms can be accumulated on dislocations to form small precipitates. It was proposed that, on subsequent cold work, the dislocations are freed from their atmospheres while the precipitates are left behind. Although still too small to be resolved using electron microscopy, they can exercise a considerable hardening effect on the steel, thereby raising the general level of the stress-strain curve.

Wilson (1957) has pointed out that carbide precipitation in steel tends to reverse a general rule that cold work increases the rate of precipitation from a supersaturated solid solution. This observation is based on the work of Andrew and Trent (1938) and others, who showed that cold working of a quenched low carbon steel or iron-nitrogen alloy can prevent the formation of visible precipitates during subsequent ageing at 200 to 250°C.

Wilson himself showed that the precipitation of ε-carbide in the first stage of tempering in medium and high carbon martensites can be suppressed by cold work. In addition, he showed that carbides precipitated during a tempering treatment tended to redissolve during subsequent deformation. Wilson suggested that this phenomenon was due to a strong interaction between carbon atoms and lattice defects in iron.

This hypothesis has been substantiated by Cottrell (1953) and others (from Baird, 1963) who have shown that when a carbon or nitrogen atom is situated near the centre of an edge dislocation in iron, the elastic interaction will give a binding energy which is greater than that binding those atoms in iron carbide or nitride precipitates, even when a relatively high density of solute atoms on dislocations exists (i.e. above 10 atoms per atom plane).

Gul and Babich (1980) have analysed the mechanisms of strain ageing in medium and high carbon steels from a thermodynamic viewpoint. They proposed that higher carbon steels contain lower proportions of solute (carbon and nitrogen) in solution in the ferrite. For low carbon steels, the total (C + N) content in ferrite ranges between 10^{-4} and 10^{-3} weight percent, while for medium and high carbon steel this value is in the range 10^{-7} to 10^{-3} weight percent (Mogutnov et al, 1972). This phenomenon was explained in terms of the larger volume fraction of cementite present, and of the shorter ferrite diffusion paths.

They and other workers (i.e. Sarrak et al, 1969; Gul, 1973; and Yamada, 1974) maintain that the carbon and nitrogen in solution in the ferrite of higher carbon steels would not be sufficient to produce any strain ageing. It would thus be reasonable to assume partial dissolution of cementite to occur to provide carbon atoms for dislocation pinning.

However, this cannot be explained solely in terms of an enthalpy advantage, and it can be shown that the free energy can be further reduced by an increase in the configurational entropy, S_{CONF}. Thus:

$$S_{CONF} = S_{CONF}.(V_m - V_{dp}) > 0 \quad \ldots\ldots\ldots\ldots\ldots\ldots (2.6)$$

where V_m = volume of ferrite matrix containing dislocations of a certain density;

and V_{dp} = volume of the decomposing phase.

At room temperature, it follows that the locking of dislocations during strain ageing must be weak since, thermodynamically, only the first stage of ageing (i.e. Cottrell atmosphere formation) is possible. In addition, since the activation energy for strain ageing tends to increase with the carbon content of the steel, the additional energy required may be accounted for by that additional energy required to rupture the carbon-cementite bond.

The implications of a higher activation energy are that natural strain ageing (i.e. at room temperature) should occur at a comparatively low rate, but that the ageing rate should increase more rapidly with temperature. For example, calculations based on the Cottrell-Bilby-Harper equation (2.2) show that an increase of 33% in the activation energy of the strain ageing process over the activation energy for carbon diffusion in alpha-iron, lowers the ageing rate by 1000 times at 27°C, but only 10 times at 400°C. It might therefore be reasonable to expect essentially no natural strain ageing in high carbon steels, and this has apparently been confirmed experimentally (Gul and Babich, 1980). However, the general validity of this observation, especially at high prestrains, is questionable, based on the observed deterioration in properties of hard drawn pearlitic steel wire with time (see Section 2.4).

2.2.4 The Behaviour of Carbon and Nitrogen in Cold-Worked Alpha Iron

Nitrogen and carbon differ slightly in their behaviour during strain ageing in low carbon steels. The behaviour of nitrogen can be summarised as follows (from Baird, 1963):

* By virtue of its reasonably high solubility, its low diffusion coefficient, and the severity with which it locks dislocations, nitrogen is expected to be an effective solute in producing strain ageing. This has been confirmed by experimental evidence drawn from iron-nitrogen alloys.

* Iron-nitrogen alloys show appreciable ageing below 100°C. This is due partly to the fact that equilibrium solubilities at low temperatures are not often approached, and partly because dissolution of iron nitride particles can contribute to strain ageing.

* The amount of ageing in an iron-nitrogen alloy is not very sensitive to prior heat treatment.

* The amount of nitrogen required to produce strain ageing is uncertain, but values of as low as 0,0001% appear to be sufficient to produce detectable ageing.

In contrast, the behaviour of carbon can be summarised in the following manner:

* In slowly cooled specimens, the carbon held in solution is likely to be lower than nitrogen, so that the ageing due to carbon is likely to be less significant than that due to nitrogen.

* If held in supersaturated solid solution, carbon should be able to produce ageing below 100°C, since carbon can diffuse and lock dislocation almost as rapidly as nitrogen. This has been demonstrated experimentally.

* As little as 0,0001% carbon in solid solution can give appreciable strain ageing at 100°C, and the amount of ageing increases as the level of dissolved carbon is raised to about 0,002%.

* Large amounts of carbon probably decrease strain ageing, by decreasing the proportion of free ferrite present.

The above conclusions were derived by Baird from the work of many authors. However, it should be noted that the materials used were largely restricted to very low carbon and nitrogen iron alloys.

2.2.5 The Effect of Alloying Elements on the Strain Ageing of Carbon Steels

There are two classes of alloying elements which may be distinguished in connection with strain ageing. The first class consists of solutes which may pin dislocations and hence may cause strain ageing. These elements must have high diffusivities in alpha-iron, and are thus limited to those elements which go into interstitial solid solution (e.g. carbon, nitrogen, oxygen, boron, and hydrogen). It has been demonstrated (by Low and Gensamer, 1944; and Fast, 1950) that carbon and nitrogen are the only causes of strain ageing in commercially important steels.

The second class of elements includes those which may influence the strain ageing process by altering the solubility or mobility of those elements in the first class. These may be divided into four main groups, as follows:

1. elements which interact only weakly with carbon or nitrogen (e.g. copper, nickel, manganese, phosphorus);
2. nitride-formers (e.g. aluminium, silicon, boron);
3. carbide-formers (e.g. molybdenum);
4. nitride- and carbide-formers (e.g. chromium, vanadium, niobium, titanium).

2.2.5.1 Copper, Nickel, Manganese, Phosphorus

Of these elements, copper and nickel are considered not to interact appreciably with carbon or nitrogen, whereas phosphorus may be a weak nitride-former, and manganese a weak carbide- and nitride-former. In high carbon steels, manganese enters the carbide phase as $(Fe,Mn)_3C$.

Copper and nickel have been shown by Edwards et al (1940) to slightly increase the strain ageing of a 0,025% carbon iron at 250°C, indicating that these elements may increase the solubility of carbon or nitrogen in iron.

Manganese and phosphorus, which tend to attract nitrogen atoms when in solid solution. probably slow down low temperature ageing (Erdmann-Jesnitzer et al, 1960), but the evidence is limited. Contradictory evidence of the effect of manganese on the ageing response of steel is available. Fast (1960) found no effect of up to 0,5% manganese on low carbon steels up to 250°C, but Murphy (1968), while investigating 0,6% carbon wire, found that manganese reduced the ageing response.

2.2.5.2 Nitride-Forming Elements

Aluminium is used extensively in the steelmaking industry to tie up oxygen to produce killed or semi-killed steels. It also interacts strongly with nitrogen to form aluminium nitride, AlN. The effect of aluminium alone in reducing strain ageing due to nitrogen is not great; low temperature ageing can be reduced by aluminium alone, but not eliminated (Leslie and Rickett, 1953).

Aluminium additions appear to most effective when low austenitising temperatures are used to reduce the tendency for aluminium nitride to dissociate. However, in combination with silicon, aluminium appears to be far more effective. The precipitates formed are isomorphous (Arrowsmith, 1963), and it appears that silicon nitride precipitates on aluminium nitride particles during cooling. Both aluminium and silicon form stable carbides but each has a strong affinity for iron which tends to counteract the formation of these carbides.

Langenscheid (1979) investigated the precipitation of aluminium nitride during the cooling of steel. The solubility of the nitride in austenite is given by:

$$\log K = \frac{-7750}{T} + 1.8 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (2.7)$$

where K=[Al].[N] and T is the absolute temperature. Thus the solubility decreases as the temperature falls. The precipitation of aluminium nitride is a diffusion-controlled process, and thus the temperature of precipitation is important. The danger arises that some of the nitrogen may not be fixed by aluminium, and hence would be available for ageing.

Langenscheid has shown that precipitation occurs rapidly at about 750°C but at the normal transformformation temperatures of around 600°C for wire rod, the reaction is very slow.

It should be noted that of the common alloying elements, silicon is the only one which increases the activity coefficients of carbon and nitrogen in ferrite, and will, therefore, increase the kinetics of ageing. However, it is thought that this effect may be cancelled by a reduction in the diffusion rate of the interstitial elements in the ferrite, by silicon (Smith, 1985).

Boron has been shown by Morgan and Shyne (1957a, 1957b) to significantly reduce strain ageing in steels, through the formation of boron nitride. The best results have been obtained with additions of about 0,004% boron. One advantage of using boron as a stabilising element is that, being itself an interstitial solute, it can diffuse quickly at low temperatures and so precipitate nitrogen under most cooling conditions.

2.2.5.3 Carbide-Forming Elements

Molybdenum is the only important element in this group. Its effectiveness, however, as an ageing "inhibitor" is questionable, since nitrogen will remain in solution to promote ageing. For instance, in one experiment by Leslie (1959) an unsuccessful attempt was made to produce a non-ageing rimmed steel by the addition of molybdenum.

2.2.5.4 Nitride- and Carbide-Forming Elements

The majority of important alloying elements fall into this group. In general, for metals such as chromium, vanadium, niobium and titanium, the nitrides tend to be more stable than the carbides. This implies that the nitrogen is often largely combined with these elements, while carbon, due mainly to its presence in larger quantities, is incompletely combined.

Potentially, all the elements which form both carbides and nitrides can eliminate high temperature strain ageing if present in sufficient quantities. Low temperature ageing tends to be more easily reduced, presumeably because of the greater importance of nitrogen below about 100°C.

The effectiveness of the alloying elements in preventing strain ageing increases according to their affinity for carbon and nitrogen, in the order chromium, vanadium, niobium and titanium. The elements molybdenum, manganese, chromium and vanadium have been shown by internal friction techniques to interact with nitrogen in ferrite (Dijkstra and Sladek, 1953), but not in any comparable way with carbon (Wert, 1952).

The effect of such an interaction could be due to slow precipitation during cooling, thus implying an increased supersaturation after cooling. Alternatively, the migration of the interstitial element during strain ageing may be hindered. These alternatives may occur together, in which case the effects may cancel each other out.

Rashid (1975), has investigated the strain ageing kinetics of vanadium- and titanium-strengthened high strength low alloy (HSLA) steels. He proposed that in these steels, strain ageing may occur in two stages:

1. Snoek rearrangement;
2. Cottrell atmosphere formation.

The process of Snoek rearrangement was proposed by Nabarro (1948), and various investigators have attributed the effect to dislocation pinning by local atomic rearrangement, or short range ordering of interstitials in dislocation strain fields. Due to the proximity of the interstitial atoms to the dislocations involved in Snoek rearrangement, when compared with those interstitials diffusing through the lattice to form Cottrell atmospheres, the former mechanism occurs much faster than the latter.

Rashid derived an activation energy for Cottrell atmosphere formation only, since the effect of Snoek rearrangement on mechanical properties is very small. Using only small amounts of prestrain (2,6%) to ensure the maximum response to ageing, and ageing temperatures between 205°C and room temperature, the activation energy for Cottrell atmosphere formation in both vanadium and titanium HSLA steels was found to be close to 144 $kJmol^{-1}$. This high value for the activation energy was attributed to the results of interactions between interstitial solutes and the elastic strain fields which surround coherent precipitates in the HSLA steels (Rashid, 1976).

2.2.6 Summary

Strain ageing is generally considered to be caused by the migration of interstitial atoms (particularly carbon and nitrogen) to dislocation sites, thereby effectively pinning them. The effect of this phenomenon is to increase the tensile properties (notably the yield point) and to reduce ductility.

Strain ageing in mild steel is a diffusion-dependent process, and as such, follows an Arrhenius-type rate law. The ageing rate is thus markedly influenced by temperature. The rate also depends on the migrating element, on the concentration of this element, and on the amount of prior deformation applied.

The normal commercial method for restricting strain ageing is to tie up excess interstitial solute by using nitride- and/or carbide-formers as alloying elements. Aluminium is frequently added to combine with free nitrogen in steels, while strong carbide- and nitride-formers, such as vanadium or titanium, are probably the most effective additions.

2.3 THE PRODUCTION OF CARBON STEEL WIRE

2.3.1 Introduction

Cold drawn wire is a commercially important material, with a unique combination of mechanical properties. Tensile strengths far in excess of 1600 MPa are readily achieved, even with relatively inexpensive plain carbon steels, while adequate ductility is maintained. Such a combination of strength and ductility is superior to that obtainable by means of heat treatment or by the addition of alloying elements alone.

The mechanical properties of cold drawn wire are determined by the chemical composition of the steel, the heat treatment applied prior to drawing, the extent of cold working applied, the drawing conditions and the nature of any heat treatment applied after drawing. A number of interacting factors may therefore affect the quality of the finished product, and this section attempts to summarise some of these factors.

2.3.2 The Patenting Process

Patenting is a term used in the wire industry to describe the heat treatment applied to the as-rolled rod, or partially drawn wire, prior to drawing. Generally, with eutectoid steels, the object of patenting is to obtain a fine uniform pearlitic structure across the rod section, although, in some instances pearlite and bainite mixtures are preferred. Alternative methods of obtaining the required microstructure, such as controlled cooling or step-patenting have been recently investigated, with excellent results. The traditional patenting method, though, remains the most important commercial method in South Africa.

Typically, pearlite interlamellar spacings of the order of 70 to 90 nm are required. Such a fine (and uniform) microstructure is important in wire drawing, since fine cementite lamellae can bend during deformation (Duckfield, 1971a; and Nishimura et al, 1980), while thicker lamellae will tend to fracture, and may thereby initiate failure. In addition, thin carbide plates restrict void formation at the tail of the plate, since the ferrite can more easily accomodate the required deformation (Pickering, 1965).

For a given reduction in area during drawing (using similar drawing conditions) the tensile strength of the finished wire depends largely on the as-patented strength. A fine pearlitic structure ensures a high strength prior to drawing, which will reduce the deformation required to achieve a given tensile grade. The work-hardening rate also increases with pearlites of smaller interlamellar spacing (Godfrey, 1963).

The patenting process consists of three stages; heating to the austenitising temperature, soaking at temperature, and, finally, cooling to the tranformation temperature. The process method may be either continuous or batch.

The heating cycle is maintained to as short a time as possible, although rapid rates of heating are not normal in the patenting process (Payne and Smith, 1968).

During soaking, time is normally allowed for some degree of austenite homogenisation. This eliminates carbon concentration gradients which can result in an inferior microstructure after transformation. However, for economic reasons the soaking time is kept short. A fine grain size is ideal for the final wire properties, but manufacturers often favour coarse grain sizes since lower drawing loads are required, and an increased hardenability is obtained (Duckfield, 1971a).

The third stage, cooling to the transformation temperature, is *the most critical, since it is the metallurgical reactions which* occur during cooling which greatly influence the final properties of drawn wire. Cooling is generally performed in lead maintained at about 540°C for plain carbon rod, although the transformation temperature range may be as high as 650°C to 670°C, depending on the rod size. The variations in cooling rate across the rod section, especially for thicker rods, can give rise to coarse pearlite, which cannot be drawn to very high strains, and may also lead to inferior mechanical properties.

Once transformation has started, the heat of transformation, together with an adequate lead bath, is then sufficient to ensure subsequent uniform transformation temperature conditions.

Franklin et al (1980) studied the effects of composition and patenting conditions on the patenting response of 0,74-0,80% carbon steels. These authors state that patenting is essentially a compromise heat treatment, since the attainment of a fine, uniform patented microstructure requires the use of larger austenite grain sizes, and high alloying contents to improve hardenability. Higher austenitising temperatures and increased alloying contents, however, tend to lead to an increased transformation time, and, apparently, to an increase in the pearlite interlamellar spacing, with a consequent decrease in strength.

It was found that the highest as-patented strength levels, regardless of composition, were associated with lower austenitising temperatures (850 to 950°C) and lower quench bath temperatures (450 to 500°C).

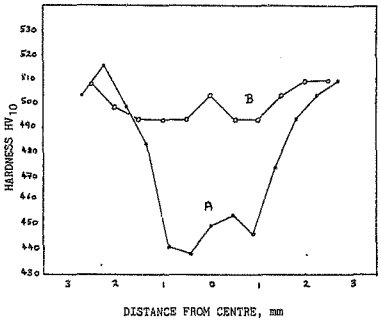
Lower quench bath temperatures had a tendency to form bainite and pearlite/bainite mixtures but these microstructures apparently presented no problems, even on drawing to 91% reduction in area. Fully pearlitic microstructures were, however, noted to exhibit higher work hardening rates, particularly for reductions greater than about 80%.

2.3.3 Wiredrawing

Wire drawing is essentially a work hardening process, although many other factors may operate during production processing. The most notable of these are ageing effects. Some of these factors will be described separately below.

2.3.3.1 The Mechanics of Wire Drawing

The true mode of deformation involved in wire drawing is not yet clear (Smith et al, 1973), although a substantial amount of work has been performed on evaluating the drawing stresses required (see e.g. Sachs, 1927; Johnson and Sowerby, 1969; Atkins and Caddell, 1968; and Caddell and Atkins, 1968). It is generally agreed (Caddell and Atkins, 1971) that the deformation patterns imposed on metals as they plastically flow through a normal conical die, are such as to produce marked strain inhomogeneities in the deformed material (Figure 2.4).



A : Drawn with 14° Angle Dies Throughout;
B : Drawn with 14° Angle in First and Second Dies, and 8° Angle in Last Five Dies.

Figure 2.4 : Hardness Traverses of 5,72mm 1800MPa Spring Wire After Timiney (1985).

The strain inhomogeneities are due to redundant deformation occurring in the wire during drawing, and are associated with the generation of hydrostatic tension along the wire axis. The redundant deformation can be considered to be that required to change the shape of the wire during deformation, but which is not reflected in the final wire size. The amount of redundant work performed on the wire during drawing is dependent on the approach angle in the die and on the reduction in area and, to some smaller extent, on the initial wire diameter, and on the extent of prior deformation (Figure 2.5). Thus the redundant work is at a maximum when large diameter wires of annealed metals are drawn using small reductions and large die angles (Timiney, 1985).

Redundant deformation has a number of effects which are detrimental to wire properties. Central bursting in the wire during drawing is due to high hydrostatic tensions along the wire axis. Drawing stresses are increased, and the total strain applied to the wire is increased. Finally, variations in properties across the wire cross section occur and the magnitude of residual stresses is increased.

Timiney (1985) has quoted some results from a finite element analysis on residual hoop stresses in high strength wire (Figure 2.6). At the surface, residual hoop stresses as high as 1200 MPa have been predicted, which may easily induce cracking in both the radial and the longitudinal directions. The residual stress pattern also tends to raise the apparent tensile strength of the wire.

It is apparent from the above that the use of a narrow die angle is indicated for the drawing of high strength steel wire. However, it must be recognised that the use of a lower die angle tends to increase the amount of frictional work required. This will lead to an increased drawing force, higher wire/die interface temperatures and possible concomitant lubrication breakdown problems. These factors should therefore be considered when selecting die geometries for the production of high strength wire.

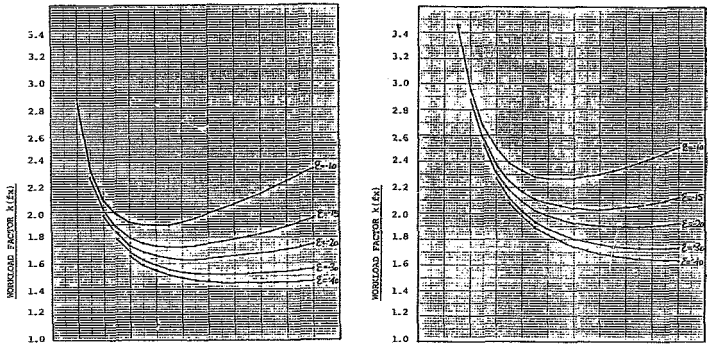
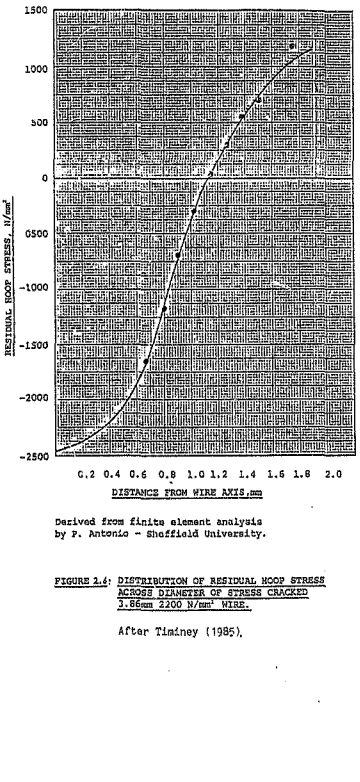


FIGURE 2.5: WORKLOAD FACTOR VERSUS DIE ANGLE.

After Timiney (1985).



Derived from finite element analysis
by P. Antonio - Sheffield University.

FIGURE 1.4: DISTRIBUTION OF RESIDUAL HOOP STRESS ACROSS DIAMETER OF STRESS CRACKED 3.86mm 2200 N/mm² WIRE.

After Timiney (1985).

Godecki (1972) investigated the effect of the die angle and drafting schedule on the properties of steel wire, and in particular on the torsional ductility. He proposed that poor ductility was associated with fragmentation of the cementite lamellae in pearlite, and that heavy drafting results in the fragmentation process occurring sooner, and more extensively than when lighter drafting is employed.

In addition, if a high reduction in area is liable to cause premature fragmentation of the cementite, then it might be expected that increasing the die entry angle will have a similar effect. A large entry angle may create bending stresses in the wire, in excess of the bending strength of the cementite lamellae giving rise to fracture.

On the basis of these theories, a so called "hump-backed" draft has been recommended to ensure the optimum tensile strength and ductility in the finished product. This employs light initial drafting to orientate the cementite lamellae, followed by heavy drafts decreasing again to light drafts at high strains.

2.3.3.2 Work Hardening Characteristics

The work hardening processes involved in wire-drawing have been investigated in some detail, and Figure 2.7 (after Duckfield, 1971a) shows a typical work hardening curve relating tensile strength to true strain. It can be seen that the work hardening process appears to occur in two stages. At a true strain of about 2,0 (corresponding to about 87% reduction in area) a change of slope occurs in the work hardening curve.

This behaviour is commonly quoted (Duckfield, 1971a; Stephenson et al, 1983) but other observers have discounted the distinct two-stage work hardening rate (Whyte, 1985), attributing the rise in the apparent work hardening rate rather to strain ageing occurring during drawing at high strains.

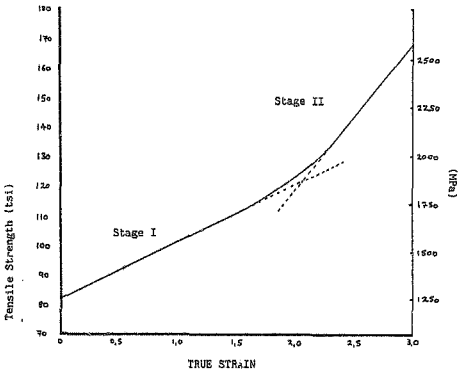


Figure 2.7 : Work Hardening of 0,8% Carbon Steel During Drawing After Duckfield (1971).

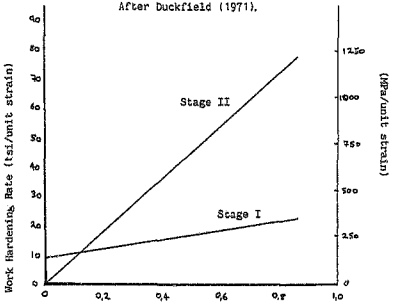


Figure 2.8 : Work Hardening Rates During the Two Stages of Deformation for Steels of Different Carbon Contents After Duckfield (1971).

The work hardening rate depends to a large extent on the carbon content of the wire (Shipley, 1963; Heape, 1983), the effect becoming more marked in second stage hardening (Figure 2.8). In addition, the work hardening rate is enhanced by small pearlite interlamellar spacings. These effects have been attributed by Godfrey (1963), in part, to the fact that dislocations may originate at the ferrite/cementite interface. Thus, an increase in the carbon content or a decrease in the interlamellar spacing would lead to an increased carbide surface area, and hence to an increase in the number of dislocations generated.

Duckfield (1971a) has attributed the two stage hardening process to the following, based on thin foil electron microscopy observations. During the early stages of wire drawing, deformation of the pearlite is largely confined to the ferrite lamellae, which initially show rapid dislocation multiplication, followed by the formation and progressive refinement of distinct sub-cell structures. The work hardening rate during this stage is normally about 350 MPa/unit strain for a high carbon steel wire.

The second stage of hardening derives from the deformation of cementite, which results in the formation of distinct sub-cell structures within the cementite lamellae. This occurs only at high strains, and a work hardening rate of about 950 MPa/unit strain is typical during Stage II hardening.

The ability of cementite to deform in this manner is not particularly surprising since high hydrostatic pressures develop during drawing. There is, however, some doubt as to the point at which cementite deforms. Nishimura et al (1980), for instance, examined some cementite lamellae after just 19% deformation, and found dislocation densities apparently within the lamellae of the order of 10^{14} m^{-2}, which is significantly higher than in as-patented steels.

Godfrey (1963) attributed the increased Stage II hardening rate to an additional strengthening effect arising from the preferred orientation of the ferrite grains, which only becomes observable after about 50% reduction in area.

Kelly and Nutting (1963) have shown that the dislocation density is unlikely to increase much above 10% strain, at which point the dislocation density should be about 5×10^{15} m^{-2}. Varo (1964), however, pointed out that the number of active dislocations decreases with strain at high strain levels, and that this accounts for the increase in strength of the material. Evans et al (1972) have proposed that an abnormally high number of dislocations are produced during warm straining (corresponding to production wire drawing). This phenomenon is probably due to the mechanism of Gilman and Johnson (see Section 2.2.1).

Embury and Fisher (1966) investigated three possible strengthening mechanisms in operation during the deformation of pearlite. The first mechanism was that of fibre strengthening, due to the orientation of the cementite lamellae along the deformation line, such that the structure behaves as a composite material. The authors discounted this mechanism for a number of reasons, not least of which being that drawn pearlite behaves similarly to drawn bainite, ferrite and tempered martensite, in which no fibre strengthening can occur.

The second mechanism, that of dispersion strengthening by the Orowan mechanism, was also discounted, due to the simplicity of the model.

The third source of strengthening was due to the substructure generated by drawing. The microstructure of drawn pearlite wire indicates that the material consists of cells orientated in the drawing direction. The cell dimension perpendicular to the drawing direction was observed to decrease with increasing drawing strain. It was assumed that the cell walls were the barriers responsible for strengthening, analogous to that of the Hall-Petch relationship for the effect of grain size, and their experimental results support this assumption.

2.3.4 Wire Properties

The strength of drawn wire depends on the chemical composition of the steel, the heat treatment applied, the extent of cold working, and the drawing conditions. Chemical composition and heat treatment have been briefly reviewed above; this section

attempts to illustrate the general effects of the other parameters on wire properties.

It has already been shown that the tensile strength of drawn wire depends on the amount of strain applied during drawing. This effect is illustrated in Figure 2.9 (after Shipley, 1963). Both the tensile strength and the ductility of the drawn wire vary according to the production technique used (drawing schedule, drawing speed, lubrication etc.). Great care must be taken to avoid a phenomenon known as "overdrawing", in which the wire tends to break up during deformation.

Overdrawing may refer to the stage at high strains where the cementite lamellae rapidly disintegrate, leading to microcrack formation and a severe loss in ductility. Alternatively, it may refer to a point at which extensive dislocation pile-ups lead to micro-fissuring, and an eventual collapse in the material properties (Lanner, 1981). In either case, overdrawing tends to occur earlier in steels with higher carbon contents, since their ability to deform plastically is reduced.

The trend in material property changes which occur during the drawing of carbon steel wire under production conditions is shown in Figure 2.10. It can be seen that the method by which ductility is evaluated (e.g. elongation at fracture, reduction in area at the fracture, twists to failure or number of reverse bends), will give marked differences in the measured ductility. In particular, the elongation at fracture decreases sharply, while the number of reverse bends and twists to failure maintain a low level during the early stages of drawing. Beyond about 45% reduction in area (a true strain of 0,6) the latter properties increase up to an optimum value at about 70% reduction (a true strain of 1,2). During this time, however, elongation at fracture decreases further.

Cold drawn wire characteristically possesses a low elastic limit. Aitchison (1923) attributed this to the presence of planes in cold worked metals on which slipping could occur easily; these planes being those on which slipping had taken place during the cold working of the metal.

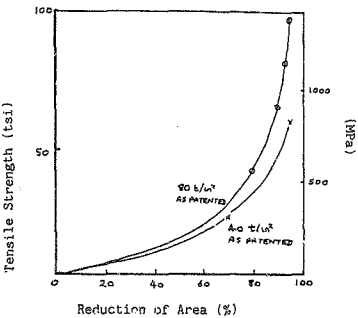


Figure 2.9 : Effect of Cold Drawing on Tensile Strength. After Shipley (1963).

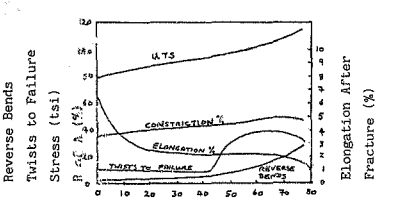


Figure 2.10 : Influence of Reduction of Area by Drawing on the Mechanical Properties of Steel Wire. After Shipley (1963).

The application of a low temperature heat treatment has been used in the wire industry for many years to recover a high elastic limit : *UTS* ratio, particularly for material used for springs and prestressed concrete wire. The effect of such a heat treatment on wire properties can be summarised by Figure 2.11.

For roping purposes, it is desirable to retain the low elastic properties, since forming of the wire into strand is more easily achieved. In addition a low elastic limit enables the wires to accommodate the high interwire forces which occur within a rope.

2.3.5 The Effects of Alloying Elements on Wire Properties

2.3.5.1 Carbon

The strength of hypo-eutectoid plain carbon steel wire is directly related to the carbon content of the wire, the strength increasing with the volume fraction of pearlite present.

In addition, it has been observed that higher ductilities may be recorded from steels whose microstructures do not exhibit any free ferrite (Franklin et al, 1980). This therefore implies that the optimum combinations of strength and ductility are provided by eutectoid compositions. Although further strength increases can be achieved using hyper-eutectoid steels, in wire drawing practice it is unusual to use steels with a carbon content greater than 0,85%. This is due to the increased difficulty in heat treating and cold working such material, since ductility is adversely affected by the presence of grain boundary cementite (Shipley, 1969).

2.3.5.2 Manganese

Manganese is usually found in plain carbon steels, partly due to its use as a desulphuriser. It has been shown by Franklin et al (1980) that manganese slightly delays the austenite decomposition during patenting, and also increases the transformation time. Thus, although manganese increases hardenability, the longer transformation times may lead to increased pearlite interlamellar spacings, and hence lower strength levels.

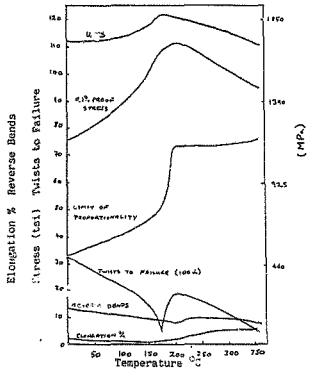


Figure 2.11 Effect of low-temperature heat treatment (30 min. soak) on the mechanical properties of a 0,144" (3,65mm) dia. plain carbon steel wire (0,81% C). After Shipley (1963).

Excess manganese can cause problems due to microsegregation, and thus manganese additions are usually limited to less than 1%. In addition it has been shown that lower manganese contents are beneficial in promoting high as-patented strengths (Cahill and James, 1968; and Nakamura and Fujii, 1974). Franklin et al (1980) demonstrated that compositions containing only 0,6% manganese and 0,76% carbon yielded excellent as-patented strengths, compared to compositions containing up to 0,8% carbon and up to 1% manganese.

2.3.5.3 Molybdenum

Molybdenum has a marked effect on the hardenability of steel even at low levels (typically 0,1%). However, molybdenum has only a small effect on the as-patented strength, while it also gives excessively long transformation times (Reeves, 1985).

Molybdenum also displaces the nose of the pearlite transformation curve to higher temperatures, thereby allowing easier formation of bainite. Such bainite need not necessarily be detrirental to material strengths on drawing, although ductility may be adversely affected.

2.3.5.4 Silicon

Silicon is commonly used as a deoxidiser in steels used in wire drawing, and levels are often found in excess of 0,2%. Silicon, like molybdenum, has little effect on the as-patented strength (Franklin et al, 1980; and Yamakoshi et al, 1977), and affects hardenability in a similar manner.

The use of silicon as a solid solution strengthener therefore appears to have limited potential, but it may tend to inhibit lattice diffusion in the ferrite, due to lattice distortion, which may inhibit detrimental strain ageing (Smith, 1983).

2.3.5.5 Other Elements

The residual elements sulphur and phosphorus have little effect on wire properties as long as they are maintained within certain well-defined limits (i.e. less than about 0,04%; Shipley, 1963).

Nickel and chromium can have marked effects on properties, but may, as for other elements, increase hardenability excessively, and also may introduce segregation problems.

Copper and tin may have a profound detrimental influence on the properties of cold drawn wire, due to localised hot-shortness at surface grain boundaries. Both these elements must, for ultra-high tensile wire, be maintained at very low levels (i.e. below 0,05% for copper and below 0,02% for tin).

2.3.6 Wire Testing

A number of traditional wire tests have been in use for many years and have been specified by British Standard B.S.2763:1982 for rope wire. These tests are for diameter, tensile strength, and number of torsions and number of reverse bends to failure.

The number of twists to failure in the torsion test is considered to be the principal criterion of ductility, and has been the subject of a number of investigations (e.g. Shipley, 1963; Duckfield, 1971b; Godecki, 1969; Nishioka and Nishioka, 1971; and Middlemiss and Hague, 1973). The required performance levels are empirical and vary with wire diameter, and tensile grade and type, but the test does provide a reproducible method for comparing the torsional properties of wires of similar strengths and diameters.

The major problem with the torsion test is the lack of good correlation between the number of twists to failure and wire performance in a rope (Stephenson et al, 1983). Recent unpublished work at British Ropes Ltd, quoted by Stephenson et al, has shown that the type of torsion fracture may be used to some effect in selecting wires of different performance potential, but the correlation is still not entirely adequate.

Elongation and constriction in the tensile test, and the proof stress : tensile strength ratio, can also provide some indication of the likely performance of a wire in a rope. However, again the correlation is poor except in certain extreme situations. Such extreme cases occur particularly where there are significant strain ageing effects, induced either

deliberately by low temperature heat treatment or during drawing using inadequate cooling (see Section 2.4.3).

Stephenson et al developed the "shear test", which attempts to simulate the principal loading conditions in a rope wire, which are most severe at the strand cross-over points in the rope. If the wire is brittle, a characteristic in-service failure occurs with an oblique fracture with little or no elongation.

The test can initiate similar failures but may not provide an absolute measure of subsequent rope performance since the test conditions were derived empirically. Test results are therefore comparative.

The test is performed in a standard wire tensile testing machine, with a special hydraulic compression jig attached to the wire within the gauge length. The jig provides a sideways compressive force on the wire, such that the resultant of this force and the tensile force is a shear force at approximately 45° to the tensile axis. The wire is then tested to destruction. The so-called shear elongation is an estimate of the extent of elongation experienced after the maximum load has been reached. A wire exhibiting good ductility will reveal a high shear elongation value, with a tensile-type fracture surface. A wire of low ductility may show zero elongation and will exhibit an oblique shear fracture surface.

2.4 STRAIN AGEING IN DRAWN PEARLITIC STEELS

2.4.1 Introduction

In general, the effects of severe strain ageing in high carbon steel wire can be summarised as :

(i) an increase in wire tensile strength;

(ii) an increase in the tensile proof stress as a proportion of the UTS;

(iii) a reduction in the number of twists to failure in the torsion test (usually associated with localised twisting, spiral splitting (or delamination) and a brittle fracture); and

(iv) a decrease in the elongation after the maximum load is reached in the shear test (accompanied by a change in the fracture mode from tensile to oblique shear).

In order to identify the prime cause of the drop in ductility of carbon steel wire, Middlemiss and Hague (1973) conducted a series of detailed investigations into the torsional behaviour of carbon steel wire. Their results clearly showed that the observed deterioration in properties was due entirely to strain ageing, and not to surface effects, internal stresses or microstructural damage.

2.4.2 The [illegible] Mechanism

Yamada (1976) has performed artificial ageing experiments on a variety of eutectoid steel wires, drawn at very low speeds in order to maintain a very low wire temperature during drawing. This procedure was adopted to minimise (or eliminate) strain ageing during the drawing process. The material was subsequently aged at various temperatures for different times. The ageing process was monitored by internal friction techniques and changes in electrical resistivity, in order to infer the mechanisms involved.

In this work three stages of ageing were identified as described below and illustrated in Figure 2.12. The given temperatures apply to the experimental conditions of artificial ageing: isochronal ageing for five minutes.

The first stage, which occurs below about 150°C, corresponds to the first stage in Cottrell's theory of ageing (Section 2.2.1). Thus, supersaturated carbon and nitrogen in solution in the ferrite migrate to dislocations, thereby locking them. The yield stress increases mildly, and a decrease in the electrical resistivity can be measured. It was also found that the carbon and nitrogen Snoek peaks observable using internal friction techniques, and a background attributable to dislocation motion, all decrease in height, with the Snoek peaks eventually disappearing completely.

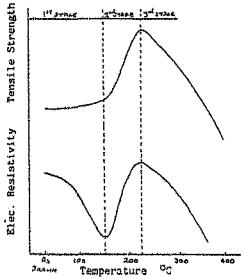


Figure 2.12 Schematic representation of static strain ageing characteristics of cold drawn 0,80% C steel. After Nakamura et al (1976).

The carbon supersaturation after patenting was assumed to be small, and only a small part of first stage ageing was attributed to carbon. The more significant role was assumed by nitrogen. The activation energy was calculated to be about 84 $kJmol^{-1}$, which corresponds closely with that for interstitial solute diffusion of nitrogen in ferrite (Section 2.2.3).

The second stage of ageing occurs at temperatures between approximately 150°C and 250°C. During this stage, electrical resistivity and tensile properties increase, while the internal friction background decreases further. Analysis of these results yielded an activation energy for the stage of 117 $kJmol^{-1}$, and an order of reaction of 2,2 to 2,4.

Second stage ageing was not apparently influenced by the nitrogen content of the steel, and it was shown that for very low carbon wires, no increase in resistivity occurred.

The inference from these results is that after first stage ageing, the carbon supersaturation is essentially removed, but that not all the dislocations are pinned. This is evidenced by the still significant internal friction background. Therefore, for age-hardening to occur, interstitials must be furnished to dislocation sites. Yamada proposed that cementite dissolution coincides with the observed decreases in internal friction.

Support for this hypothesis has been provided by studies of the thermodynamics of strain ageing in medium and high carbon steel by Gul and Babich (1980). The driving force for strain ageing in these steels was found to be the reduction in free energy due to the entropy increase associated with the migration of carbon atoms from cementite to dislocations in ferrite.

The strain ageing process can be expressed as follows:

$$Fe_3C = 3Fe + C \quad \text{.....(2.8)}$$

$$C + D = C.D \quad \text{.....(2.9)}$$

$$Fe_3C + D = 3Fe + C.D \quad \text{.....(2.10)}$$

where D is a dislocation site to be occupied by a C atom; and C.D is a carbon atom occupying a dislocation site.

A rate equation can be derived such that:

$$\frac{d(n_{CD})}{dt} = k.n^2_{cm} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2.11)$$

where $(1 - n_{CD})$ is the fraction of carbon atoms already occupying the dislocation sites.

Equation 2.11 represents a second order reaction and thus corresponds closely with Yamada's experimental value for the order of the reaction. The "true" reaction rate can then be expressed by (Yamada et al, 1983):

$$\frac{dn}{dt} = K_o.n^{2.3}.\exp\left(\frac{-Q}{R.T}\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots(2.12)$$

Where K_o = constant; $3{,}6 \times 10^{11}\ s^{-1}$;
R = gas constant; $8{,}314\ JK^{-1}mol^{-1}$;
and Q = activation energy; $117\ kJmol^{-1}$.

The driving force for the reaction can be shown to be due to an enthalpy advantage. In the literature, values for the binding energy of carbon to cementite range from 40 to 54 $kJmol^{-1}$, and for carbon to dislocations from 50 to 70 $kJmol^{-1}$ (the actual value varies according to the position of the carbon atoms on the dislocation line).

The binding energy of carbon to cementite can be considered to be equal to the difference in the activation energies of *cementite dissolution and precipitation. Since* Yamada's *second* stage of ageing apparently coincides with cementite dissolution, the activation energy for cementite dissolution was found to be 117 $kJmol^{-1}$. Reported values for cementite precipitation are about 84 $kJmol^{-1}$, which thus implies a value for the binding energy of carbon to cementite of about 33 $kJmol^{-1}$. This value is slightly lower than the quoted values, but this could be explained in terms of plastic deformation and fragmentation of the cementite lamellae (Yamada, 1976).

Further evidence to support the theory of cementite dissolution is provided by the effect of an intermediate second stage ageing treatment applied between two dies. This produced more significant strengthening during subsequent first stage ageing,

and this could be accounted for by the additional carbon in solution provided by cementite dissolution. This result has been confirmed by Heape (1984a) and Hardy and Boxall (1957), who also found that repeated straining and ageing of a low carbon rimming steel produced a greater increase in tensile properties than if ageing were carried out only after the completion of straining.

The importance of carbon content has been demonstrated by Babich (1969) and Krishtal et al (1964). Babich found that the extent of strain ageing was dependent on the pearlite content, from which he also inferred that cementite was providing carbon atoms for strain ageing. It was found that the carbon in solid solution was completely precipitated when a low carbon steel was drawn to a 75% reduction in area, and in this way, strain ageing eventually stops. However, in high carbon steel wire, strain ageing may continue indefinitely at room temperature, some property changes still occurring after eight years.

The third and final stage of ageing, according to Yamada (1976), only occurs significantly above 250°C and corresponds to overageing. Here, the tensile strength and electrical resistivity decrease, as does the internal friction background.

Varo (1964) investigated the effect of a commercial low temperature heat treatment on the properties of cold drawn wire. Such a low temperature heat treatment is commonly applied to wire for the production of springs, prestressed concrete strand, etc. and is often referred to as a stress relieving treatment; the object is to increase the elastic properties of the wire.

Varo also summarised the effects of this ageing treatment on the mechanical properties of high carbon steel wire in terms of three mechanisms. At temperatures below about 150°C, a rapid recovery in moduli is observed due to the rapid release of residual stresses. Between 150°C and 200°C, strain age hardening occurs by the precipitation of carbon and nitrogen on dislocation sites, and finally, at temperatures above about 200°C, further strain age hardening occurs by the interaction of dislocations. The latter phenomenon occurs as dislocations in pile-up groups, at grain boundaries and cementite particles, move under the influence of a back stress and interact with other dislocations, thereby becoming sessile.

Varo's conclusions were derived from an extensive series of tests on production wire, drawn with minimal interpass cooling. It is thus reasonable to assume that significant ageing of the wire had already taken place during drawing. Nevertheless, the observed changes in mechanical properties (Figures 2.13 and 2.14) agree with those observed by Yamada (1976) for material drawn under closely controlled low temperature conditions.

2.4.3 The Effect of Drawing Conditions on the Ageing Behaviour of Steel Wire

A considerable amount of work has been carried out on the effects of various drawing parameters on the ageing behaviour of steel wire. Unfortunately, the number of interacting factors is large, and attributing observed effects to certain production parameters is extremely difficult. Such parameters include drawing speed, lubrication conditions, strain rate, overall reduction, drawing schedule (i.e. reduction in area per pass), die and wire cooling efficiency and the residual stresses introduced. In addition, clearly the starting rod must be considered in terms of composition, homogeneity, inclusion level, casting defects, patenting temperature, and pearlite spacing and uniformity.

All of these parameters may have some effect on the ageing susceptibility of drawn wire. Based on the extent of the present knowledge, an attempt will be made to isolate the effects of the various production parameters on ageing behaviour.

2.4.3.1 Wire Temperature

From the point of view of ageing, temperature is the most important factor in wire drawing. The wire temperature is influenced by the wire strength and diameter, the drawing speed, the strain rate, and the efficiency of both lubrication and cooling. Duckfield (1971a) has proposed that ageing during drawing contributes from 50 to 150 MPa to the UTS of drawn carbon steel wire.

The drawing speed is largely predetermined by production constraints such as economics and machine capabilities. Typically, production wire is produced at rates of about 2 to

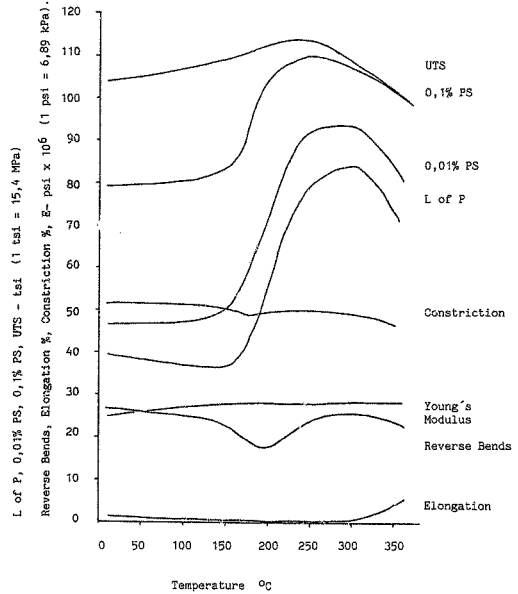


Figure 2.13 : Effect of Low Temperature Heat Treatment (5min soak) on the tensile properties of 0,048" (1,22mm) spring wire. After Varo (1964).

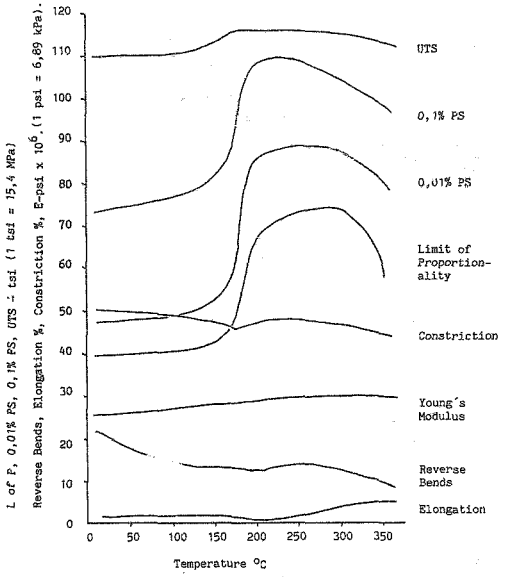


Figure 2.14 : Effect of a Low Temperature Heat Treatment (5min soak) on the Tensile properties of 0,0915" (2,32mm) spring wire. After Varo (1964).

10 ms^{-1} at the final pass. Smith (1973) has shown that from a metallurgical viewpoint, there is no objection to speeds up to 20 ms^{-1} as long as adequate interpass cooling is maintained. Indeed, the dry sodium and calcium soap lubricants appear to increase in efficiency towards the higher speeds, resulting in a reduction in the drawing load required. The mode of deformation was noted to be unaffected by the drawing speed.

The effect of the drawing speed on the wire temperature is not clear, but from measurements made under production conditions (Evered, 1985), it appears that the bulk wire temperatures at the die exit remain approximately constant for drawing speeds in the range 3 to 7 ms^{-1}, as long as efficient interpass cooling is maintained. Wire surface temperatures within the die, however, are markedly influenced by the drawing speed, due to the inability of the cooling system to dissipate the additional frictional heat (Figure 2.15; Pawelski and Vollmer, 1973).

The temperatures in the wire bulk immediately after drawing are typically between 150 and 250°C, while surface temperatures may instantaneously reach temperatures in excess of 500°C (Paxton, 1959; Pawelski and Vollmer, 1973; and Yamada et al, 1983).

Sudo and Yutori (1971) carried out dynamic strain ageing experiments on patented high carbon steel, using a tensile testing method. From their results, Yamada et al (1983) deduced that dynamic strain ageing is not expected to occur during normal wire drawing practice, although the conditions do favour static strain ageing.

Middlemiss and Hague (1973) maintain that dynamic strain ageing can occur during drawing at heavy reductions. This view is supported by the work of Evans and Bhattacharya (1972) and Reynolds (1982). It is possible that due to the exceptionally high surface temperatures which can be produced during wire drawing, strain ageing will occur dynamically within the surface layers, and statically within the wire bulk. The effect on wire properties of either mechanism will be similar in both cases.

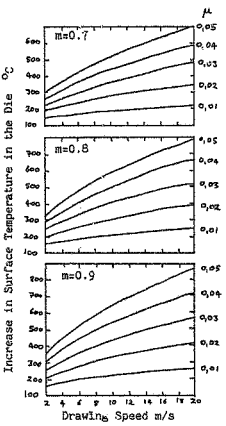


Figure 2.15 : Increase in the Surface Temperature of Steel Wire in the Drawing Die for various speeds and coefficients of friction. After Pawelski and Vollmer (1973).

μ = coefficient of friction, and
m = proportion of frictional heat which remains in the wire.

On the basis of Equation 2.12 Nakamura et al (1976) have calculated a temperature-time relationship for the beginning of static strain ageing embrittlement (Table 2.1). From these data, it can be seen that embrittlement will occur after two hours at 100°C, and after only 700 microseconds at 420°C.

The temperature distribution in a die under typical drawing conditions was also calculated using finite element analysis, and this is given in Figure 2.16. On the basis of these results, it is probable that strain ageing embrittlement will occur very rapidly within the die, during drawing.

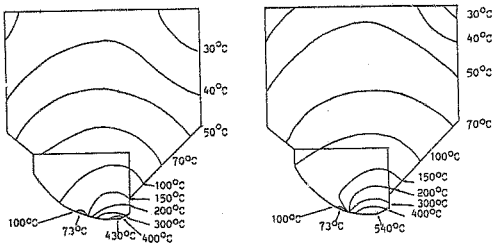
TABLE 2.2 : A TEMPERATURE/TIME RELATIONSHIP FOR THE BEGINNING OF STATIC STRAIN AGEING EMBRITTLEMENT
(after Nakamura et al, 1976)

Temperature °C	Time s	
100	7000	Specimen is a 0,8% carbon steel wire drawn from 1,8mm to 1,0mm diameter.
140	320	
180	20-30	
220	1,5	
260	0,2	
300	0,04	
340	0,008	
380	0,002	
420	0,0007	

The need for an efficient cooling system in wire drawing, in order to prevent or restrict strain ageing during manufacture has been emphasised by many authors. Such systems will be discussed in Section 2.5.2.

2.4.3.2 The Effect of Drafting Schedule and Die Angle

The temperature rise within the die is determined by the amount of useful work performed, and by the amount of redundant deformation, and the friction conditions. A reduced die angle is indicated for the reduction of redundant work (Timiney, 1985), and this would be expected to reduce the maximum temperature achieved. However, the frictional component is increased, and the extra heat thus generated tends to counter the beneficial effects of a reduced die angle in restricting the occurrence of ageing during drawing (Heape, 1984b).



New Cooling Method

Conventional Method

Figure 2.16 Temperature distribution in the die (drawing speed 222m/min, 3,4mm to 2,9mm diameter, 0,63%C, 0,5%Mn, SM-Processed 5,5mm diameter). After Nakamura et al (1976).

The use of a "hump-back" draft to restrict cementite lamellae fragmentation has been shown by the same author to significantly improve the shear properties at high strains, with little improvement in tensile strength. Also, the ageing susceptibility of wires drawn using the hump-back draft was significantly reduced, when compared with the normal taper drafting schedule.

Based on the work of Godecki (1972), this improvement in ageing response may be due to the delay in the point at which the cementite fragments. It was proposed that freshly fractured cementite may have a high surface energy, thereby enabling more rapid cementite dissolution, and a consequent reduction in the activation energy for second stage ageing. Unfortunately, the drafting schedule used by Yamada in his 1976 work was not reported, and thus the results of Heape (1984b) and Yamada cannot be compared directly.

The strain rate during drawing is a function of the wire diameter, the reduction in area per pass, the drawing speed and the die semi-angle, as indicated by Equation 2.13 (after Avitzur, 1968).

$$\frac{\bar{\phi}}{\phi_a} = \frac{3}{2}\cdot\frac{V_e}{R_e}\cdot\frac{f(a)}{(R_o/R_e)-1} \qquad \text{.....(2.13)}$$

where $\frac{\bar{\phi}}{\phi_a}$ = relative average strain rate;

V_e = wire exit velocity;

R_o = wire entry diameter;

R_e = wire exit diameter;

a = die semi-angle; and

f(a) is a somewhat complex function of the die semi-angle.

Thus, in the range of die angles applicable to wire drawing, reducing the total die angle from e.g. 15° to 8° reduces the average strain rate by a factor of only about 1,4.

However, according to this equation, small reductions in area lead to significantly higher strain rates. For instance, a decrease in the reduction from 12% to 4,5% to a wire size of 1,33 mm increases the strain rate by a factor of about 3.

The strain rate is typically a maximum at the last die, where values of the order of $10^3 s^{-1}$ may be achieved. Stephenson et al (1983) showed that the ageing temperature at which the ductility (in the shear test) falls to zero decreased with increasing overall drawing strain. It is, however, not clear from their work whether this is simply a result of the wire more nearly approaching its limit of ductility (or overdrawing limit), or whether the kinetics of the ageing process are enhanced by the more heavily worked microstructure. Nevertheless, the critical level of deformation for overdrawing is probably influenced by the extent of previous strain ageing.

2.4.4 Evaluating Strain Ageing Susceptibility

A number of methods of evaluating the strain ageing response of a hard drawn wire are available, based both on the measurement of mechanical property changes, and on certain physical properties (such as electrical resistivity and internal friction), which are affected by ageing.

2.4.4.1 Tensile Properties

Many authors (e.g. Baird, 1963; and Vrtel, 1967) advocate the use of the change in yield stress or proof stress as the most sensitive mechanical property change to detect ageing. It has been pointed out previously, however that cold drawn wire exhibits a very low elastic limit, and usually does not exhibit a yield point. In addition, the "elastic portion" of the curve is often not linear, and thus proof stress values are also difficult to obtain with confidence. In instances where values can be derived, the scatter band tends to be very large, and thus experience dictates that the elastic properties are not normally used to monitor the progress of [illegible] Shipley, 1985).

The tensile strength has been shown [illegible] affected by ageing, but to a much lesser extent than yield stress. Yamada (1976) showed that tensile strength changed significantly only in the second stage of ageing, and thus the parameter will not be likely to reveal the effects of first stage. Experience elsewhere (Heape, 1985b) supports this view.

The constriction and elongation at fracture in the tensile test also do not provide reliable data on the extent of ageing, since the results are subject to excessive scatter.

2.4.4.2 Hardness Tests

Hardness values can often be interpreted approximately as the stress at about 8% strain, and are therefore expected to follow the ageing process in a manner intermediate between yield stress and tensile strength. The problem with hardness tests is that the tests are performed on specific planes of the material, while the tensile test, for instance, measures the weakest wire properties along the gauge length of the wire. In addition, hardness values tend to vary across the cross section of a wire (Figure 2.4).

Thatcher and Whyte (1985) have shown that the hardness does change as the ageing process continues, and that the ratio of UTS to hardness may provide a good measure of ageing. This ratio decreases as the degree of ageing increases.

2.4.4.3 The Torsion Test

Lanner (1983) has described the effects of ageing on the torsional properties of aged carbon steel wire, as follows. The wire becomes more inclined to break within the grips (i.e. it becomes more notch-sensitive), while the occurrence of a stepped fracture and a seamy condition, or delamination, appears along the wire length. The test pieces may also start to bulge, and to fail at lower numbers of twists to failure. These observations have been confirmed by a number of authors (e.g. Stephenson et al, 1983; and Heape, 1985b) with the additional observation that in many cases the scatter in the results increases at the onset of ageing (Reynolds, 1982; and Niedzwiedz and Pyka, 1979).

It has been suggested that the mode of failure in the torsion test seems to be a more reliable measure of ageing than the number of twists to failure (Stephenson et al, 1983). The torsion test thus appears to be a good indicator of the onset of ageing, although the results tend to be qualitative, since the failure mode is difficult to quantify, while the measured number of twists to failure is unreliable.

2.4.4.4 The Shear Test

The shear test (as developed by Stephenson et al, 1983; and described in Section 2.3.6) has been shown to be an efficient and fairly consistent indicator of the onset of wire embrittlement by ageing (Heapa, 1985b).

The difficulties with this test at present are that the results again are subject to some scatter at the onset of ageing, while the quantitative data derived from the test are difficult to interpret in terms of wire performance. In its present form, the test is perhaps too crude to monitor the ageing process throughout, since shear elongation values tend to fall to zero rapidly after ageing has commenced.

2.4.4.5 Electrical Resistivity

Yamada (1976) obtained much of his quantitative data on the kinetics of ageing from the change in electrical resistivity during ageing (see Section 2.4.2). The change in resistivity appears to follow reliably the changes in mechanical properties, such that the temperature at which maximum resistivity is achieved coincides with the highest measured 0,2% proof stress, and UTS, i.e. with the maximum strain age hardening.

King and Greenough (1960) and Andrews (1973) have described how resistivity is affected by lattice defects such as interstitial atoms, dislocations, stacking faults and grain boundaries. The theories involved are complex, but essentially any such defect introduces localised strain fields in the lattice which increase the electrical resistivity. Therefore any process which reduces the amount of local strain fields, such as ageing, will decrease the resistivity. Yamada's observation that resistivity increases during second stage ageing can therefore only be accounted for if the concentration of lattice defects increases during this time.

Elementary calculations of the amount of carbon in solution in the ferrite can be made, based on the work of Volgar (1961) who showed that the carbon contribution to the resistivity increase is 2×10^{-7} Ωm per wt% carbon.

It is advisable to measure resistivity at low temperatures (e.g. in liquid nitrogen) to avoid thermal scattering effects. Thus the monitoring of ageing using resistivity would probably involve ageing at the required temperature followed by quenching into liquid nitrogen for the resistivity measurement. The procedure would therefore be stepwise rather than continuous.

Resistivity measurements of cold worked alloys can yield much useful information regarding the microstructure of the alloy. However, ambiguities frequently arise in the interpretation of the results, and it is recommended that results are obtained simultaneously by other techniques, to avoid confusion.

2.4.4.6 Damping Capacity

The amplitude of free vibration of any solid decreases with time. This is due to a progressive transformation of vibrational energy into other forms, the most notable of which is heat. Some of the fall in amplitude can be attributed, for example, to friction with the surrounding medium (e.g. air), friction at the sample supports, or to accoustic energy, but an inherent damping capacity (or internal friction) is possessed by all solids, whereby the solid itself converts vibrational energy into other forms. Entwhistle (1960) has reviewed the subject in some detail.

Quantitatively, damping capacity (or internal friction) is measured by the amount of energy transformed within the solid during one cycle of vibration. This may be determined from the rate of decrease of the amplitude of vibration of the solid, or by the energy required to maintain a predetermined amplitude. The magnitude of the damping capacity is dependent on the microstructure of the solid, and thus the value of the damping capacity can be used to identify microstructural features.

Experimental techniques can be broadly classified into two groups (according to Entwhistle, 1960):

1. A bar of uniform section is freely suspended and is set to vibrate at one of its natural frequencies.

2. A bar or wire specimen is provided with rigid inertias to reduce the frequency of vibration.

Great care must be taken in the design of the measurement apparatus, in order to account for the effects of friction, etc.

Typically, the internal friction of the sample material is measured over a range of temperatures, in order to obtain internal friction vs temperature profiles. This is because different microstructural features contribute in different proportions to the damping capacity at different temperatures. Thus, for example, peaks in the profile may be ascribed to the effect of carbon atoms in solution in ferrite, or to vacancies. The relative heights of the peaks can be used to estimate, for instance, the amount of solute present.

In order to monitor the ageing of a steel wire on a continuous basis, the wire would have to be held at the Snoek peak temperatures for the interstitial elements under consideration, (i.e. 38°C for carbon and 25°C for nitrogen).

The interpretation of the internal friction/temperature profile can be very difficult, as is the case for electrical resistivity testing. Interpretation generally becomes more difficult as the microstructure under investigation becomes more complex.

2.5 THE SUPPRESSION OF STRAIN AGEING IN DRAWN PEARLITIC STEELS

2.5.1 Introduction

Essentially, there are two methods which can be considered in an attempt to eliminate the strain ageing phenomenon in drawn pearlitic steel wire. The first and most important method commercially, is to restrict temperatures during drawing to a minimum using interpass cooling systems. This would be immediately applicable to certain current industrial production facilities with other benefits, such as increased die life. The second method is to alter the chemical composition of the rod to raise the activation energy for strain ageing to a level where no significant natural ageing occurs after drawing.

One problem with restricting ageing during drawing is that ageing effects may account for between 50 and 150 MPa of the as-drawn UTS, and this contribution is probably important in the production of ultra-high tensile wire. Thus, if ageing during drawing were to be prevented by efficient wire cooling, the additional strength required would probably have to made up by increasing the extent of cold working (i.e. by drawing to higher strains). However, alloying additions may be used to restrict ageing, and to increase the as-patented strength of the material, and thereby satisfy both requirements. Each of these aspects will be described below.

2.5.2 Cooling During Drawing

Traditional methods of interpass cooling have focussed on cooling the wire on the block (or capstan), using forced air and internal water cooling of the block to assist heat transfer. The heat transfer situation here is extremely complex, since three media (steel, air and water), and two significant modes of heat transfer (conduction and forced convection) are involved.

Alexander (1975) has used advanced computer analysis to study this problem and his results correlated well with experimental values. It was then possible to analyse different wire drawing situations to find the optimum conditions.

It was found that peak wire temperatures on a drawing frame increased with increasing drawing speed (although not in direct proportion). However, the temperature could be maintained at a constant level by increasing the block diameter and wrap height (see Figure 2.17) so that the total wrap area was increased in proportion to the speed. The analysis also indicated that changes in coolant temperature had little effect.

From the analysis of the drafting sequence, the use of a taper draft schedule was recommended to minimise the peak wire temperatures. An increase in the number of passes was found to reduce the temperature rise through each die.

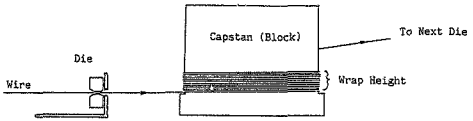


Figure 2.17 Schematic of Die/Capstan Arrangement Showing Wrap Height.

Middlemiss (1972) states that a temperature rise of the order of 120°C per pass may be expected when drawing high carbon steel wire at reductions of about 20%. He therefore developed a more direct cooling system than simple internal cooling of the block, whereby water is sprayed directly onto the wire on the block. Excess water was removed using a compressed air wipe at the block take-off. This latter point is important since any water present on the wire can lead to immediate lubrication breakdown in the subsequent dies.

The efficiency of this system is illustrated by Figure 2.18 which shows that incoming wire at 170°C can be cooled to the coolant temperature before leaving the block. This data was derived from a test on a commercial six-hole draw bench, using water cooling only on the fourth block.

The effect of this procedure on the tensile strength of the wire is illustrated in Figure 2.19. Using the same finishing speed the tensile strength obtained was 30 to 70 MPa lower than that produced in identical wire drawn without any cooling. The implication from this work is that the finishing speed, and hence production levels can be increased without degrading the mechanical properties.

Nakamura et al (1976) have developed an alternative type of cooling system, whereby both the die and the wire exiting the die are directly cooled with water (Figure 2.20). At a finishing speed of 850 $m.min^{-1}$, wire exit temperatures were reduced by 100°C to about 140°C, thereby possibly inhibiting second stage ageing. The results (Figure 2.21) again showed that the work hardening rate decreases with cooling, although this can be rectified by increasing the drawing speed. An additional advantage of superior cooling, commercially, is that die life is extended, an observation confirmed by Alexander (1975) and Middlemiss (1972).

Nakamura et al also analysed the temperature distribution in the die, and compared this to the conventional method without direct cooling. Figure 2.16 shows that peak die surface temperatures were also reduced by over 100°C which may account for the extended die life.

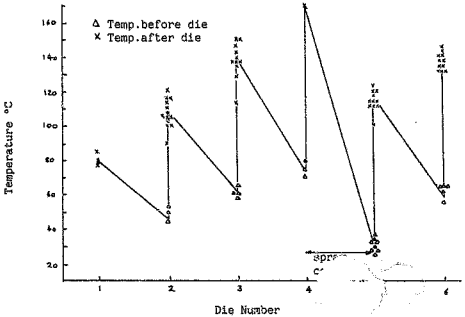


Figure 2.18 : Wire Temperature Measured Before and After the Die (finishing speed 180 to 195 m/min). After Middlemiss (1972).

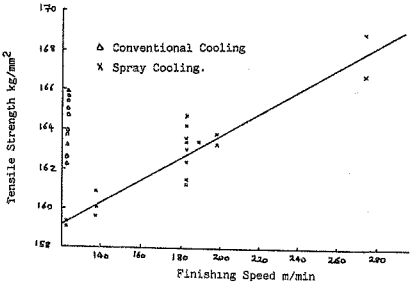


Figure 2.19 . Influence of Finishing Speed on Tensile Strength of Drawn Wire. After Middlemiss (1972).

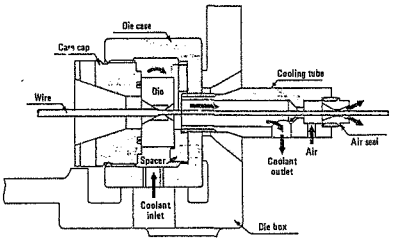


Figure 2.20 Schematic Cross-Section of the New Cooling Equipment After Nakamura et al (1975).

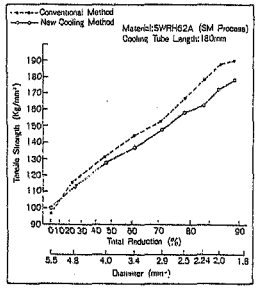


Figure 2.21 Variations in Tensile Strength (finishing speed 850 m/min). After Nakamura et al (1976).

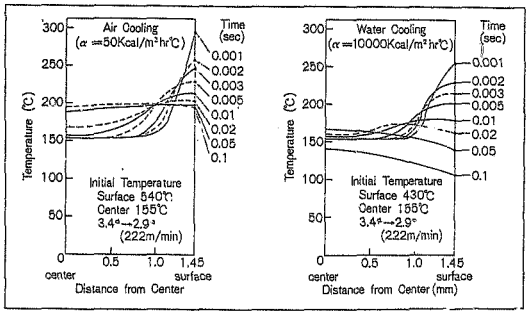


Figure 2.22 Temperature Distribution in Drawn Wire. After Nakamura et al (1976).

Analyses of the temperature distribution in the wire itself (Figure 2.22) showed that, with their cooling system the wire temperature can be brought to below 150°C within about 0,05 seconds, under their operating conditions. The cooling tube length required can then be calculated on the basis of the required finishing speed.

It was noted, however, that dynamic strain ageing may still occur within the surface layers within the die hole. Unfortunately, it seems to be impossible at present to reduce the peak die temperatures still further.

Heape (1985c) and Middlemiss et al (1973) attempted to eliminate strain ageing by pre-cooling the wire before the die using dry ice (solid carbon dioxide), in combination with very low drawing speeds. Using this technique Middlemiss et al demonstrated that natural ageing subsequent to drawing could be completely suppressed if wire temperatures could be maintained low enough. This, however, required a drawing speed of $1,4 \times 10^{-3} ms^{-1}$, which is obviously of no commercial value. Even with drawing speeds as low as $0,05\ ms^{-1}$ (Heape, 1985c), in combination with additional direct water cooling, a small degree of embrittlement could be induced by ageing at 50°C for 200 hours.

It is apparent therefore that the complete elimination of ageing in hard drawn wires, even at low temperatures (i.e. natural ageing), is not possible by using only elaborate cooling systems. However a definite improvement can be achieved, and for commercial purposes, this may be sufficient.

2.5.3 Chemical Composition of Wire Rod

The chemical composition of the wire may have a marked effect on the rate and extent of ageing of the wire, both during and after drawing. This aspect will be considered below.

Heape (1983) has shown the possible benefits of slightly reducing the carbon content of the wire. Initial tests showed that a wire with a carbon content of 0,72% can be drawn to higher true strains than a wire containing 0,80% carbon, thereby

giving higher strengths and improved ductility. In addition, such wires also yielded improved ageing properties. These results, however, are subject to confirmation in further tests.

Some work performed on a 0,76% carbon steel which had been specially heat treated to give a particularly uniform pearlitic interlamellar spacing prior to drawing (Heape, 1984c) was also found to have an excellent combination of tensile and shear properties. The ageing properties were, however, not evaluated.

A finer pearlite interlamellar spacing has been shown (Heape and Davies, 1984) to increase the drawn tensile strength as would be expected, but also to increase the ageing susceptibility. This is probably due in part to the high work hardening rate, but may also be due to the shorter ferrite free path.

As described in Section 2.2.5, commercial control of strain ageing normally involves the addition of strong nitride-formers to tie up excess interstitial solute. Nitrogen, however, is unlikely to have a major effect on ageing in drawn pearlitic steels, according to Yamada (1976), and therefore the addition of, for instance aluminium or titanium may not prove beneficial.

Since the strain ageing rate may be presumed to be determined by the rate of diffusion of carbon in ferrite, it would seem appropriate to consider alloying elements which will inhibit carbon diffusion. The diffusion rate of carbon in ferrite was given by Equation 2.4:

$$D_c = 2{,}0 \times 10^{-6}.\exp\left(\frac{-84\,100}{R.T}\right)\ m^2s^{-1} \quad \ldots\ldots\ldots\ldots(2.4)$$

Thus, in order to reduce the diffusion rate, the pre-exponential term must be reduced, while the activation energy term should be increased.

Data on the effect of alloying elements on the diffusion rate of carbon in ferrite are relatively scarce, but trends referring to carbon diffusion in austenite should also apply to diffusion in ferrite (Smith, 1985; and Krishtal, 1970).

Krishtal (1970, collected information from a number of sources on the effect of various alloying elements on the diffusivity of carbon in austenite and his results are shown in Table 2.2.

TABLE 2.2 : THE DIFFUSIVITY OF CARBON IN ALLOYED AUSTENITE
Carbon Content = 0,7% (after Krishtal, 1970)

Alloying Element	Alloy Content wt %	Diffusion Coefficient $m^2s^{-1} \times 10^{-6}$	Activation Energy $kJmol^{-1}$
-	-	0,096	134,7
Ni	4	0,10	129,7
Mn	1	0,11	132,2
Co	6	0,10	127,6
Cr	0,21	0,112	136,4
	0,39	0,126	139,3
	0,62	0,123	141,0
	0,86	0,107	142,3
	2,5	0,19	154,8
Mo	0,9	0,29	141,4
Si	0,3	0,152	133,9
	0,64	0,104	129,7
	0,93	0,035	117,1
	1,22	0,024	110,0
	1,60	0,11	133,9
Al	0,39	0,120	134,7
	0,58	0,124	135,6
	0,83	0,125	136,8
	0,97	0,126	137,6
Cu	1,02	0,10	132,2
	2,04	0,087	130,1

It can be seen that most elements appear to increase the diffusion coefficient of carbon, but some reduce it if added in sufficient quantities (i.e. of the order of 1%). Some elements also increase the activation energy for diffusion, and in particular, chromium raises the activation energy at 1% addition by about 6%. Silicon generally tends to reduce the activation energy for diffusion.

Krishtal also found that combinations of alloying additions are often of greater benefit than single additions; this is evident in Table 2.3, where 1,2% silicon is added in combination with other elements. It can be seen that the effects of two elements are not additive, and there is clearly a synergistic influence.

TABLE 2.3 : THE DIFFUSIVITY OF CARBON IN AUSTENITE CONTAINING 1,2% SILICON AND ONE OTHER ALLOYING ELEMENT.
Carbon Content 0,7% (after Krishtal, 1970)

Alloying Element + 1,2% Si	Alloy Content %	Diffusion Coefficient $m^2s^{-1} \times 10^{-6}$	Activation Energy $kJmol^{-1}$
Al	0,17	0,0138	115,5
	0,33	0,0252	123,8
	0,45	0,0346	129,7
Co	0,96	0,278	138,1
	1,88	0,278	138,1
Cu	0,09	0,0204	110,5
	0,21	0,0214	110,5
W	0,18	0,0451	125,9
	0,39	0,0316	125,4
	0,61	0,0153	125,9
Mn	1,00	0,0513	129,7
	1,52	0,0718	136,4
Mo	0,23	0,0254	125,5
	0,40	0,0257	126,4
V	0,21	0,0257	128,0
	0,60	0,0320	136,4
	0,96	0,0449	138,9
Cr	0,20	0,0089	124,3
	0,38	0,01	128,0
	0,59	0,0067	130,1
	0,82	0,0074	134,3
	1,03	0,0065	135,1

The most promising combination of alloying additions in Table 2.3 is a 1,2% silicon, 1% chromium alloy. It would appear that silicon depresses the value of the pre-exponential term, while chromium compensates for any depreciating effect silicon has on the activation energy for diffusion.

Krishtal also studied the diffusion characteristics of carbon in alloyed ferrite and found that small additions of chromium (of about 1%) have even more marked effects on the activation energy for diffusion. Activation energies as high as 142 $kJmol^{-1}$ can be achieved in combination with a significant reduction in the pre-exponential term. Molybdenum has a similar effect on carbon diffusion in ferrite. The synergistic effect of a combination of alloying additions was again noted, in particular with chromium-molybdenum-manganese mixtures.

Essentially, the reason for the large reduction in the diffusivity of carbon in chromium or molybdenum steels is the strength of the binding forces between the alloying element and carbon. The magnitude of these binding forces may be inferred from the melting points of the chromium carbides which tend to be very high (e.g. for Cr_7C_3, the melting point is 2170°C).

As stated in Section 2.3.5, segregation can be a significant problem in alloyed rod, and may lead to a non-uniform rod structure. Also, the above additions increase the hardenability of the steel, which may lead to unacceptable transformation times. For example, Thatcher (1983) found that the addition of 0,14% molybdenum to a eutectoid steel can lead to required isothermal transformation times of greater than 15 minutes at 500°C.

Recent work by Benson (1984) has shown the benefits of small additions of chromium, silicon and vanadium with regard to ultimate tensile strength after drawing. With judicious control of the composition and the patenting process used, strengths of beyond 2500 MPa have been achieved. The implications for ageing are that reduced amounts of deformation may be required in order to achieve a particular desired tensile strength, thus leading to reduced ageing susceptibility from this aspect also.

Heape (1984d) has shown that steels containing additions of 1,3% chromium and 0,6 and 1,9% silicon exhibit very good ageing properties after drawing, when compared to similarly produced plain carbon (eutectoid) wire. Similarly, Honda and Inoue (1970) showed that a 0,7% chromium, 1,6% silicon, medium carbon (0,53%) steel wire gave good resistance to low temperature tempering, such that the wire only attained a maximum value of UTS during tempering at some 50°C higher than a high carbon music wire with a similar initial UTS.

On the basis of the above, it would appear that alloying can give a marked improvement in the ageing properties of steel wire, and may also give increased tensile strength.

2.6 SUMMARY AND CONCLUSIONS

An overview of the strain ageing phenomenon in steels has been presented, together with a general description of the cold drawing process for the production of steel wire. Each of these subjects is immense, and an attempt has been made only to extract information of direct relevance to the project.

The phenomenon of strain ageing in drawn pearlitic steels has been investigated and the results of several authors have been presented. A general concensus that the progress of ageing can be separated into three broadly distinct stages is apparent, but different interpretations of these stages have been published. The theory that cementite partially dissolves to provide interstitial atoms for ageing appears to be valid, particularly since the dislocation density in drawn steel wires is high, and the interstitial solute concentration is low.

A number of methods by which strain ageing can be detected have been described, and it would appear that, for heavily worked steels the change in yield point is not suitable, due to uncertainty in the accuracy of such measurements in these materials. Other tests, therefore, such as the torsion test and the shear test may be more relevant to this project. Monitoring the change in resistivity with ageing time also appears to be a useful method of following the progress of ageing.

Methods of inhibiting ageing have been discussed. Of undoubted commercial importance is the interpass cooling of wires, to minimise temperature build up and hence to restrict strain ageing during drawing. Direct information on the effect of alloying elements on the strain ageing susceptibility of drawn wire is deficient, but certain alloy combinations such as small additions of silicon and chromium may prove to be beneficial in restricting ageing in finished wires.

CHAPTER THREE : THE ESTABLISHMENT OF EXPERIMENTAL PROCEDURES

3.1 INTRODUCTION

Although the wire drawing process is, superficially, a relatively simple operation, there are a great number of material and process variables which must be considered in any investigation involving wire drawing. Some of these parameters which affect the rod or wire properties, especially those which may affect the strain ageing of steel wire, are listed below.

Material Composition: interstitial elements (e.g. carbon and nitrogen); normal alloying additions (e.g. manganese and silicon); special alloying additions (e.g. chromium, molybdenum and vanadium); etc.

Microstructure: free carbon and nitrogen in ferrite; pearlite interlamellar spacing and uniformity; decarburisation; grain size; etc.

Rod Properties: tensile strength; ductility; surface quality.

Drawing Conditions: drafting schedule; die angle; temperature distribution; residence time at temperature; friction; draw speed; cooling; redundant deformation; total strain; etc.

Post Drawing Conditions: coiling; forming; storage conditions; operating temperatures; etc.

The number of variables is therefore very large, and many are interrelated. It was important from the outset that the ideal experimental conditions be established so that adequate quantities of each wire could be produced which were consistent in metallurgical and mechanical properties along their length such that identical samples were provided for different experiments. Preliminary work therefore concentrated on these aspects, and this is described in the following sections.

3.2 PREPARATION OF WIRE SAMPLES

3.2.1 Material Preparation

The first criteria which required selection were material composition and size. At present, ultra-high strength steel wire is manufactured commercially from plain carbon steel of eutectoid composition. Since the ageing phenomenon was of most relevance to this material, conventional commercial material was used for the initial studies.

Much ultra-high strength wire is required with a final diameter of about 3,1 mm for roping applications, and this is drawn from patented rod of between 9 and 11 mm diameter depending on the desired strength of the finished product. This corresponds to a true strain after drawing of between 2,1 and 2,5.

Initially it was decided that wire of normal size (9 mm or 9,5 mm) be used, since this would have most relevance to "real" conditions. Subsequently, it was decided to change the feed size to 4 mm for the following practical reasons. The smaller feed size enabled closer control of both the heat treatment conditions (across the wire diameter) and the drawing conditions; in particular the drawing speed and wire cooling after the die (and thus ageing during drawing). The finished wire sizes of 1,3 or 1,2 mm (which gave equivalent strains) were also more suitable for the measurement of electrical resistivity. Finally, the amount of wire sample which could be produced from the starting material was vastly increased. This became relevant when it was decided to manufacture experimental alloys for further studies, and the length of wire sample available was severely limited.

3.2.1.1 Commercial Materials

Over a reasonable length, commercially manufactured and processed material possesses generally consistent properties, and the chemical composition and mechanical properties do not alter significantly between the front and back of a sample coil. This is due to the large scale of the operation, which limits local variations in material properties as a result of chemical composition or heat treatment conditions.

The commercial material used in this project was manufactured either by Thyssen* of Germany, or by ISCOR^, locally. Patenting was carried out at Haggie Rand Ltd according to normal production practice (i.e. austenitisation at 960°C for about 6 minutes in a controlled nitrogen atmosphere, followed by quenching into lead at 560°C for 4 mm material, or 540°C for 9 or 9,5 mm material). The 4 mm feed material was supplied by ISCOR in 5,5 mm form, and was therefore drawn to 4 mm diameter prior to repatenting at this size.

3.2.1.2 Vacuum-Melted Material

For certain of the experiments it was required to have close control over the chemical composition of the steel. Use was therefore made of the alloy preparation facilities of the University of the Witwatersrand, as will be described below.

A vacuum induction furnace was used to prepare 5 kg ingots of the required steels. The melting base was pure iron in the form of electrolytic flakes, and this was melted with carbon under an argon partial pressure. Pure manganese and silicon were then added to make up the desired composition. Where control of the nitrogen level was required, a partial pressure of nitrogen (about 13 kPa) was maintained above the melt for several minutes. Close control of the nitrogen content by this method was found to be difficult, and the correct compositions were often achieved by trial and error.

In order to restrict the formation of pipe in the ingot, especially in alloys containing silicon, the superheat was lowered as much as possible before pouring into the mould. Cooling was carried out in the argon atmosphere in order to prevent oxidation.

After cooling, the ingots were rolled into rod suitable for drawing. This was carried out using an experimental rolling mill of 30 tonne capacity.

* Thyssen Edelstahlwerke AG, Krefeld, West Germany

^ ISCOR: Iron and Steel Corporation of South Africa, Vanderbijlpark, South Africa.

The ingots were preheated at 1100°C for two hours prior to rolling and were first cogged down to 25 mm square bar with at least two further reheats of up to 30 minutes each. The rolls were then changed to allow further rolling down to 14 mm or 10 mm or rod. The 10 mm rolls were only used once, since this size caused a number of problems, including laps in the finished rods.

A decarburised zone of about 1 mm existed after rolling, which had to be removed by machining, or by some other method (for example, acid dissolution, Section 4.3.2).

3.2.1.3 Heat Treatment

Heat treatments were carried out at Haggie Rand Ltd either in the works, which uses continuous patenting lines, or in the research laboratory for the batch processing of short wire lengths. The equipment for the latter will be described below.

Austenitisation was performed in a Russ 22 kW muffle furnace, which, for short straight specimens, was equipped with a heat resisting stainless steel tube which protruded through the door, and, within which, the sample was held in the hot zone of the furnace. Nitrogen gas was passed through the tube to restrict oxidation.

Austenitisation temperatures between 900°C and 960°C were used. The furnace temperature was maintained within 10°C using a Eurotherm 020 temperature controller.

Isothermal transformation was then achieved by quenching the rod into a lead bath held at the desired temperature, holding in the bath for approximately one minute, then water quenching. The dimensions of the lead bath were approximately 420 mm by 220 mm by 250 mm deep, and the temperature was again controlled using a Eurotherm controller. The lead temperature was monitored using a type K thermocouple and a Fluke model 8086 digital multimeter. Control of the lead bath temperature throughout the transformation time was within 5°C of the required temperature. The lead was stirred manually to maintain an even temperature throughout the bath.

3.2.2 Analytical Equipment

Chemical analyses of the materials were performed in the Haggie Rand Ltd laboratory (Jupiter works), or at ISCOR (Pretoria works), using the following methods:

carbon and sulphur: Leco carbon and sulphur analyser;
manganese: atomic absorption spectrophotometer (AA);
silicon: gravimetric;
aluminium: AA;
nitrogen: Leco nitrogen analyser (ISCOR);
phosphorus: spectrometer (rod samples only).

Metallographic specimens were prepared using standard techniques. Hot mounted specimens were ground on successively finer grades of silicon carbide paper to 1000 grit, and final polishing was achieved using 3, 1, and finally 1/4 μm diamond lapping compounds.

For light microscopy, specimens were etched in a 2% nital solution, and viewed using a Zeiss inverted metallurgical microscope and a Nikon Optiphot microscope. A Zeiss Ultrephot projection microscope was used to produce photomicrographs.

A Philips model 505 scanning electron microscope (operating at 30 kV) was used for the estimation of the minimum pearlite interlamellar spacing of the steels, and to identify any wire defects. The energy-dispersive X-ray spectrometer (EDS) facility of this SEM was used to determine if there was any gross segregation of substitutional alloying elements. Mounted and etched specimens were gold coated in a Polaron E5100 SEM coating unit, to a coating thickness of about 15 nm.

The minimum pearlite interlamellar spacing was estimated by *scanning the surface of transverse sections* of the steel for fine pearlite colonies, and measuring the interlamellar spacing against a 1 μm marker superimposed on the viewing screen. Ten such colonies were measured for each sample.

3.2.3 Wire Drawing Apparatus

All material was prepared for drawing in the Haggie Rand Ltd works, according to normal factory procedures, as follows:

1. pickling in inhibited hydrochloric acid for 20 minutes to remove mill scale and surface rust;
2. rinsing thoroughly;
3. dipping into a tank of zinc phosphate at 70°C for 6 minutes;
4. rinsing again; and
5. dipping into a borax solution at 90°C for 2 minutes.

This procedure applies a corrosion-protecting phosphate coating to the rod surface, and also a coating of borax. The coatings aid pick-up of the drawing lubricant (soap powder) during drawing, and thereby limit the heat rise in the die due to friction, while also protecting the wire surface from damage.

In his 1976 work (described in Section 2.4.2), Yamada prepared wire sample material at drawing speeds of 50 to 80 $mm.min^{-1}$ in order to avoid temperatures in the steel wire of greater than 30°C. These values compare with finishing speeds of up to 15 ms^{-1} under commercial conditions. Due to the apparent success of Yamada's studies, it was decided to investigate the practicalities of preparing samples at such low speeds.

To this end, a special die and soap box attachment was constructed to fit onto the "live" head of an Avery 1000 kN horizontal tensile test machine normally used to perform tensile testing of rope specimens. An attachment was also made to enable the use of a conventional "pulling-in-dog" or grip in the other head, so that the wire could be pulled through the die. The wire was passed through a conventional sodium soap before entering the die, to assist lubrication.

The tensile test machine was equipped with infinitely variable speed control from 0 to 200 $mm.min^{-1}$. The total available extension for test purposes was about one metre, but by adjusting the position of the heads on the tracks, a gauge length of up to eight metres could be attained. Thus wire could be drawn in short sections up to this length. The wire drawing arrangement is shown in Plates 3.1 and 3.2.

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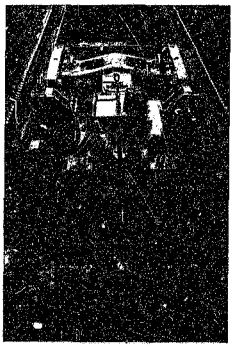


Plate 3.1 : General View of Slow Wire Drawing Apparatus

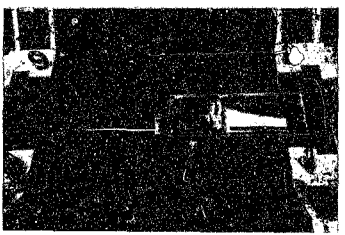


Plate 3.2 : Detail of Die Box Assembly

The work hardening rate of wire during drawing is considered to be a good indicator of the amount of ageing which has occurred during the drawing process (Whyte, 1985). Ageing during drawing will increase the apparent work hardening rate in any one pass. The shape of the tensile strength vs drawing strain curve should be smooth after the first die, and therefore this can be used to indicate the occurrence of ageing, since ageing will introduce steps into the curve. The overall work hardening rate may also be affected.

It was therefore decided to carry out drawing trials using a variety of drawing speeds and cooling conditions (see Table 3.1 for details), using the slow drawing apparatus, in order to determine the work hardening rate. This was monitored by sampling the wire after each die and measuring the tensile strength. The work hardening curves are shown in Figure 3.1, from which it can be seen that no appreciable difference existed between these experiments in terms of ageing during drawing.

TABLE 3.1 : CONDITIONS FOR SLOW DRAWING TRIALS

Rod Feed Size: 9,0 mm
Die Angle: 14°
Lubricant: Calcium Soap

Drawing Speed	Wire Cooling
Expt 1: 14,8 mm.min^{-1} at exit, constant	None
Expt 2: 63,5 mm.min^{-1} at exit, constant	None
Expt 3: 15 mm.min^{-1} at first die, increasing in proportion to reduction in area.	Die cooled by dry ice in methanol

Die No	Die Size mm	Reduction in Area %	True Strain
1	8,06	19,8	0,223
2	6,86	27,6	0,543
3	5,94	25,5	0,831
4	5,20	23,4	1,105
5	4,57	22,6	1,364
6	4,06	21,2	1,582
7	3,65	19,2	1,802
8	3,37	14,5	1,977
9	3,11	14,8	2,131
10	2,87	15,1	2,286
11	2,67	13,5	2,430

During these trials, it was noted that the measured drawing forces were significantly higher than those expected at normal drawing speeds, and this was attributed to poor lubrication (Honer, 1985). At these speeds, the wire apparently proceeds through the die in small discrete steps (which are difficult to detect) and not smoothly. This can give rise to non-uniform wire properties, and, more importantly, to surface damage of both the wire and the die. The internal stress field after drawing is also likely to be unrepresentative of production material.

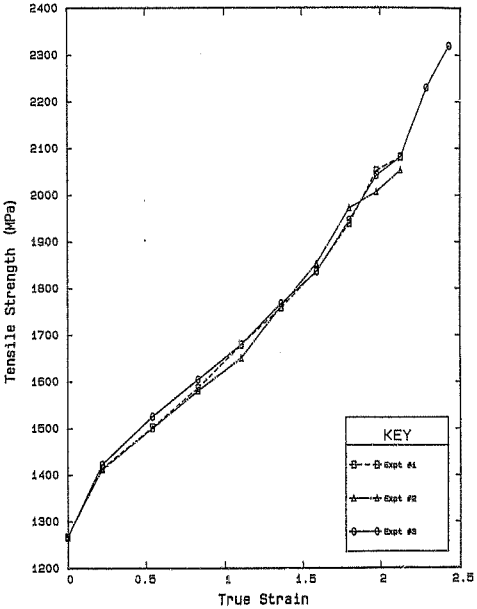


Fig 3.1 Work Hardening Curves from Slow Drawing Expts

Experiments were therefore performed with certain different lubricants in an attempt to solve this problem. These lubricants included molybdenum disulphide, wool grease and graphite, and various mixtures of these.

In order to work effectively as a boundary lubricant, molybdenum disulphide (MoS_2) powder should, be bonded to the steel surface by hard rubbing or "burnishing". This requirement added considerably to the difficulties of using molydenum disulphide, so that an attempt was also made to use the lubricant without the burnishing operation.

A commercial plain carbon (0,8% C) 4 mm diameter feed material was prepared, and the different lubricants applied before drawing at various speeds. The drawing force, which is affected by the friction conditions in the die, was monitored during two passes. The results are listed in Table 3.2, from which it is apparent that no improvement to the situation could be made.

TABLE 3.2 : SUMMARY OF RESULTS FROM DRAWING LUBRICANT TRIAL

Lubricant	Die Size mm	Drawing Speed $mm.min^{-1}$	Drawing Force $\pm 0,2$ kN
Phosphate+Borax coated			
Calcium soap	3,408	20	7,4
Calcium soap		65	7,5
Ca soap + Graphite		65	7,8
Ca soap + Graphite		200	7,9
Moly disulphide (MoS_2)	3,408	20	7,5
MoS_2 + Ca soap			7,2
Wool grease (WG)+Graphite			7,35
WG + Graphite + Ca Soap			7,35
MoS_2 Anti-Scuff Spray			7,75
MoS_2 only	3,408	200	7,4
MoS_2 + Ca Soap			6,9
WG + Graphite			7,5
WG + Graphite + Ca Soap			7,2
Clean Wire			
Calcium soap	3,408	20	7,1
Ca Soap + MoS_2 (burnished)			7,35
MoS_2 only			7,8
WG + Graphite			7,5
Calcium Soap	2,902	50	5,9
Ca Soap + MoS_2 (burnished)			5,8
Ca Soap + MoS_2			6,0
MoS_2 only			6,7

The traditional calcium soaps generally yielded the lowest drawing forces, while molybdenum disulphide was only found to be beneficial in combination with the calcium soap.

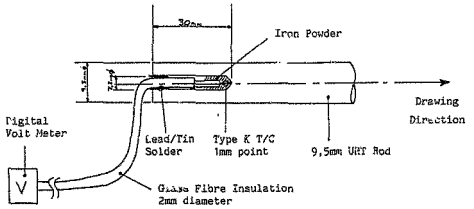
Both wool grease and graphite are noted boundary lubricants in certain applications. For instance, wool grease is added to rope lubricants for this purpose. However, it is thought that the friction conditions within the die in this experiment were too severe for either of these products to be effective.

The temperature which the wire reaches during the drawing process is of considerable importance to the ageing rate, and hence to the ductility of the wire after drawing. Thus, the temperature in the bulk and surface of the wire during slow drawing was estimated by inserting a 1 mm K-type thermocouple into holes drilled into the rod as shown in Figure 3.2. Temperatures were only measured for the first two passes of a 9,5 mm rod drawn to 8,05 mm and 6,86 mm, for practical reasons. Due to the large amounts of deformation experienced, it can be considered that the temperature rise in the first two passes is probably larger than in subsequent passes.

The results from this experiment are summarised in Table 3.3. It is apparent that the maximum temperature measured in the wire was just 34°C above ambient at the wire centre. In addition it was noted that the residence time at these temperatures was of the order of seconds, and it can therefore be concluded that no significant ageing was likely to occur during drawing under these conditions (see Table 2.1).

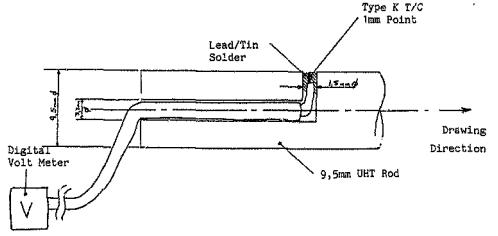
TABLE 3.3 : SUMMARY OF TEMPERATURE MEASUREMENTS OF THE WIRE IN THE DIE DURING SLOW DRAWING

Sample No.	Wire Bulk or Surface	Die Size mm	Draw Speed mm/min	Draw Force kN	Max Temp Rise °C	Max Temp Achieved °C
1	Bulk	8,09 6,87	20 20	37 29	7,8 10,4	25 31
2	Bulk	8,09 6,87	100 100	37 30	31,1 33,8	48 55
3	Surface	8,09	100	35	30,0	52



A: BULK WIRE TEMPERATURE MEASUREMENT

Figure 3.2 : Schematic of Wire Temperature Measurement Apparatus



B: WIRE SURFACE TEMPERATURE MEASUREMENT

Due to the inherent variability in the mechanical properties of wire samples, it is always necessary to repeat tests in order to ensure that the results obtained are statistically valid. A large amount of wire sample is therefore required for any ageing investigation, where a multiplicity of time/temperature ageing treatments are tested. Thus, the slow drawing technique of sample preparation was found to be impractical for a project of this duration, and an alternative drawing method was therefore required. The unrepresentative friction conditions with respect to those normally encountered also prompted the rejection of the slow drawing wire preparation technique.

A single-hole, commercial drawing machine (shown in Plate 3.3) was therefore employed. This machine was capable of drawing speeds ranging from about 1 $m.min^{-1}$ to 4 ms^{-1} at no load, but the actual values obtained depended somewhat on the drawing forces required. For instance, large drawing forces necessitated that the speed be increased to avoid stalling, and little control of the actual speed was possible. At the smaller wire feed sizes, speed control to within 0,2 $m.min^{-1}$ was attainable due to the lower drawing forces involved.

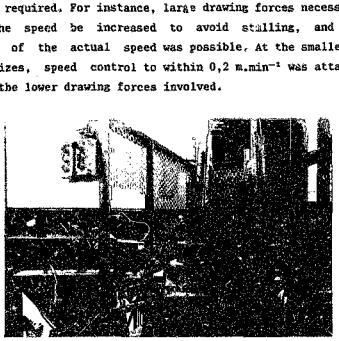


Plate 3.3 : Commercial Single-Hole Wire Drawing Machine

A direct water cooling attachment was used to cool the outgoing wire, and this is shown in Plate 3.4. This cooler was similar in principle to that used by Nakamura et al (1976) (see Figure 2.20), but in the present study, water cooling of the die itself was provided separately.

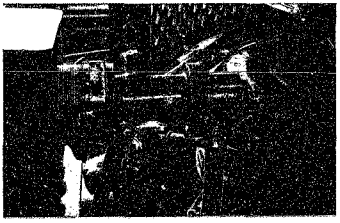


Plate 3.4 : Direct Water Cooling Apparatus

The cooler operates as follows. Water at the local mains pressure is fed into the upper inlet of the cooler, with a flow rate of about 20 $lmin^{-1}$ while air is injected into the right-hand orifice. The air mixes with the water, ensuring turbulent coolant flow conditions for the maximum heat transfer rate from the wire. The effective cooling length was 230 mm.

The air/water mixture exits through the large down pipe at the centre, but part of the air blast exits at the wire outlet, and this helps to wipe the excess surface water from the wire.

Comparative tests between the slow and fast drawing apparatus showed that there was no apparent disadvantage as regards either the as-drawn properties or the ageing behaviour of wire drawn using the commercial machine, as long as efficient post-die cooling was maintained. The number of tests was, however, limited.

The drafting schedules used in all the drawing trials were designed to reflect a traditional taper draft, as might be used in production conditions. Equivalence in terms of the reduction in area per pass was maintained when the wire feed size was changed from 9 or 9,5 mm to 4 mm.

After drawing, the wire was straightened manually and divided into lengths of appropriate size for the test methods envisaged, and stored in a domestic freezer maintained at -22°C to inhibit the occurrence of ageing during storage. Storage under liquid nitrogen, as used by Yamada (1976) was investigated, but was found to be prohibitively expensive.

3.2.4 Ageing Procedure

Artificial ageing treatments were carried out using a constant temperature bath containing silicon oil. The bath was maintained at the desired ageing temperature by an RKC model PF-62 0-200°C temperature controller (accurate to within 1°C) connected to a type J thermocouple immersed at a central location in the bath (Plate 3.5). The oil temperature was checked using a mercury-in-glass thermometer, while agitation of the fluid to restrict temperature gradients was provided by two laboratory overhead stirrers.

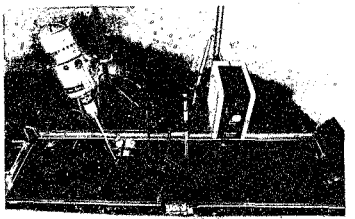


Plate 3.5 : Silicon Oil Bath Used for Ageing Treatments

Silicon oil was selected for the heating medium due to its low viscosity at the desired temperatures, and due to its high temperature stability at up to 200°C.

The wires were aged in batches, and always in the same region of the oil bath to ensure consistent ageing treatments. The wires were suspended in wire cages at the mid-level of the bath. Care was taken to ensure that bundles of wires were bound loosely to allow free circulation of the oil around individual wires.

On completion of the ageing treatment, the wires were quenched into a bath of trichloroethylene at room temperature. The organic solvent helped to remove residual silicone oil from the wire surface, as well rapidly cooling the wire. Aged samples were stored at -22°C prior to testing.

3.3 WIRE TESTING

Wire testing procedures have been described in some detail in Section 2.4.4, and their applications will therefore not be discussed in detail here.

Appropriate mechanical tests for the detection and measurement of ageing should have the following qualities:

1. a continuous variation in the measured property throughout all stages of ageing;
2. sensitivity (i.e. magnitude of the response); and
3. repeatability and reproducibility.

Ideally, the test would also be non-destructive, so that the ageing response of any one sample could be measured continuously thereby eliminating the variance between samples. However, this is at present impossible to achieve with a mechanical test.

Over the course of the project, testing took the form of mechanical tests (i.e. tensile testing, torsion testing and shear testing) as well as electrical resistivity measurements. The appropriate methodology will be described below.

Initially, the ultimate tensile stress (UTS) was the only property measured, since past experience had ruled out the measurement of the elastic properties (i.e. proof stress) on the basis of repeatability of the results, while the shear and torsion tests were considered to be too qualitative in nature. Subsequent difficulties with the measurement of the UTS (linked to the tendency of the wires to break in the grips) forced the re-evaluation of the shear test, and of the determination of the tensile proof stress at a later stage.

3.3.1 The Tensile Test

All tensile testing was performed using an Instron hydraulic screw-driven universal tensile test machine, equipped with a 100 kN load cell provided with full scale ranges of 2, 5, 10, 20, 50 and 100 kN. Generally, tensile tests were repeated four or five times, to obtain more significant results.

When required, extension measurements were made using an Instron 50 mm clip-on strain gauge extensometer with a 5 mm full scale deflection (Plate 3.6) which connected directly into the processing unit. The load/extension curve could then be plotted directly on the x-y plotter built into the machine.

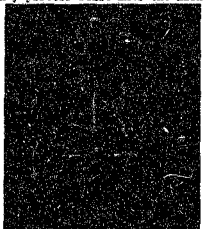


Plate 3.6 : Apparatus for the Measurement of Load-Extension Data in the Tension Test.

One of the biggest problems associated with the determination of the proof stress resulted from the shape of the load/extension curve, which rarely demonstrated a true "elastic" portion, especially in freshly drawn (i.e. unaged) material. Although the wires were manually straightened as accurately as possible, it was proposed by Reynolds (1986) that the curvature in the initial part of the load/extension record was due in part to the residual curvature of the wire itself.

A small jig was designed and built to straighten the wire in the tensile machine prior to attaching the extensometer, and this was tested against normal wire samples. However, no advantage was found with using the jig, and it appears therefore that the observed non-linearity was due to an inherent wire property.

To facilitate the analysis of the load/extension curves to determine the proof stress, a Hewlett-Packard model HP 85 microcomputer was connected to the Instron machine via an appropriate analogue to digital converter. Computer programmes were written to log the load/extension data directly from the test, and to store this data on tape. Typically, about 250 data points were stored from each test. The programme listings are included in Appendix A.

After testing a batch of wire, a second programme was called to analyse the test data for the (true) 0,1 and 0,2% proof stress. The elastic slope was estimated automatically using a least squares technique to fit a straight line to the first 75 points recorded, although provision was made to adjust this number if required. This method was found to work very well, and only rarely was it found necessary to re-analyse a test result using different points to estimate the elastic slope. An example of the output from the analysis routine is shown in Figure 3.3.

Reynolds (1980) has shown that ageing of steel wires tends to decrease the work hardening rate in the tensile test. It was decided to investigate this effect in order to determine whether or not this phenomenon could be quantified, and hence could be used as a parameter to detect ageing. Reynolds' method was used to investigate this property, in the following way.

The work hardening rate is defined by an expression of the form:

$$\sigma_T = A.\epsilon_T^{n(a)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.1)$$

where n(a) is the apparent work hardening exponent;
ϵ_T is the true strain;
σ_T is the true stress (MPa);
and A is a constant.

The value of n(a) can therefore be derived from the slope of the graph of log stress vs log strain, in the plastic region.

DATA SUMMARY FOR 451503

Wire Diameter (mm)	1.221
Gauge Length (mm)	50
U.T.S (Eng) (MPa)	2479
U.T.S (True) (MPa)	2519
No of Readings	178

PROPORTIONAL LIMIT

Point No.	86
STRAIN	.935 %
STRESS	1789 MPa

ELASTIC SLOPE

E-mod	196 GPa
Std Error	18 MPa

0.1% P.S.= 2188 MPa

0.2% P.S.= 2380 MPa

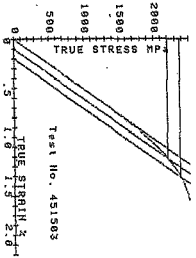


Figure 3.3: Example of Computer - Generated Output from the Stress/Strain Analysis.

Sample: 0,6% Carbon commercial steel wire 1,22 mm; aged at 130°C for 5 minutes.

X-MIN -3.58344530092E-2
X-MAX .528036660496
Y-MIN 7.51627551027
Y-MAX 7.92865795225

Point Nos. 90 To 170

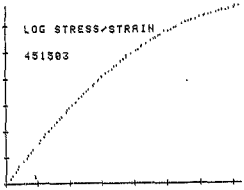


Figure 3.4: Expanded Plastic Region of the Stress/Strain Data in Figure 3.3 (after taking logarithms).

The apparent work hardening exponent, however, is calculated using the sum of the elastic and the plastic strains. The true work hardening exponent should only consider the plastic strain. Thus, if ϵ_p is the elastic strain at the proportional limit:

$$\sigma_T = A.(\epsilon_T - \epsilon_p)^{n(t)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.2)$$

and the true work hardening exponent, n(t), is then given by the slope of the log stress vs log $(\epsilon_T - \epsilon_p)$ curve.

In order to determine the value of ϵ_p, the true proportional limit must be derived, using the point at which the equations describing the elastic properties and the plastic properties intersect. Thus:

$$A.\epsilon_p^{n(t)} - E.\epsilon_p - C = 0 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.3)$$

Computer programmes were written to perform the log stress/log strain analyses automatically. However, it was found to be impossible to obtain a log stress/strain plot which showed linear behaviour in the plastic region (Figure 3.4). This implied that the apparent work hardening exponent (required in Equation 3.3) cannot be represented by the exponential equation (3.1). It was therefore found to be impossible to determine the true work hardening rate.

The reasons why freshly drawn wire (in particular) behaves in this manner is unclear, but may arise from the internal stresses in the wire after drawing. Reynolds' work was performed on spring wire which undergoes stress relieving heat treatments in the course of processing, and this may account for his success with this method.

3.3.2 The Shear Test

The shear test which was described briefly in Section 2.4.4, attempts to simulate the severe contact forces operating on a rope wire in service by placing a side force on the wire during the tensile test. It is evident that the shear test tends to measure the tranverse properties of a wire, and that these will differ significantly from the tensile properties due to the anisotropic nature of the microstructure.

The side force was applied using a hydraulic ram, which forces the wire against an anvil with a fixed radius, as shown in Figure 3.5. The magnitude of the side force required was determined from the empirical results obtained at British Ropes Ltd. (Stephenson, 1983), and was dependent only on the wire diameter. The side force was monitored using a pressure gauge on the oil feed line. The ram was applied to the wire at a point midway on the gauge length (which by convention is 100 mm).

The wire is loaded progressively at a constant crosshead speed. A load vs elapsed time record is made of the portion of the record which includes the attainment of maximum load and final failure. The shear elongation is then estimated from the chart to be the total extension to failure after maximum load has been achieved, and this is essentially the extension at the neck.

Wires which have been severely embrittled (e.g. by strain ageing or overdrawing) may not show any extension after the maximum load has been achieved (i.e. they do not exhibit any ductility). During the course of ageing, the maximum load recorded is found to increase, until the wire becomes embrittled, when the wire appears to break prematurely. Such wires having zero shear elongation usually exhibit a characteristic oblique fracture at approximately 45° to the tensile axis (see Plate 3.7). Once a wire has attained zero shear elongation in the test, the further progress of an embrittling effect such as ageing cannot be monitored.

Plate 3.7 : A Typical "Shear" Fracture in the Shear Test (Sample is a production 3,05mm wire) 20x

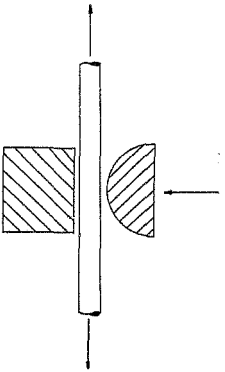


Figure 3.5 : Shear Test Configuration.

In an attempt to extract more useful (i.e. quantitative) information from the shear test, a record was made of the complete load/extension curve, and estimates of the strain energy to failure were made using the HP 85 microcomputer. Unfortunately, the variability of the test results precluded the use of this technique to follow the progress of strain ageing.

The Mining Equipment Research Unit of the CSIR* has investigated a test similar to the shear test described above, in an attempt to quantify the behaviour of wire in a rope.

This method applies a side force to the wire in a similar manner, but instead of using an anvil of fixed radius, a wire of identical size to the one under investigation is used, orientated at a known angle to the wire axis. The side force used is a known proportion of the wire breaking force.

In an attempt to refine the shear test, and to encourage a more gradual deterioration of properties, the CSIR shear test method was briefly investigated.

Difficulties were encountered with the setting of the side loads, since the wire size under investigation was very small (1,22 mm). The effect therefore of small variations in the side force was found to be significant with respect to the type of failure which was observed (i.e. ductile or shear). It became apparent that this test method, for this wire size, and with the equipment used, was prone to as much scatter as the standard version, and was therefore no more suitable for this project.

3.3.3 The Torsion Test

The torsion test measures the number of twists which a wire can accommodate before separation, over a gauge length which is arbitrarily defined to be equal to 100x the wire diameter. A note is also made of the appearance of the wire after the test, and of the type of fracture exhibited.

* CSIR - Council for Scientific and Industrial Research, Pretoria.

Stephenson et al (1983) and Lanner (1983) have shown that a ductile wire exhibits a square-ended fracture, with uniform deformation along the wire length. However a wire whose ductility has started to deteriorate will begin to show a seamy condition, with splitting along its length, and a fracture which is helical in appearance. A secondary fracture behind the primary break may also occur.

Although the torsion test has been described as a sensitive measure of ageing, it shares the problem of the shear test in that it is lacking in reliable and repeatable quantitative output, while it also suffers from a large variability in results.

A test was performed on 1,45 mm and 1,36 mm wire to determine the response of this wire size to ageing, by the torsion test. The material used was a commercial high carbon steel, drawn using the normal experimental drawing practice, as described previously (see Section 3.2.3). The wires were tested in the as-drawn condition, and after ageing at 100°C for 8 hours. The results of torsion and shear tests on these wires are shown in Table 3.4.

TABLE 3.4 : VARIATION IN TORSION AND SHEAR PROPERTIES AFTER AGEING AT 100°C FOR 8 HOURS.

		Torsion Test		Shear Test
Wire Diameter (d) mm	Condition	Number of Twists (on 100xd)	Fracture Type (1)	Av. Shear Elongation mm
1,445	As Drawn	40 35 40 40	A1 A1 A2 A2	0,95
1,445	Aged	30 33 27 31	A2 A2 A2 A2	0,25
1,360	As Drawn	33 37 30	A2 A2 A1	0,80
1,360	Aged	29 29 33	A2 A2 A2	0,71

Note (1): Torsion Test Quality Rating (after Haggie Rand Ltd):

A1 = No splitting; no waviness; even twisting; fracture square-ended; no secondary breaks.

A2 = As A1 but secondary fracture permitted.

B = As A2 but primary fracture may be stepped or helical.

An ageing treatment of 8 hours at 100°C can be considered to be relatively severe, as evidenced by a significant decrease in the shear elongation to failure. The number of twists to failure had also decreased, although with some scatter, but the quality of the wire fracture altered little. It is probable that the insensitivity of the fracture type in this investigation derived from the smaller than usual wire diameter, which might be expected to favour good quality torsion fractures. From this, and other tests, it was evident that the torsion test offered no advantage over the shear test for this project.

3.3.4 Electrical Resistivity

The measurement of electrical resistivity is, in essence, very simple, since the value of the resistivity is derived from the resistance of the wire, and its dimensions:

$$\rho = \frac{R.A}{l} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.4)$$

where ρ is the resistivity (Ωm);
R is the resistance of the specimen (Ω);
A is the area of cross-section (m^2);
and l is the specimen length (m).

The resistance of the sample is easily calculated using Ohm's law, from the potential drop measured along the specimen length while a constant known current is passed through. The difficulty with resistance measurement, however, is in the level of accuracy of the test method, since this will depend on a number of factors, such as:

* contact resistance at the potential measurement points;
* resolution of the potential measurement equipment;
* dimensional accuracy;
* temperature of measurement, and its stability; and
* current stability.

The chosen current must be sufficient to yield a measurable potential drop, without affecting the temperature stability of the wire. As mentioned in Section 2.4.4.5, the accuracy of resistivity tests is increased if the temperature of measurement is maintained as low as possible.

A special cooling cell was constructed to contain the wire sample, the cooling medium (liquid nitrogen), and the potential measurement leads (Figure 3.6). The wire protruded through rubber seals to enable attachment of the current leads outside the cell. Gold plated Kelvin type clips were used initially to connect the potential measurement leads to the wire, but these were later substituted by point contact devices (Figure 3.7). These allowed for more accurate placement of the gauge length, and also reduced the variance in the contact resistance.

Initial tests were performed on 3,1 mm diameter wire using a variety of currents to determine the heating effect of this factor. The results are shown in Figure 3.8, from which it is apparent that a marked heating effect does occur as the current is increased beyond about 0,1 A. A current of 1 A was however required for future test work, in order to give sufficient sensitivity for measuring the potential drop.

Attempts were then made to determine the ageing behaviour of steel wires, as measured by the electrical resistivity. Wire of 3,1 mm was manufactured on the tensile test machine (see Section 3.2.2) from 9,48 mm diameter rod, at a constant speed of 50 $mm.min^{-1}$. The finished wire had a drawn tensile strength of 2135 MPa, after drawing to the true strain of 2,23. The wire drawn on the tensile machine had the advantage of being straight as drawn, and therefore required no straightening before insertion into the cooling cell. This eliminated one source of error, since the plastic deformation due to straightening could be expected to affect the measured value of the resistivity.

Averaged results from this experiment are shown in Figure 3.9. The measured wire behaviour was never consistent, and prone to significant error. This was observed even though the tensile properties could be seen to be increasing at a steady rate during ageing. Further work on this apparatus confirmed this

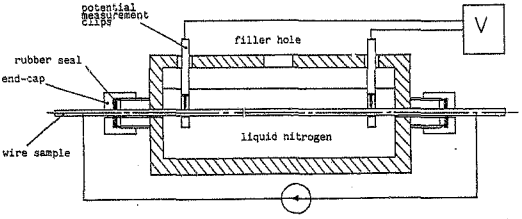


Figure 3.6 : Schematic of Cooling Cell for the Measurement of Resistivity.

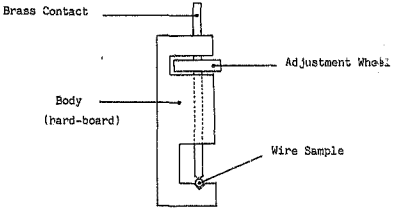


Figure 3.7 : Point Contact Measurement Devices for Electrical Resistivity Tests.

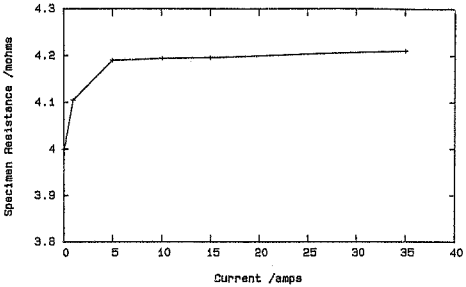


Fig 3.8: Increase in Resistance with Test Current

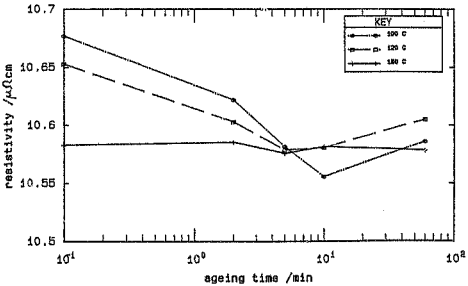


Fig 3.9: Resistivity Ageing Curves - 0.8% carbon steel

trend. It was therefore concluded that the test technique used in the present study was inadequate and work using this apparatus was therefore abandoned.

3.4 SUMMARY

This chapter has described the efforts made to establish reliable test procedures, from the preparation of materials, through testing, to final data analysis.

The materials used initially were commercial grade plain carbon steels, prepared for drawing under factory conditions. A wire feed size of 4 mm was selected for reasons of better control of the wire preparation procedures.

The slow wire drawing apparatus was successful in operation, but for practical reasons, had to be withdrawn. These reasons included the amount of time required to prepare wire sample, and more importantly perhaps, the comparability of slow-drawn wire with that produced under conditions of more ideal lubrication.

All wire samples were therefore subsequently prepared on a commercial single hole drawing machine, equipped with a direct water cooler.

Artificial ageing of the drawn wire was carried out in a silicon oil bath, the temperature of which was closely controlled. Initially the progress of ageing was monitored using the tensile strength only, for reasons of reproducibility and smoothness of the response to ageing. However, at a later date, the shear test was introduced as a regular wire test, while the tensile proof stress was also determined. For this work, the torsion test was considered to offer no advantage to the shear test as an alternative to the tensile test.

The measurement of the change in electrical resistivity with ageing was investigated, but after some initial work, was abandoned in favour of the more relevant and measurable mechanical properties.

CHAPTER FOUR : INVESTIGATIONS INTO THE EFFECT OF VARIOUS PARAMETERS ON STRAIN AGEING.

4.1 INTRODUCTION

For any scientific experiment, some knowledge of the behaviour of a standard product is required before the effects of changes to the experimental procedure can be determined. A large volume of work was therefore carried out on commercial wire (with typical compositions) in order to characterise its behaviour.

After the establishment of base data for the standard material, preliminary investigations into the ageing phenomenon were carried out. These included studies of the effect on the rate of ageing of total strain, wire size, carbon and nitrogen contents, and a low temperature anneal applied prior to drawing.

Subsequently, the effect of nitrogen was studied in more detail, and the influence on the rate of ageing of certain other interesting parameters (the pearlite interlamellar spacing and the silicon content) was determined using specially prepared steels, manufactured under vacuum. From this work, it was hoped that the ageing mechanism would be identified, and a solution to the problem proposed, which could then be investigated further.

This chapter describes the above work in some detail, and the conclusions which could be drawn from the results obtained.

4.2 PRELIMINARY INVESTIGATIONS ON COMMERCIAL PLAIN CARBON STEELS

4.2.1 Conventional High Carbon Steel Wire

For reasons described in Section 3.2.1, a relatively small starting wire size before drawing of 4 mm was considered to be beneficial from the point of view of maintaining close control of the experimental conditions. A sample of patented 4 mm high carbon steel wire was supplied by the Haggie Rand Ltd factory, for the initial test work. This material had the composition, and microstructural features described in Table 4.1.

TABLE 4.1 : COMMERCIAL 0,8% CARBON STEEL FEED MATERIAL

	C %	Mn %	Si %	P %	S %	Total N %
Chemical Analysis	0,83	0,81	0,21	0,015	0,022	0,0067

Microstructure: 100% Pearlite
Minimum Observed Pearlite Spacing: 65 nm
ASTM grain size: 7

Tensile Strength: 1335 - 1380 MPa
Constriction: 38 - 44 %
Average Diameter: 3,98 mm

Drawing of the feed material into wire was performed on the single-hole drawing machine described in Section 3.2.2, using the die set given in Table 4.2, which was considered to be roughly equivalent to a typical works drafting schedule. The final wire size was 1,36 mm, at a drawing strain of 2,16.

The direct water cooler with a cooling length of about 230 mm, was used to cool the wire leaving the die. A constant drawing speed of 2,1 $m.min^{-1}$ was used for each pass, so that the immersion time in the cooler was about 7 second. This was found to be more than sufficient to cool the wire to room temperature before the wire exited from the cooler.

The drawn wire was aged at various temperatures in the range 80°C to 130°C using the method previously described in Section 3.2.3, and subsequently tensile tested for the UTS only.

TABLE 4.2 : DIE SET FOR 4 mm FEED MATERIAL

Die Number	Die Size mm	Reduction in Area %	True Strain
1	3,408	27,4	0,320
2	2,906	27,1	0,639
3	2,530	24,2	0,916
4	2,208	24,0	1,190
5	1,955	21,5	1,432
6	1,749	20,0	1,655
7	1,570	21,5	1,896
8	1,441	15,9	2,071
9	1,360	10,8	2,158

A plot of the average increase in UTS with time at the ageing temperature is shown in Figure 4.1. It can be seen that, if the increase in the tensile property is plotted against the logarithm of the ageing time, then the ageing process can be described by a straight line (in the regime covered by the ageing treatments applied). The temperature of ageing alters the "offset" of the tensile ageing curves and thus the kinetics of the ageing process are accelerated by an increase in the ageing temperature, as would be expected.

The activation energy for the reaction to proceed was estimated by using the cross-cut method (as used by Yamada, 1976) on the tensile ageing curves. Thus a temperature/time relationship is derived for the reaction to proceed to some arbitrarily defined point, determined by some change in a measured property. In this case increases in the measured UTS of 50 MPa and 100 MPa were used, and the ln(time) vs reciprocal of the ageing temperature graphs are shown in Figure 4.2. This essentially follows a recommended practice for estimating the mechanism for a reaction (Perry and Chilton, 1973), by examining the rate constant after a small amount of the reaction has proceeded.

The graphs in Figure 4.2 are approximately straight lines; this indicates that the ageing process is thermally activated, and can be described by an Arrhenius-type rate law:

$$\text{Rate} = A.\exp\frac{(-Q)}{(R.T)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(4.1)$$

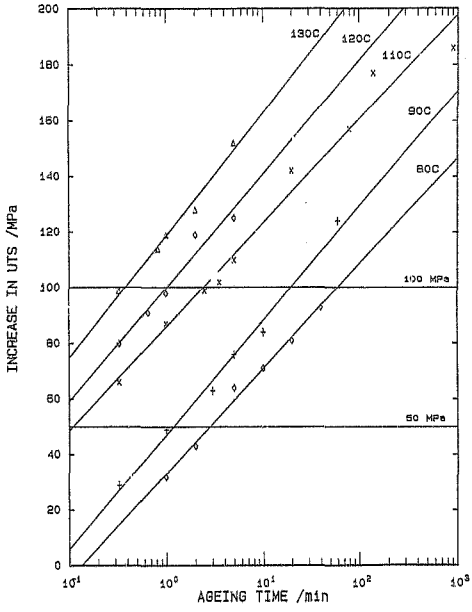


FIGURE 4.1 : THE AGEING BEHAVIOUR OF 1,36mm 0,8% CARBON COMMERCIAL STEEL WIRE MEASURED BY THE INCREASE IN THE UTS.

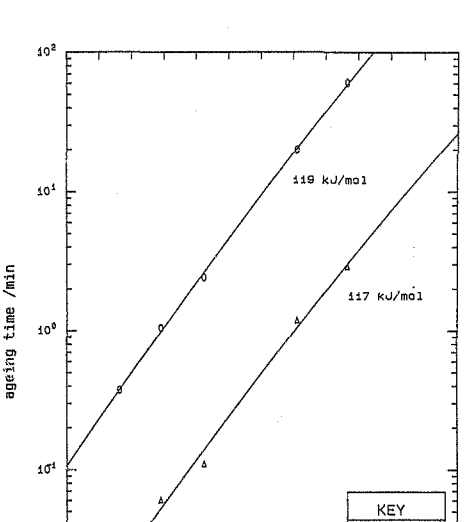


FIGURE 4.2 : THE ACTIVATION ENERGY REQUIRED TO INCREASE THE MEASURED UTS BY A GIVEN AMOUNT (from figure 4.1).

or:

$$\ln(\text{time}) = \ln(A) + \frac{Q}{R}\cdot\frac{1}{T} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4.2)$$

where Q = activation energy ($kJmol^{-1}$);
R = gas constant (8,314 $Jmol^{-1}K^{-1}$);
T = absolute temperature (K);
A = reaction constant.

The slope of the line therefore yields the activation energy when multiplied by the gas constant. The activation energy represents the minimum energy required for the reaction (or process) to proceed, and thus characterises the rate-controlling step. The reaction constant, A, represents, in chemical terms, the frequency factor which can be considered to be a "collision frequency" between two reacting species; this is dependent on the square root of the (absolute) temperature, on the concentration of the reacting species, and on the mean diffusion distance. This term is also, therefore, time-dependent.

The activation energies for 50 MPa and 100 MPa increases in UTS were found to be 117 $kJmol^{-1}$ and 119 $kJmol^{-1}$ respectively. These values compare exactly with that determined by Yamada (1976) for the second stage of ageing (see Section 2.4.2) from electrical resistivity measurements.

The kinetics of the ageing reaction can be investigated by applying an equation of the form:

$$n = 1 - \exp(-k.t^m) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4.3)$$

where n = fraction of the reaction progressed;
k = reaction constant (note: not equal to A, above);
t = ageing time;
and m = rate or time exponent.

This empirical equation was proposed by Johnson and Mehl (1939) to describe reaction kinetics on the basis that the decrease in the precipitation rate with time will be proportional to the fraction already precipitated.

The temperature dependence of k, the reaction constant, corresponds to the energy of activation for the reaction. The rate exponent, m, however, describes the reaction kinetics; thus it is temperature dependent, and is also affected by the concentrations of the reacting species. The rate exponent can therefore be considered to be equivalent to the pre-exponential term in Equation 4.1.

The rate exponent is determined from the slope of the plot of ln.ln(1/(1-n)) vs ln(t). This procedure is shown for some temperatures in Figure 4.3, and the results are shown in Table 4.3. For the calculation, an estimate of the maximum increase in the UTS was required, and this was taken to be 200 MPa. The significance of these results will be discussed in Section 6.3.1

TABLE 4.3 : VALUES OF THE RATE EXPONENT DERIVED FROM FIGURE 4.3

Ageing Temperature °C	Rate Exponent (m)
80	0,234
110	0,247
120	0,256
130	0,265

This experiment was repeated using a different 0,8% carbon feed material (with the composition given by Table 4.4) also supplied by Haggie Rand Ltd. A new starting material was used since the significant variation in the tensile properties of the original material along its length was found to be repeated in the finished wire. The new material had more consistent properties, and therefore gave more reliable results.

The results from the repeat experiment are given in Figure 4.4, which shows the progress of ageing at 100°C over an extended period, as measured by the tensile test. The rate exponent was determined to be 0,275 (see Figure 4.5), which agrees reasonably well with the results in Table 4.3.

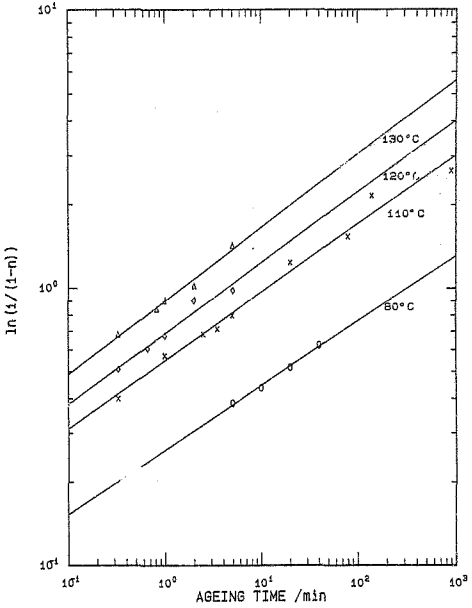


FIGURE 4.3 : DETERMINATION OF THE RATE EXPONENT (m) FOR THE RESULTS GIVEN IN FIGURE 4.1.

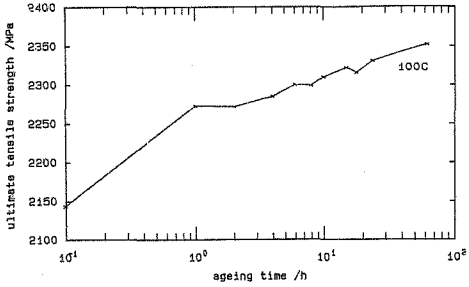


FIGURE 4.4 : THE AGEING BEHAVIOUR OF THE 1,36mm 0,8% CARBON REPLACEMENT COMMERCIAL STEEL WIRE, MEASURED BY THE TENSILE TEST.

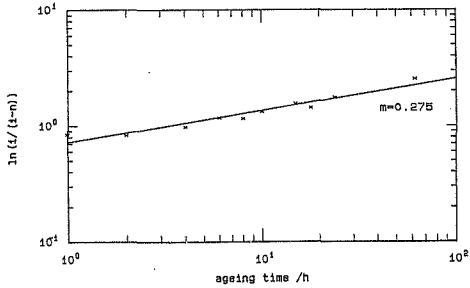


FIGURE 4.5 : DETERMINATION OF THE RATE EXPONENT (m) FOR THE RESULTS GIVEN IN FIGURE 4.4.

TABLE 4.4 : NEW COMMERCIAL 0,8% CARBON FEED MATERIAL

	C %	Mn %	Si %	S %	P %	Total N %
Chemical Analysis	0,80	0,74	0,26	0,012	0,024	0,0059

Microstructure: 100% Pearlite
Minimum Observed
Pearlite Spacing: 65 nm
ASTM Grain Size: 6

Average Diameter: 3,95 mm
Tensile Strength: 1306 - 1317 MPa

4.2.2 The Effect of Drawing Strain

The drawing strain was investigated using the feed material defined in Table 4.4, and the drawing conditions specified in Section 4.2.1. Wire was sampled at 2,20 mm (true strain = 1,17), at 1,58 mm (strain = 1,83), and at 1.45 mm (strain = 2,01), and compared with the 1,36 mm diameter material described above.

Ageing treatments were performed at 100°C over a limited range of times at temperature. Figure 4.6 shows the results of the absolute increase in UTS vs ageing time, for each of the wires considered, while Figure 4.7 shows an attempt to normalise the data by relating the increase in UTS to the as-drawn UTS. The drawing strain, as might be expected, markedly affects the rise in UTS (Figure 4.6), but no simple correlation appears to exist between the increase in UTS on ageing and the as-drawn UTS (Figure 4.7); nor is there a simple correlation to the amount of drawing strain.

The slopes of the increase in UTS vs ln(time) graphs are similar at each strain, and this implies that the rate constant does not change significantly with strain, and thus the reaction mechanism is probably also unaffected. Drawing strain does, however, accelerate the reaction kinetics, as might be expected.

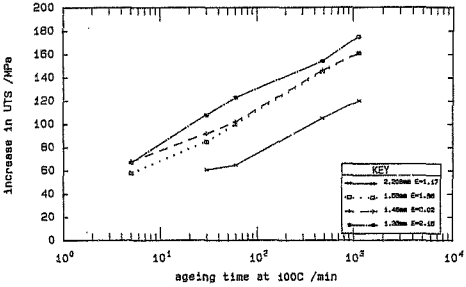


FIGURE 4.6 : THE EFFECT OF DRAWING STRAIN ON THE RATE OF AGEING MEASURED IN THE TENSILE TEST.

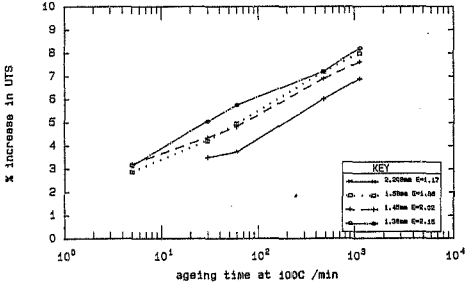


FIGURE 4.7 : THE EFFECT OF DRAWING STRAIN ON THE RATE OF AGEING AFTER NORMALISING THE RESPONSE IN THE TENSILE TEST.

Shear tests were also performed on the aged wires, and the variation in the shear elongation (i.e. total elongation after maximum load) with ageing time is shown in Figure 4.8. These results showed an interesting anomaly. The shear elongation might be expected to decrease more rapidly during ageing as the drawing strain increases, in line with the increase in UTS. This is indeed the case at 2,2 to 1,45 mm. However, the final wire size of 1,36 mm showed a significantly improved ageing response, with little drop in measured ductility throughout the ageing treatment.

This phenomenon was possibly a consequence of the empirical nature of the test, such that comparisons between wires of different diameters are not necessarily valid. Alternatively, it may be linked to the drafting schedule. The die set used in this experiment was specified in Table 4.2; and the reduction in wire cross-section in the penultimate pass (1,45 mm) was 15,9%, while that in the final pass was just 10,8%. The reduction in area at the final or penultimate pass is known to affect the finished wire properties, as will be discussed below.

Further investigations were therefore performed using the same feed material, and a similar die set, with the addition of a 1,33 mm final die. Four wire samples were produced under nominally identical conditions with different final reductions in area, as defined in Table 4.5. The wire was aged at 100°C for up to 62 hours, and tensile and shear tests were performed. The results are illustrated graphically in Figures 4.9 and 4.10.

TABLE 4.5 : INVESTIGATION INTO THE EFFECT OF REDUCTION AT THE FINAL PASS - SPECIFICATION OF WIRE SAMPLES

Sample Number	Wire Diameter mm	True Strain	No of Passes	Final Redn %	Drawn UTS MPa	Shear Eloug. mm
1	1,441	2,017	8	15,9	2115	0,95
2	1,360	2,133	9	10,8	2144	0,81
3	1,330	2,177	10	4,4	2164	0,77
4	1,330	2,177	9	14,8	2173	0,79

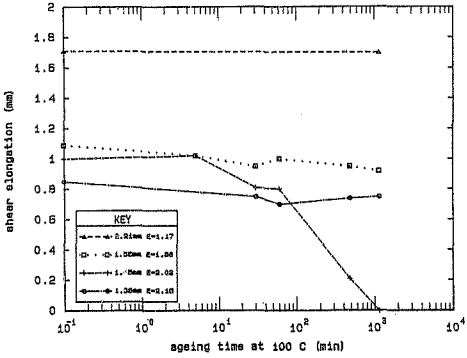


FIGURE 4.8 : THE EFFECT OF DRAWING STRAIN ON THE RATE OF AGEING MEASURED BY THE SHEAR TEST.

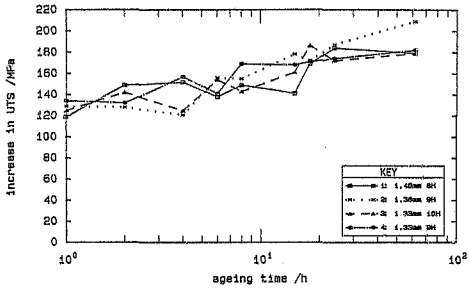


FIGURE 4.9 : THE EFFECT OF THE DRAFT SCHEDULE ON THE RATE OF AGEING MEASURED BY THE TENSILE TEST.

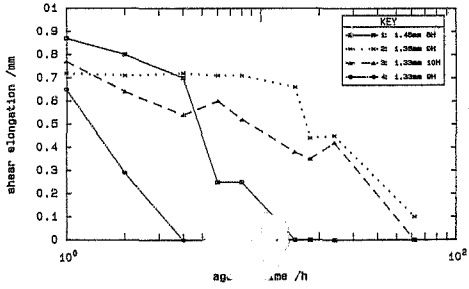


FIGURE 4.10 : THE EFFECT OF THE DRAFT SCHEDULE ON THE RATE OF AGEING MEASURED BY THE SHEAR TEST.

The tensile results here showed a larger degree of scatter than was observed previously, and this was attributed to a new set of grips for the Instron tensile machine. These grips were found to cause consistent wire failures in the gripped length, at loads which were sometimes below the true breaking load of the specimen (i.e. with little or no necking at fracture). The problem could not be solved simply since a replacement set of grips was not available.

It is apparent from Figure 4.9 that the reduction of area in the final pass did not affect the tensile properties significantly. However, the shear test (Figure 4.10) revealed a marked deterioration in the transverse properties after ageing. Wire which had undergone a small reduction in the final pass was found to retain its as-drawn shear properties for a significantly longer period of time (by an order of magnitude) than wire finished at a higher reduction, when tested at the same diameter.

Recent work by Sadok and Kowalski (1986) has shown that the use of a small reduction in area (of about 1-5%) in the final die can cause an appreciable fall in the internal stresses of the wire, and they showed benefits in terms of the fatigue strength of the finished wire. It is therefore possible that a less severe internal stress distribution is beneficial to the ageing properties of wires, as measured by the shear test.

Alternatively, as shown by Equation 2.12 (in Section 2.4.3.2), a lower reduction in area will have the effect of increasing the average effective strain rate. Increasing the rate of strain in a deformation process may be considered to be equivalent to lowering the temperature of deformation (in terms of thermally activated deformation), so that it is not unreasonable to assume that the effect is similar to increasing the efficiency of wire cooling during drawing (see Section 2.5).

It is difficult to see how the relative effects of these parameters can be separated. Further work in this area was, however, curtailed because of time constraints.

4.2.3 The Effect of Carbon Content

Since it is generally presumed that the phenomenon of strain ageing occurs in the ferrite phase of a pearlitic steel, it might be supposed that the rate of ageing, or at least the maximum extent of ageing, might increase if the volume fraction of ferrite were increased. A feed material of lower carbon content (0,70%) was therefore procured for investigation.

This material had the chemical analysis and microstructural features described in Table 4.6. The wire was drawn using nominally identical conditions to those described previously, into 2,2 mm and 1,36 mm diameter wire. The finished wire size size of 2,2 mm was equivalent to a drawing strain of 1,17, which was similar to that most often used by Yamada in his 1976 work.

The wire was aged at temperatures between 80°C and 150°C for various times, and were tensile tested only. The increase in UTS vs ageing time is shown in Figure 4.11.

TABLE 4.6 : COMMERCIAL 0,7% CARBON FEED MATERIAL

	C %	Mn %	Si %	S %	P %	Total N %
Chemical Analysis	0,71	0,70	0,30	0,012	0,014	0,0067

Microstructure: Pearlite + 5% pro-eutectoid ferrite
Minimum Observed
Pearlite Spacing: 70 nm
ASTM Grain Size: 6

Average Diameter: 3,94 mm
Tensile Strength: 1250 MPa
Constriction: 41,3%

For the derivation of the activation energy for the reaction, the intercept at about 50% of the reaction was used. For 2,2 mm material this was estimated to be at about an 80 MPa increase in UTS, and for the 1,36 mm material at about 125 MPa. The energy of activation was determined using Figure 4.12, and the rate exponent was estimated from Figure 4.13. The results of these analyses are given in Table 4.7.

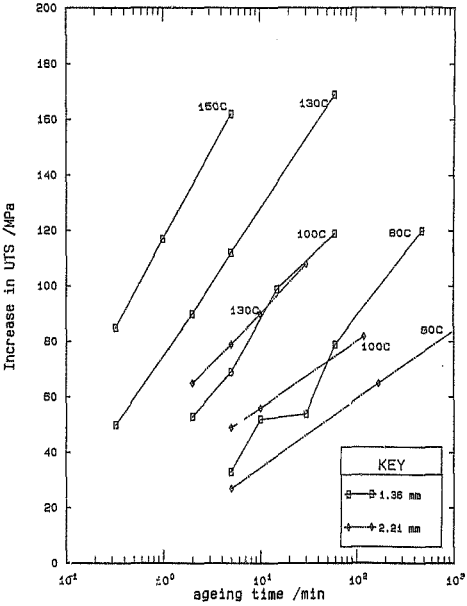


FIGURE 4.11 : THE AGEING BEHAVIOUR OF 1,36mm 0,7% CARBON COMMERCIAL STEEL WIRE MEASURED BY THE INCREASE IN THE UTS.

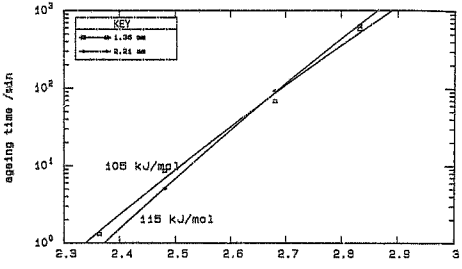


FIGURE 4.12 : DETERMINATION OF THE ACTIVATION ENERGY REQUIRED TO INCREASE THE UTS OF 0,7% C STEEL WIRE BY 100 MPa.

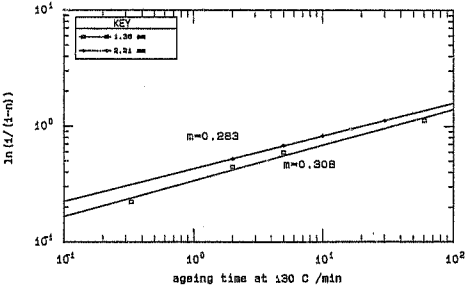


FIGURE 4.13 : DETERMINATION OF THE RATE EXPONENT AT 130°C FOR THE 0,7% C STEEL.

TABLE 4.7 : ACTIVATION ENERGY FOR AGEING OF 0,7% CARBON STEEL

Wire Diameter mm	Drawing Strain	Cross-cut UTS at MPa	Activation Energy $kJmol^{-1}$	Rate Exponent at 100°C
2,208 1,360	1,17 2,15	80 125	115 105	0,283 0,308

There is, naturally, some error in the rate exponents derived, since this value depends on the maximum change in properties achieved, and this could only be estimated on the basis of experience.

The results for the activation energy for ageing are similar to those obtained previously for 0,8% carbon material, allowing for experimental error. However, a small increase in the rate exponent is apparent over those values obtained for the 0,8% steel at the same strains (see Table 4.3).

The implication of these results is that the reaction rate is increased in the presence of a larger volume fraction of ferrite (but not by a large degree) but that the reaction mechanism is unaffected.

This phenomenon is also reflected in the increased ageing rate on drawing further from 2,20 mm to 1,36 mm. The reaction kinetics are again accelerated by the higher drawing strain, due probably to an increased dislocation density and to a finer microstructure, resulting in a reduction in the mean ferrite diffusion path required for ageing to occur.

It is not certain if a difference in the activation energies for ageing at each wire size actually exists. The implication from the Table 4.7 is that ageing will occur more rapidly at the smaller size due to lower barriers to the ageing reaction.

4.2.4 The Effect of Annealing Prior to Drawing

It was considered that the rate of ageing might be dependent on the amount of carbon held in supersaturation in the ferrite after drawing, and that this in itself might be dependent on the amount of carbon in the ferrite prior to drawing.

A supersaturation of the ferrite undoubtedly exists after the patenting process, where the final heat treatment takes place at about 540°C, followed by quenching to room temperature.

According to Reed-Hill (1973), the equilibrium carbon concentration [C] in ferrite in the presence of cementite is given by the equation:

$$[C] = 2{,}55 \times \exp\left(\frac{-40\ 585}{R.T}\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4.4)$$

where R is the universal gas constant, 8,314 $Jmol^{-1}K^{-1}$;
and T is the absolute temperature (K)

Therefore, the following ferrite carbon contents can be considered to be in equilibrium with cementite at the following temperatures:

25°C : $1{,}9 \times 10^{-7}$ wt % carbon
200°C : $8{,}4 \times 10^{-5}$ wt % carbon
540°C : $6{,}3 \times 10^{-3}$ wt % carbon.

The supersaturation of carbon at 25°C is therefore about 32000x after quenching from 540°C. By comparison, if the steel is allowed to equilibrate at 200°C, the supersaturation is only about 400x and $6{,}2 \times 10^{-3}$% carbon should be removed from solution, which is a significant reduction.

However, the ferrite after quenching is heavily dislocated, and it might be expected, therefore, that the excess carbon would precipitate on dislocations during annealing, thereby effectively removing it from solid solution in the ferrite. This is energetically more favourable than precipitation on the cementite (see Section 2.2.4). In this way, the yield stress might be expected to increase, and with it the UTS, although to a smaller extent.

A number of experiments were performed to evaluate the effect of an annealing treatment on the feed wire properties, and on the final wire properties. These were performed on both the 0,8% carbon and the 0,7% carbon materials. An annealing treatment of 200°C for 65 hours was selected, and this was carried out in a tempering furnace.

If the mean diffusion distance is taken to be $\sqrt{2.D.t}$ (Kemp, 1987) (where D is the diffusion coefficient (m^2s^{-1}), and t is the time (s)) then, from Equation 2.4 (which gives the diffusion coefficient of carbon in ferrite), the average carbon atom can travel several nanometers per minute at 200°C. Since the pearlite interlamellar spacing is of the order of 90 nm in the patented material, it is apparent that equilibrium conditions should be achieved after 65 hours at temperature.

The wire was cooled to room temperature in the furnace, prior to tensile testing to determine the change in the feed wire properties. These properties are shown in Table 4.8.

TABLE 4.8 : EFFECT OF AN ANNEALING TREATMENT OF 65h AT 200°C ON THE TENSILE STRENGTH PRIOR TO DRAWING

Sample Material	Tensile Strength /MPa	
	As received	After Annealing
0,8% C	1321	1326
0,7% C	1239	1249

The wire was drawn to a diameter of 2,20 mm using the normal die set and cooling conditions, and was subsequently aged at 100°C before tensile testing. A wire size of 2,20 mm was investigated, because this gave more consistent results in the tensile test where grip breaks were a problem with the small wire. In addition, the drawing strain of about 1,17 was equivalent to that used by Yamada (1976), so that some comparison with his results could be made. The drawn properties of the wire are shown in Table 4.9, while the ageing response of the materials is shown in Figure 4.14.

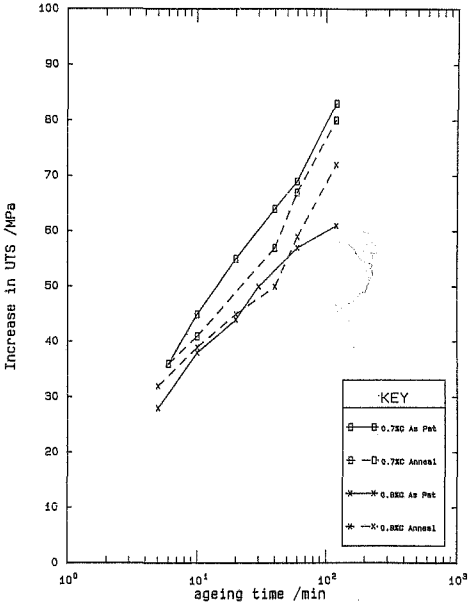


FIGURE 4.14 : THE EFFECT OF A PRIOR ANNEALING TREATMENT ON THE AGEING RESPONSE OF STEEL WIRE.

TABLE 4.9 : TENSILE PROPERTIES OF 2,20 mm WIRE DRAWN WITH AND WITHOUT PRIOR ANNEALING

Sample Material	Tensile Strength /MPa	
	Without Anneal	With Anneal
0,8% C	1748	1752
0,7% C	1625	1624

The results clearly reveal that the marginal increases in the UTS of the feed material on annealing are not observed in the drawn material (Table 4.9), nor do they influence the ageing response of the drawn material (Figure 4.14).

On consideration, this is an expected result, because if the increase in UTS after annealing is attributed to dislocation pinning by carbon or carbide precipitates, then as soon as the wire is deformed (e.g. by drawing), this effect will be removed, and the ferrite will once again effectively be supersaturated. The rate of subsequent ageing will not, therefore, be affected.

Shear tests were not carried out in this part of the programme since the sensitivity of the shear test is reduced at low drawing strains, and the test is therefore significant only at strains above about 2,0.

4.2.5 The Effect of Nitrogen

In order to investigate the effect of nitrogen on commercial steel wire, two similar 4 mm feed materials containing different amounts of nitrogen (0,006% and 0,012%) were obtained from the Haggie Rand Ltd factory. The full analyses of these materials are given in Table 4.10.

TABLE 4.10 : CHEMICAL ANALYSIS OF STEELS USED TO EVALUATE THE EFFECT OF NITROGEN ON AGEING

Specimen Number	C %	Mn %	Si %	S %	P %	N %	Al %
H	0,80	0,71	0,24	0,014	0,023	0,0122	0,013
L	0,80	0,74	0,24	0,012	0,024	0,0059	0,016

The mechanical properties of these materials are given in Table 4.11, which includes the measured properties after drawing to 2,20 mm diameter. The drawing conditions are also specified.

TABLE 4.11 : MECHANICAL PROPERTIES OF COMMERCIAL NITROGEN STEELS

Spec No	Patented UTS MPa	Average Diam. mm	Grain Size ASTM	Pearlite Spacing nm	2,204 mm Wire	
					UTS MPa	Shear Σ1 mm
H	1309	3,966	7	75	1733	1,71
L	1311	3,950	6	75	1733	1,90

Drawing Speed: 2,3 $m.min^{-1}$
Lubrication: Ca Soap
Water Cooling: Yes

The drawn wires were aged at 80°C, 100°C, 115°C and 130°C for various times, and the tensile strengths were measured. The increase in UTS with ageing time is shown in Figure 4.15.

The activation energy was determined for an increase of 60 MPa in tensile strength. This figure was selected on the basis that it was suitably bounded by the actual results obtained. The derivation of the energy of activation is given in Figure 4.16, from which it is apparent that a value of about 120 $kJmol^{-1}$ characterises the ageing rate in this test. This is in suitable agreement with previously obtained values. The conclusion here is that increasing the nitrogen content does not affect the mechanism of ageing; or, more accurately, supplying additional nitrogen does not allow a different mechanism (e.g. nitrogen diffusion rather than carbon diffusion) to become the rate controlling step in the reaction.

A negative effect of nitrogen on the ageing rate, however, is apparent, and since the rate increases with the nitrogen content, it can be concluded that increasing the nitrogen content increases the kinetics of the ageing process.

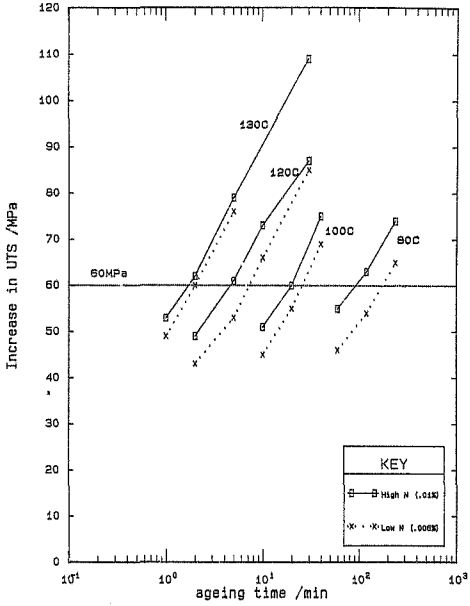


FIGURE 4.15 : THE EFFECT OF THE NITROGEN CONTENT ON THE AGEING BEHAVIOUR OF 0,8% C COMMERCIAL STEEL (1,22mm diameter), MEASURED BY THE TENSILE TEST.

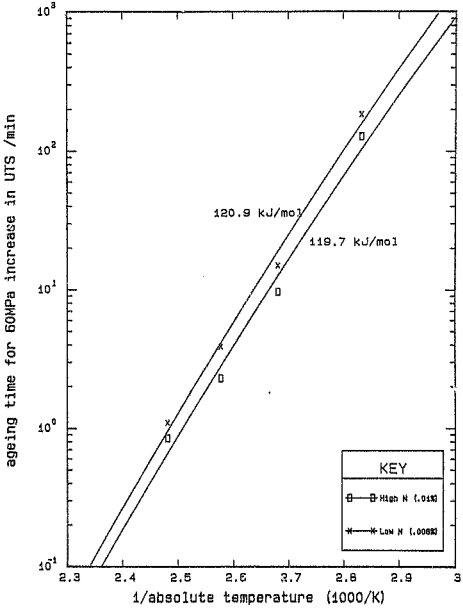


FIGURE 4.16 : THE EFFECT OF THE NITROGEN CONTENT ON THE ACTIVATION ENERGY FOR A 60 MPa INCREASE IN UTS.

4.2.6 Summary

Early work concentrated on establishing some of the process parameters which may have a significant effect on the ageing of commercial steels. Thus, the effect of drawing strain and drafting schedule; the carbon and nitrogen contents; and annealing prior to drawing were briefly considered.

An energy of activation for a small amount of ageing (as measured by the change in tensile properties) was determined for some of the conditions investigated, while the kinetics of the reaction were studied using the rate exponent. The activation energies obtained were generally close to 120 $kJmol^{-1}$, while the rate exponents was found to be between 0,25 and 0,31.

According to the measurements of the change in the UTS, the different parameters examined did not appear to markedly affect the reaction mechanism, as indicated by the activation energy.

The kinetics of the reaction were apparently increased by drawing to high strains, by increasing the volume fraction of ferrite, and by increasing the amount of nitrogen present. Annealing the feed material prior to drawing in an attempt to reduce the carbon supersaturation did not affect the ageing rate.

This work has demonstrated that the shear test, in spite of being a "crude" test, is rather better than the tensile test at determining the embrittlement of steel wires by ageing. In an instance where the UTS showed no apparent difference in the ageing behaviour of two wires, the shear test showed that the transverse properties were indeed affected differently. In this way the beneficial effect of a low drawing reduction at the final pass was noted.

The shear test is only relevant, however, for wire which can be embrittled in a reasonable time. Thus, for wires drawn to a strain of about 1,2, for example, the shear test shows only a small decrease in the shear elongation with ageing and this is unsuitable for investigations into the ageing rate. At higher strains, the effect is more pronounced, and therefore measurable.

4.3 VACUUM-MELTED PLAIN CARBON STEELS

4.3.1 Introduction

Due to the emphasis in the literature on the effect of nitrogen, especially at low temperatures, on the rate of strain ageing, it was felt necessary to investigate nitrogen further. It was hoped that by using vacuum-melting procedures, the nitrogen level could be lowered to below that at which ageing of steel is affected. The effect of nitrogen might then become clear on comparison with a similar steel containing rather more nitrogen.

It was also proposed that the interlamellar spacing of the pearlite might affect the mean diffusion distance which carbon atoms would have to travel in order to age the steel. This effect was also examined.

4.3.2 Material Preparation

The steels were manufactured as 5 kg ingots in the vacuum induction melting furnace at the University of the Witwatersrand, using the techniques described in Section 3.2.1.2. Two ingots were manufactured with the desired analyses being comparable to the commercial plain carbon steels investigated previously. A high nitrogen level in one alloy was obtained by maintaining a partial pressure of about 13 kPa of nitrogen above the melt for about 20 minutes. The chemical analyses of the ingots are given in Table 4.12.

TABLE 4.12 : CHEMICAL ANALYSIS OF VACUUM-MELTED PLAIN CARBON STEELS

Sample Number	C %	Mn %	Si %	S %	P %	N %
VH	0,72	0,70	0,30	0,012	0,005	0,034
VL	0,77	0,74	0,30	0,009	0,006	0,0014

The ingots were rolled into round bars or rods of about 10 mm diameter, and about 1 m in length; the decarburised layer (of about 1 mm thickness) left after the rolling operation was then removed by dissolving the surface layers in a dilute nitric acid solution. This acid dissolution method was employed in an attempt to overcome the practical difficulties inherent in machining long rods which are not perfectly straight.

The surface condition of the rods after this treatment was rough, since the acid dissolution tended to follow the rough rolled surface features and exaggerate the profile. However, no pitting was apparent. The rods were smoothed by pulling carefully through a die at 8,06 mm, following which the rods were baked in a tempering furnace at 200°C for 3 hours to remove, or at least reduce, the mobile hydrogen probably absorbed during the acid treatment.

Prior to further treatment, the rods were checked for quality by selecting random samples and examining the surface features in tranverse sections. About 50% of the material showed signs of laps and seams produced by the hot rolling process, and this was discarded. The large amount of defective material was a result of rolling the rod to 10 mm rather than the usual 14 mm. At the smaller rod size, control of the rod section was rather more difficult, and fins were formed very easily. These were easily rolled-in to form defects.

Patenting of the rods was carried out in the laboratory muffle furnace and the experimental lead bath described in Section 3.2.1.3, using nominally standard conditions. Since the desired wire length after final processing was required to be as high as possible, long rod lengths were employed, which resulted in a portion of the rod protruding from the surface of the lead bath during quenching. This part of the rod was marked and used for pointing the rod for the drawing operation, and was subsequently removed after drawing was completed.

The patented rod was then carefully drawn into 4,05 mm wire in five passes, at which size the wire was repatented prior to further drawing to the finished wire size. Use of the Haggie Rand Ltd production patenting facilities could not be considered because adjusting the lead bath temperatures to give different pearlite spacings would have been impractical.

To facilitate the patenting of longer lengths of wire (i.e. greater than 1 m), the wire was bound loosely into 200 mm coils. This enabled the wire to fit into the lead bath and also reduced the temperature variations across the length of the wire in the austenitising furnace. This requirement was unfortunate, since non-uniform cooling across the wire section occurred in the lead bath, at the cross-over points of the coil. In addition, no control over the furnace atmosphere during austenitising could be achieved, since the leakage of the inert gas through the furnace walls would have been too great.

An austenitising treatment of 3 minutes at 900°C was found to satisfactorily homogenise the steel, without causing too much scale formation. This was followed by a quench into lead at 550°C for the nitrogen investigation, and also at 590°C and 630°C for the pearlite spacing investigation. The details of the heat treatments are given in Table 4.13, and the typical microstructures are shown in Plates 4.1 to 4.4. It was apparent that transforming at the highest temperature led to a coarser pearlite, and also to a more broken, degenerate microstructure. This could be due to the onset of spheroidisation at 630°C. In the steels patented at 550°C and 590°C, the structures were more uniform.

TABLE 4.13 : PATENTED PROPERTIES OF VACUUM-MELTED PLAIN CARBON MATERIALS

Spec No	Lead Temp °C	Immersion Time s	Av. UTS MPa	Minimum Pearlite Spacing nm	Drawn UTS @1,33mm MPa	Work Hardening Rate MPa/ε
VH	553	60	1097	80	1882	355
VL	551	60	1115	80	1969	386
VL	592	60	1095	91	1896	362
VL	631	180	1048	100	1793	337

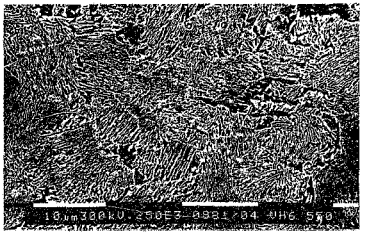


Plate 4.1 : High Nitrogen Vacuum Steel Transformed at 550°C.
2% Nital Etch 2500x.

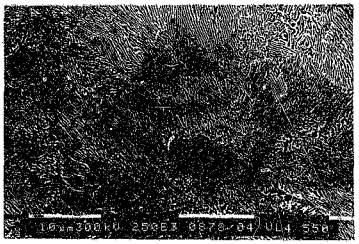


Plate 4.2 : Low Nitrogen Vacuum Steel Transformed at 550°C.
2% Nital Etch 2500x.

The variability in the tensile results was very high; for instance, in one test coil, a standard deviation of 26 MPa was determined over twelve tests on samples taken along the coil length. This scatter could not be reduced using these heat treating facilities. The lower strength and work hardening rate of the steel patented at the higher temperatures was expected, while the high nitrogen material exhibited lower tensile properties due to its lower carbon content.

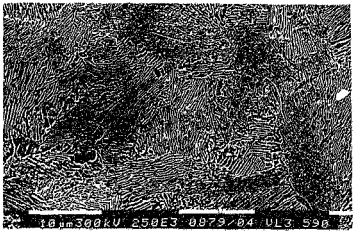


Plate 4.3 : Low Nitrogen Vacuum Steel Transformed at 590°C. 2% Nital Etch 2500x.

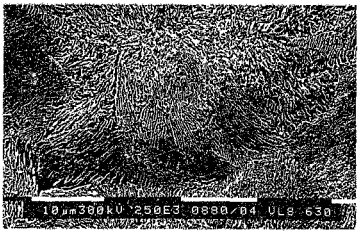


Plate 4.4 : Low Nitrogen Vacuum Steel Transformed at 630°C. 2% Nital Etch 2500x.

In order to ensure consistency in the drawing conditions between different sample coils, selected coils were butt welded together before drawing. Use was again made of the facilities at Haggie Rand Ltd for this purpose. The weld positions were marked by plating copper onto the steel surface from an acid copper sulphate solution. The weld regions were cut out and discarded at the wire final size.

The feed size after pickling was measured to be 4,02 mm diameter, on average. Drawing to 1,33 mm diameter was performed using the die set given by Table 4.14, at 1,8 $m.min^{-1}$, and the otherwise standard drawing conditions.

TABLE 4.14 : DIE SET FOR 4,02 mm FEED MATERIAL

Die Number	Die Size mm	Reduction in Area %	Total Strain
1	3,405	28,3	0,332
2	2,908	27,1	0,648
3	2,536	23,9	0,921
4	2,209	24,1	1,197
5	1,959	21,4	1,438
6	1,760	19,3	1,652
7	1,575	19,9	1,874
8	1,441	16,3	2,052
9	1,322	15,8	2,224

4.3.3 *The Effect of Nitrogen*

The wire samples which had been patented at 550°C prior to drawing were subjected to various ageing treatments, in order to determine the ageing behaviour. Tensile and shear tests were performed on the aged wires, although the limited quantity of samples restricted the number of replications of each test per condition to just three.

The results for the increase in UTS with ageing are shown in Figure 4.17. The typical scatter in the tensile results was about 40 MPa, reflecting the variation in the feed wire properties. In spite of this, an estimate of the activation energy was made for a 100 MPa increase in UTS (Figure 4.18), and for both the high and the low nitrogen steels, a similar value of about 112 $kJmol^{-1}$ was determined.

This value agrees with previous data (see *Section* 4.2), and again implies that the level of nitrogen does not affect the ageing mechanism, in terms of the tensile properties. It is apparent, however, that the high nitrogen material does maintain a higher ageing response, as evidenced by the offset in the tensile ageing curves.

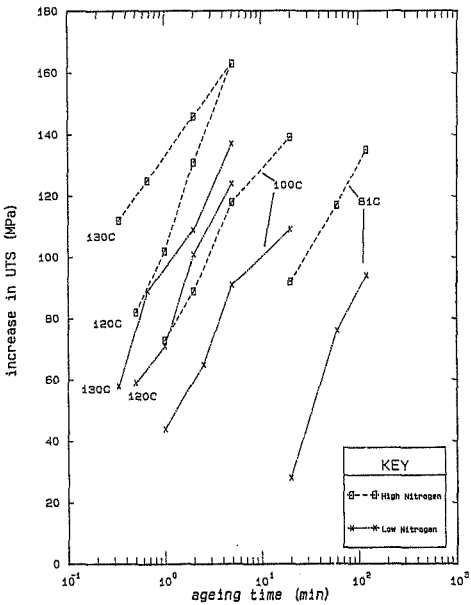


FIGURE 4.17 : THE EFFECT OF THE NITROGEN CONTENT ON THE AGEING BEHAVIOUR OF VACUUM-MELTED PLAIN CARBON STEEL, MEASURED BY THE TENSILE TEST.

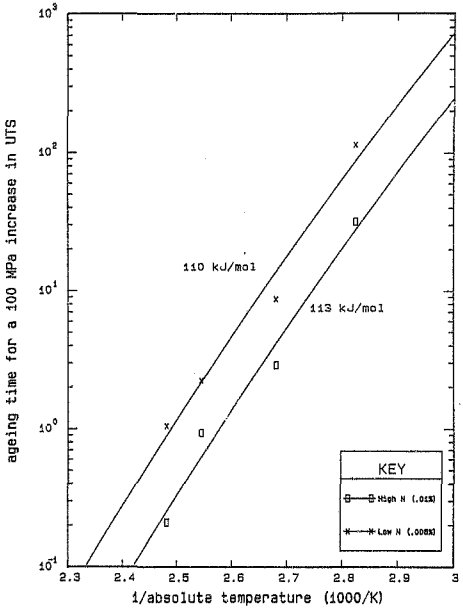


FIGURE 4.18 : THE EFFECT OF NITROGEN CONTENT ON THE ACTIVATION ENERGY FOR AGEING IN VACUUM-MELTED PLAIN CARBON STEEL.

The variation in the shear properties with ageing is of greater interest. Figure 4.19 shows clearly that the ageing behaviour of the high nitrogen alloy was far more severe than of the low nitrogen alloy. The low nitrogen steel maintained a high and satisfactory level of shear elongation for much longer ageing times throughout the temperature range considered, while the high nitrogen material became embrittled in a short time. This behaviour is difficult to quantify, due to the inherent variance in shear test results, but is reasonably consistent and reproducible.

4.3.4 The Effect of Pearlite Interlamellar Spacing

The difference in the minimum interlamellar spacing observed in the feed wires was about 10 nm between wires patented at 550°C and 590°C, and also between wires patented at 590°C and 630°C. The wires patented at 630°C also showed a more degenerate microstructure.

Tensile tests were performed on the drawn wires aged at 80, 120 and 130°C, and the increase in tensile strength on ageing is shown in Figure 4.20. Again, the slopes of the ageing curves appeared to be similar, but the ageing rate was increased as the strength of the materials increased (i.e. as the pearlite spacing decreased). It therefore seems likely that the pearlite interlamellar spacing does affect the ageing rate of the steel after drawing. It is not, however, clear whether this is due to differences in the drawn microstructure (e.g. the dislocation density etc.) or due to the interlamellar spacing itself (which influences not only the diffusion distance via the distance between the pearlite lamellae, but also the arrangement of the dislocations present and particularly in terms of cell size).

4.3.5 Discussion

4.3.5.1 Experimental Procedure

This early work on vacuum melted steels highlighted a number of practical difficulties associated with the processing of such steel into wire on a small-scale experimental basis.

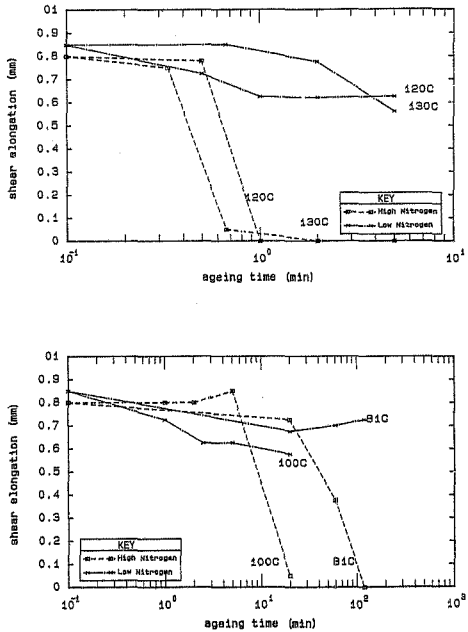


FIGURE 4.19 : THE EFFECT OF NITROGEN CONTENT ON THE AGEING BEHAVIOUR OF VACUUM-MELTED PLAIN CARBON STEEL, MEASURED BY THE SHEAR TEST.

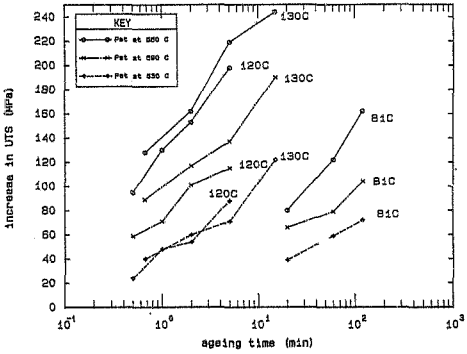


FIGURE 4.20 : THE EFFECT OF THE PEARLITE INTERLAMELLAR SPACING ON THE AGEING BEHAVIOUR OF A PLAIN CARBON LOW NITROGEN STEEL.

The biggest problem encountered centred around the requirement to manufacture as much wire as possible for any one test with consistent properties along its length, while using equipment designed for short wire lengths.

The heat treatment facilities available, for instance, were only suitable for short lengths of material (less than about 400 mm), although in fact, they still give inferior wire properties when compared to factory processed wire under the same nominal conditions. The reasons for this are not clear, but are possibly linked to the "strike temperature" (the temperature at which the material enters the lead bath on quenching), or to the overall quench rate from the austenitising temperature.

For longer wire lengths the heat treatment had to be performed on coils, because of the limited dimensions of the lead bath, and the problems were compounded by the additional errors due to non-uniform heat treatment. During austenitising, for example, the temperature variation measured across the coil diameter within the muffle furnace was found to be about 20°C. All of these factors contributed to the varying properties along the wire length, and between wire coils.

The acid dissolution technique for removing the decarburised zone on the rod surface was reasonably successful, but it was considered that the improved surface quality attainable with a machined surface was desirable. Therefore, conventional machining would be used for all future experiments of this type.

4.3.5.2 Experimental Results

This work on plain carbon steels has again shown again that nitrogen increases the ageing rate of a high carbon steel wire. The mechanism of the reaction appears to be unchanged by the level of nitrogen, even when small amounts are present and this implies that even 0,0015% nitrogen may be sufficient to cause ageing in such steels. However, it is not necessary that the sole cause of ageing is nitrogen; if an alternative mechanism controls the rate of ageing, then nitrogen will not be seen to have any effect on this.

The interesting result from this work is the marked detrimental effect that nitrogen has on the shear properties of the wire, while the tensile properties are little changed. It is therefore apparent that the ageing phenomenon affects the transverse properties in a different manner to the tensile properties.

On consideration of this effect, it might be proposed that this implies an embrittlement of the grain boundary or pearlite colony boundary regions, since these are the predominant longitudinal discontinuities in a heavily drawn structure such as wire. In addition, the dislocation density has been noted to be very high at the (longitudinally aligned) ferrite/cementite interfaces of pearlite (Bee, 1986), and would also be high at the low angle grain boundaries. Strain ageing by the diffusion of interstitial atoms to free dislocation sites would be expected to progress most rapidly at such discontinuities, and thus, embrittlement by strain ageing might be considered to be directional in its effects on the wire properties.

It is not inconceivable that two processes are occurring concurrently. Thus, for instance, nitrogen might embrittle the grain boundaries, while a different rate-controlling reaction proceeds as usual (e.g. strengthening the ferrite lattice).

The nature of the rate-controlling mechanism is unclear. Yamada, as described in detail in Section 2.4.2, found that the dominant mechanism during second stage ageing was the dissolution of carbon from cementite to furnish carbon atoms for dislocation pinning. This might imply that the pearlite interlamellar spacing is important, since this will then define the diffusion distance required for ageing to occur. The present study showed that an decreased pearlite spacing tended to increase the rate of increase of the UTS, as would be expected.

However, the pearlite interlamellar spacing affects a number of properties, such as the tensile strength in the patented condition, and the work hardening rate during drawing. It is therefore possible that the dislocation sub-structure after drawing will be affected by the pearlite spacing; generating for instance smaller cells to increase the yield strength and the work hardening rate.

Work done by Waugh et al (1981) has shown that dislocation migration may occur during ageing, and it is therefore possible that the increase in the tensile properties during ageing are due to refinements in the dislocation sub-structure, rather than to redistribution of the interstitial solute. These authors suggested that full decoration of the dislocation substructure occurred during the cold drawing operation, due to the large excess of carbon atoms present, and to the small diffusion distances involved, and therefore carbon played little or no part in the ageing of steels after drawing.

The driving force for the movement of the dislocations would be the residual stresses in the wire remaining after drawing, and a reduction of the strain energy stored in the lattice. The energy of activation required for the dislocation movement is dependent on the driving force; in the absence of any assistance by an applied stress, an activation energy equivalent to that for self-diffusion in ferrite may be assumed (Reed-Hill, 1973), and this is about 250 $kJmol^{-1}$. This would be substantially reduced if the dislocations moved in the direction of positive stress.

This work has not been able to differentiate between these two ageing theories, but the possibility of the occurrence of dislocation re-arrangement has not been eliminated.

4.4 VACUUM-MELTED STEELS WITH VARYING SILICON CONTENTS

4.4.1 Introduction

Krishtal (1970) has shown that silicon additions to steel tend to depress the pre-exponential term in the expression for the diffusion coefficient of carbon in alloyed austenite (Table 2.2, Section 2.5.2). It has been suggested that silicon, on this basis, might therefore slow the rate of ageing in steels by also inhibiting the diffusion of carbon in the ferrite to free dislocations (Smith, 1985).

An investigation was therefore conducted into the effect of silicon additions on the rate of ageing in vacuum-melted steels, and this will be described below.

4.4.2 Material Preparation

Silicon is normally added in small quantities (of about 0,3%) to steel wire rod, for deoxidation purposes. For this experiment, the desired silicon contents were 0%; 0,5%; 1%; and 2%. These compositions encompass the range of silicon contents found in normal commercial plain carbon and low alloy steels for high strength wire production. A 0,8% carbon, 0,6% manganese steel base was used for the silicon additions, and no attempt was made to compensate for the increase in the eutectoid carbon composition of the steel due to silicon additions.

The steels were manufactured under vacuum as described in Section 3.2.1.2, and the final analyses of the ingots are given in Table 4.15.

TABLE 4.15 : CHEMICAL ANALYSIS OF SILICON STEELS

Ingot No	Carbon %	Manganese %	Silicon %	Total Nitrogen %
2	0,83	0,60	0,51	0,0013
3	0,79	0,63	1,02	0,0016
4	0,81	0,65	2,04	0,0024
7	0,79	0,61	-	0,0010

Unfortunately, about 50% of the 2% silicon steel ingot had to be discarded due to a large pipe in the ingot.

The 5 kg ingots were rolled to 14 mm round bar, after which the decarburised layer was removed by machining the rods on a lathe, and centreless grinding, to 10,5 mm diameter. The rods were then butt-welded together, and patented on the continuous patenting line at Haggie Rand Ltd using the normal conditions for a plain carbon steel of this diameter (i.e. austenitise at 960°C for 5,5 minutes, and quench into lead at 540°C for about 75 s). After pickling and coating with phosphate and borax as usual, the rods were then drawn to 4,05 mm diameter in seven passes on the single hole drawing machine.

It was decided to patent the 4 mm material in the Haggie Rand Ltd works to ensure consistent heat treatment conditions along the wire length. This was in the light of previous problems with small-scale batch patenting as described in section 4.4. However, it was apparent that the heat treatment was not ideal metallurgically for the high silicon steels, since silicon has the effect of raising the temperature at the "nose" of the isothermal transformation curve. Figures 4.21 and 4.22 show that the ideal patenting temperatures should be at about 600°C and 625°C for the 1% and 2% silicon steels respectively.

The patenting conditions for the 4 mm material are given in Table 4.16, while the patented microstructures are shown in Plates 4.7 to 4.10.

TABLE 4.16 : WORKS PATENTING CONDITIONS FOR SILICON STEELS

Furnace Number	EF4
Average Temperature	940°C
Immersion Time	3 min
Lead Bath	
Temperature	560-565°C
Immersion Time	40 s

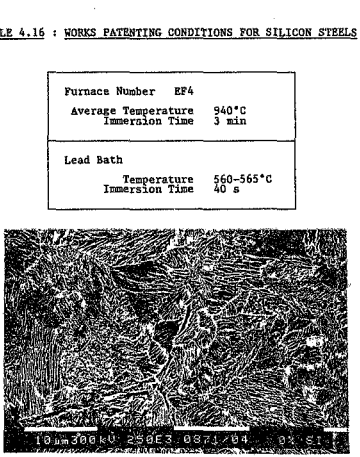


Plate 4.5 : Patented Structure of Steel Containing 0% Silicon. 2% Nital Etch 2500x.

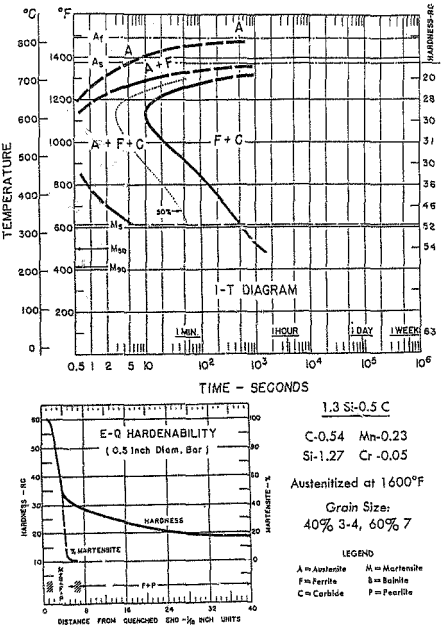


Figure 4.21: Isothermal Transformation Curve for a Steel Containing 1.27% Silicon.

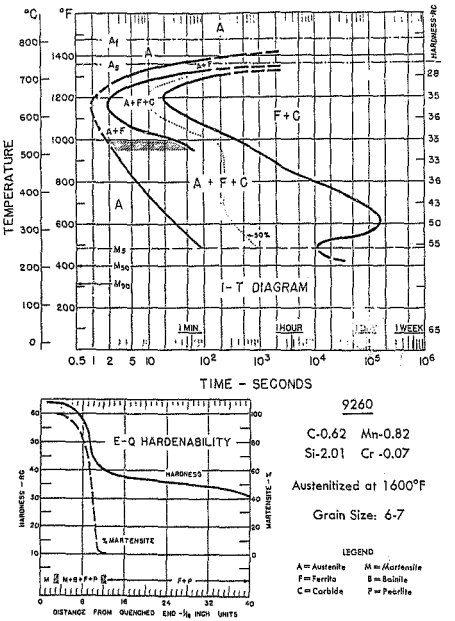


Figure 4.22 : Isothermal Transformation Curve for a Steel Containing 2% Silicon.

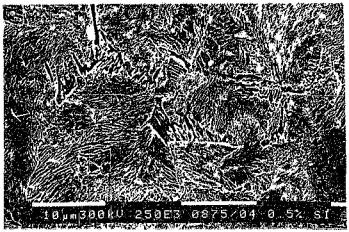


Plate 4.6 : Patented Structure of Steel Containing 0,5% Silicon 2% Nital Etch 2500x.

Little basic difference in the patented microstructures was observed in the steels containing 1% silicon and less. These generally consisted of a fine, uniform pearlite with a small volume fraction of ferrite, and some degenerate pearlite. A larger volume fraction of ferrite was noted in the 1% silicon alloy, due to its slightly lower carbon content and the effect of silicon in raising the eutectoid carbon composition, while some Widmanstatten ferrite side plates were also noted. Some of the pearlite was close to bainite in morphology, indicating that the transformation temperature was close to the bainitic region.

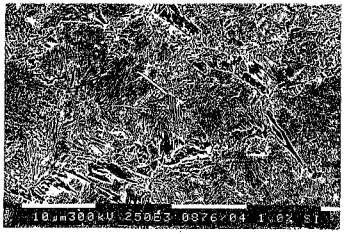


Plate 4.7 : Patented Structure of Steel Containing 1% Silicon 2% Nital Etch 2500x.

The minimum observed pearlite interlamellar spacing for each of these alloys was about 80 nm. The mechanical properties of these works patented silicon steels are given in Table 4.17.

TABLE 4.17 : MECHANICAL PROPERTIES OF WORKS-PATENTED SILICON STEELS

Alloy Number	Silicon Content %	Tensile Strength MPa	Reduction in Area %
2	0.51	1313	42.4
3	1.02	1322	34.5
7	0	1219	47.0

As expected, the silicon additions increased the strength and decreased the ductility of the steel, by solid solution strengthening of the ferrite.

The 2% silicon alloy was found to be totally unsuitable for drawing, since the structure consisted of a mixture of Widmanstatten ferrite, bainite, pearlite and tempered martensite (Plate 4.8), as would be expected after transforming at 560°C for only 40 seconds (see Figure 4.22). This alloy could not therefore be included in the initial investigation on works-patented material.

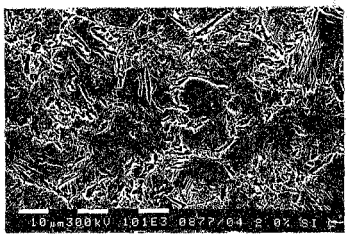


Plate 4.8 : Patented Structure of Steel Containing 2% Silicon
2% Nital Etch 1000x.

A secondary investigation was then performed on the remainder of the 1% and 2% silicon steels. Patenting trials were performed using different lead quenching temperatures to find the ideal patenting conditions, but the number of tests was limited due to a shortage of samples. The results of some of these tests on the 2% silicon alloy are summarised in Table 4.18; a compromise heat treatment for both alloys at 610°C for two minutes was selected for further investigation. The microstructures of the steels after this treatment are shown in Plates 4.9 and 4.10.

TABLE 4.18 : SUMMARY OF PATENTING TRIALS ON 2% SILICON STEELS

Austenitise at 940°C for 3 minutes				
Lead Temp °C	Immersion Time s	Mechanical Props UTS MPa	Mechanical Props Redn of Area %	Comments
559	40	1133	30,8	Macro-segregation - finer grain size + higher Vf* ferrite at centre. Widmanstatten (Ws) ferrite + bainite.
558	150	1248	33,2	Lower Vf ferrite
599	40	1341	35,2	Less Ws ferrite + bainite + some degenerated pearlite
607	120	1287	41,4	Very small amount of Ws ferrite + pearlite + 5% Vf bainite. Min. pearlite spacing 80nm

* Vf = Volume fraction

The 4 mm material from both experiments was drawn into 1,32 mm and 1,22 mm diameter wire, using the die set given in Table 4.14, with drawing speeds ranging from 4,2 to 6,5 $m.min^{-1}$. Direct water cooling, as usual, was employed on each pass. The work hardening rate of the works-patented wires was monitored by sampling after each pass and tensile testing, and these results are shown in Figure 4.23. For the 1,22 mm wire, ageing treatments were carried out at 80 C to 130°C for various times, but for the 1,32 mm wire, sufficient sample was only available for ageing at 100°C.

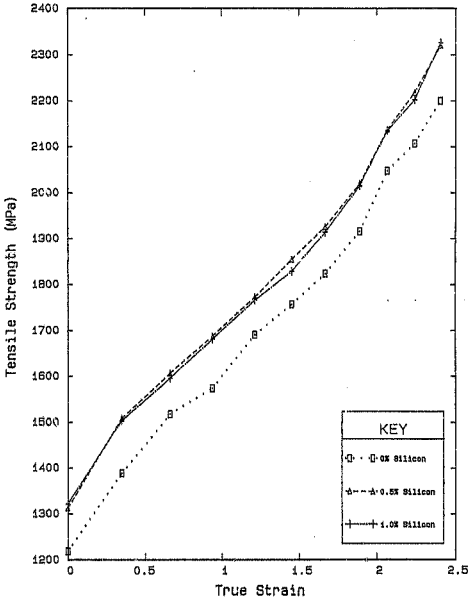


FIGURE 4.23 : WORK HARDENING CURVES FOR SILICON STEELS (works-patented material).

Plate 4.9 : 1% Silicon Steel Transformed at 610°C.
2% Nital Etch 2500x.

Plate 4.10 : 2% Silicon Steel Transformed at 610°C.
2% Nital Etch 2500x.

The wire samples were tension tested using the HP-85 computer to record the stress/strain points and to calculate the 0,2% proof stress. Shear tests were also performed.

4.4.3 Results and Discussion

Considering first the works-patented material, the results from the ageing tests are given in Figures 4.24 to 4.29. Figures 4.24 and 4.25 show the increase in the 0,2% proof stress after ageing at the two wire sizes. At 1,32 mm, the 0,5% silicon alloy was observed to age more rapidly than either the 0% or the 1% silicon material, and this effect was repeated at 1,22 mm.

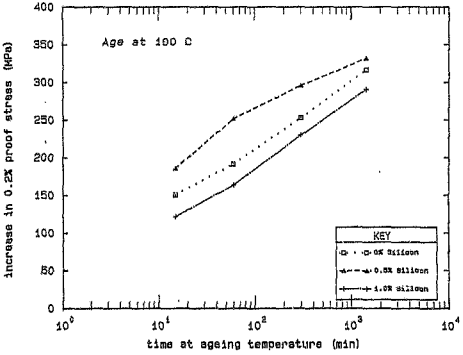
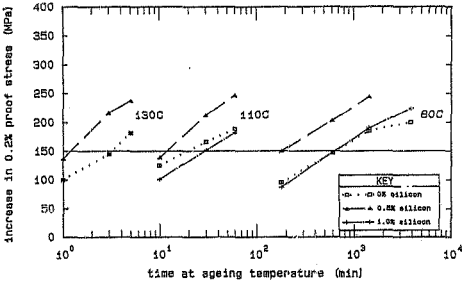


FIGURE 4.24 : THE EFFECT OF SILICON CONTENT ON THE AGEING BEHAVIOUR OF 1,32mm WIRE, MEASURED BY THE TENSILE TEST.



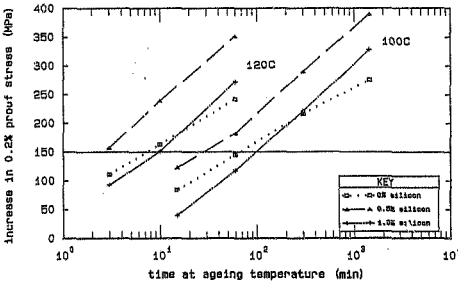


FIGURE 4.25 : THE EFFECT OF SILICON CONTENT ON THE AGEING BEHAVIOUR OF 1,22mm WIRE, MEASURED BY THE TENSILE TEST.

It was also apparent that the slope of the increase in proof stress vs ln(time) curves for the 0% silicon steel was lower than those for the steels containing silicon. This implies that a higher energy of activation was applicable for the ageing mechanism in this steel.

Activation energies were derived for ageing in the 1,22 mm wire (Figure 4.26) using an increase in the 0,2% proof stress of 150 MPa. The results from this analysis are given in Table 4.19. It is apparent that a difference does appear to exist between the steels containing silicon, and the steel without silicon.

TABLE 4.19 : ACTIVATION ENERGIES FOR A 150 MPa INCREASE IN 0.2% PROOF STRESS FOR WORKS-PATENTED SILICON STEELS

Alloy Number	Silicon Content %	Activation Energy $kJmol^{-1}$
2	0,5	120
3	1,0	119
7	0	127

For the steels containing silicon, the activation energies obtained were of the same order as those obtained previously (i.e. about 117 $kJmol^{-1}$), indicating that an increase in the silicon content from 0,3% (in commercial steels) to 0,5% or 1% probably has no effect on the ageing mechanism in drawn wire.

Small amounts of silicon are apparently detrimental, and this is reflected in the lower activation energy value obtained when silicon was added. This trend in the results might be expected from Table 2.2, which shows the variation in the diffusivity of carbon in alloyed austenite, with the concentration of alloying addition. The addition of 0.93% silicon reduced the activation energy for carbon diffusion by 13%; although in this instance the reduction was only about 6%. The reason for this effect of silicon on the diffusion rate is not clear.

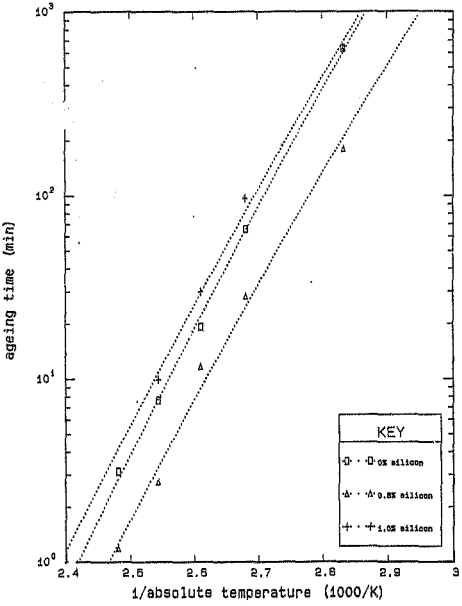


FIGURE 4.26 : THE EFFECT OF SILICON CONTENT ON THE ACTIVATION ENERGY FOR A 150 MPa INCREASE IN THE 0,2% P.S.

If the dominant ageing mechanism is carbon dissolution and diffusion from the cementite lamellae, then the effect of silicon on the activity of carbon in iron becomes important. Silicon is known to increase this value markedly (Geiger and Poirier, 1973), and thus would tend to encourage precipitation of the carbon. By Le Chatelier's principle, cementite will tend to dissolve to maintain the equilibrium concentration of carbon in the ferrite, and this may have an effect on the measured value of the binding energy for carbon to cementite (note that silicon is known to act as a graphitiser in cast irons). This would then account for the reduction in the measured activation energy for ageing with silicon additions.

For the steels patented at 610°C, unfortunately, only a small number of wire samples was available after drawing for the determination of the ageing properties. A limited number of tension tests could therefore be performed, and these results are listed below in Table 4.20.

TABLE 4,20 : THE AGEING BEHAVIOUR OF HIGH SILICON STEELS AS MEASURED BY THE CHANGE IN THE PROOF STRESS

Ageing Treatment	Increase in 0,2% Proof Stress / MPa		
	1% Si (610°C)	1% Si (550°C)	2% Si (610°C)
80°C 3h 10h	68 103	87 148	31 24
100°C 5h	213	211	156
110°C 0,5h	141	152	123

It is apparent that the 2% silicon steel exhibited a markedly reduced ageing effect when compared to the 1% silicon steel, whether the latter was transformed at 550°C or 610°C. The behaviour of the 1% silicon steel patented at the two temperatures was very similar.

The ultimately beneficial effect of silicon (at levels greater than about 1%) might be due to the increase in the ferrite lattice strain due to solid solution of the silicon, which may inhibit carbon diffusion. According to Smith (1986), the lattice distortion around a silicon atom in solution in the ferrite is in the opposite sense to that around a carbon atom. Carbon atoms in close proximity to a silicon atom will tend to relax the strain fields, and thereby effectively trap the carbon atom.

The behaviour of the aged wires in the shear test is shown by Figures 4.27 to 4.29. It is apparent that no consistent trend in the effect of silicon on the shear test ageing response could be determined, possibly due to experimental errors. It is, however, probable that in the range of silicon contents examined here, silicon has little effect on the embrittlement of steel wire.

In summary, the results from this work seem to suggest that silicon added in small quantities (up to 1% silicon) tends to increase the rate of ageing as measured by the tensile properties, while at higher levels (of about 2%), the effect is reversed to some extent. However, this trend is not very distinct, and is also not reflected in the shear test results. More work would therefore have to be done to confirm this effect.

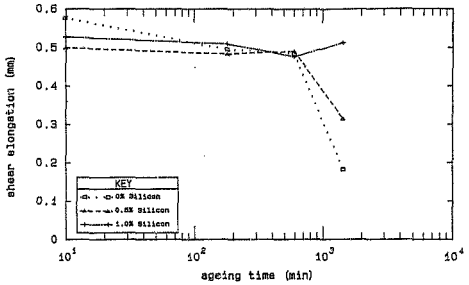


FIGURE 4.27: EFFECT OF SILICON ON THE BEHAVIOUR OF 1,22mm WIRE IN THE SHEAR TEST AFTER AGEING AT 80°C.

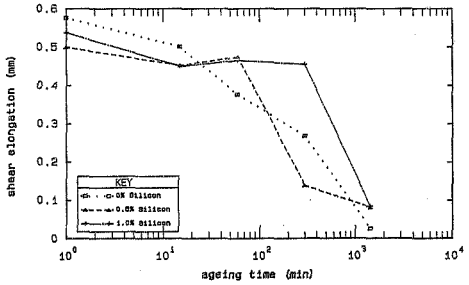


FIGURE 4.28 : EFFECT OF SILICON ON THE BEHAVIOUR OF 1,22mm WIRE IN THE SHEAR TEST AFTER AGEING AT 100°C.

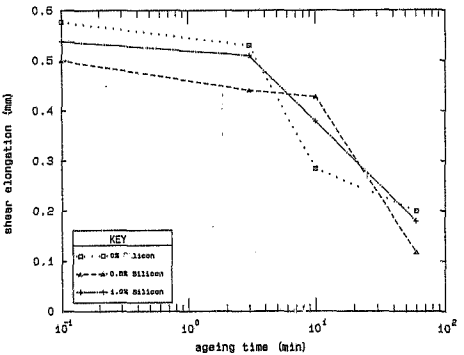


FIGURE 4.29 : EFFECT OF SILICON ON THE BEHAVIOUR OF 1,22mm WIRE IN THE SHEAR TEST AFTER AGEING AT 120°C

CHAPTER FIVE : FACTORIAL ASSESSMENT OF FOUR PARAMETERS ON THE RATE OF STRAIN AGEING

5.1 MOTIVATION

The concept of the factorial design of experiments is well established, and is described in standard texts (see e.g. Yates, 1937; Natrella, 1979). Factorial experiments are those which include all the possible combinations of several different sets of treatments or factors. In a normal experiment, such as those described in some detail in Chapter Four, one factor is varied while the other parameters are held constant, and the response of interest (e.g. some mechanical property) is measured. Very often, however, it is difficult or even impossible to maintain all the other parameters constant, due to interactions between them, and in a normal experiment these will not be detected. Factorial experiments on the other hand, will detect these interactions, and, in addition, reduce the required number of experiments, due to more efficient organisation.

The simplest form of factorial design, whereby two levels only of each factor are investigated, was selected for this work in order to restrict the number of experiments. Four factors were considered, namely the nitrogen content, the aluminium content, the drawing speed and the total drawing strain.

The detrimental effect of nitrogen on the ageing response of a steel wire has been demonstrated in Sections 4.2.5 and 4.3.3, but it was hoped here that an indication of the means of reducing its influence might be obtained. In particular, it was hoped that aluminium additions would combine with the nitrogen in solid solution in the ferrite, and eliminate its influence on the ageing process.

The possible beneficial effect of a high strain rate during drawing was proposed in Section 4.4.2, where it was shown that a small final reduction apparently reduced the embrittlement of the wire during ageing. It is not clear, however, whether these benefits were due to an improved internal stress field in the wire, to a reduction in the amount of redundant deformation, or to a higher strain rate. Since the strain rate is directly proportional to the wire exit speed from the die (Equation 2.13) it was decided to investigate the effect of the drawing speed in an attempt to isolate the effects of the strain rate.

The total drawing strain has already been shown to have a negative effect on the rate of ageing, but it was hoped that methods of reducing the effect might become apparent in the interaction of the drawing strain with other parameters.

5.2 THE FACTORIAL DESIGN

Each factor in the design is evaluated at two levels and for this design, these are specified in Table 5.1. Generally, the levels were selected so as to give the maximum possible effect while still retaining close relevance to industrial conditions.

TABLE 5.1 : FACTORIAL EXPERIMENT DESIGN LEVELS

Factor	Low Level	Mean	High Level	Unit
Drawing Speed	4	32	64	$m.min^{-1}$
Total Drawing Strain	2,22	2,30	2,38	-
Aluminium	0	0,05	0,10	wt %
Nitrogen	0,002	0,006	0,01	wt %

Steel Composition	Experimental Constants
0,80% carbon	Drafting schedule
0,65% manganese	Heat treatment
0,30% silicon	Surface condition
	External cooling conditions

The low nitrogen level of 0,002% is well below that which might be expected in commercial steels which have not been specially treated (e.g. by vacuum degassing). The upper level of 0,01% is approximately the maximum level which may be achieved in steels manufactured in an electric arc furnace.

The aluminium addition of 0,1% is, stoichiometrically, some five times more than would be required to combine with all the available nitrogen as AlN. Some of the aluminium would be expected to form compounds with oxygen or silicon to form non-metallic inclusions, but the inclusion content of the steels was found to be very low.

The two levels of drawing strain are in the region relevant to commercial ultra-high strength steel production, and would be expected to correspond to finished tensile strengths of about 2150 MPa and 2250 MPa respectively. It has been suggested that strain ageing only becomes a significant problem in roping wire at drawing strains of greater than about 2,27 (Shipley, 1986). If this is so, it is possible that some event occurs at about this point which may alter the ageing mechanism, or the ageing rate. The strains selected here fall on either side of this point.

The selected drawing speeds bear little resemblence to practical conditions, since the exit speed was maintained at a constant level at each pass, unlike on a commercial multi-pass machine. The low level represents normal laboratory conditions, while the high level is of the same order of magnitude as normal production finishing speeds for this type of wire. Higher speeds could not successfully be used here because of wire pay-off and take-up problems on the single hole drawing rig.

A total of 16 experiments were required for the 2^4 design; and the configuration of each experiment in the factorial design is specified in full in Table 5.2, where each factor is represented by a minus sign for the low level and a plus sign for the high level. This configuration follows a standard format (Yates, 1937) which enables the systematic analysis of the results by standard techniques.

Yates' analysis procedure is described in Appendix B, together with a copy of a computer programme written to perform the analysis for the four-factor design.

TABLE 5.2 : FACTORIAL DESIGN SPECIFICATION

Expt No	Design Variables N	Al	Ē	Sp	N %	Al %	Total Strain (Ē)	Speed (Sp) m.min^{-1}
1	-	-	-	-	0,002	0	2,22	4
2	+	-	-	-	0,01	0	2,22	4
3	-	+	-	-	0,002	0,1	2,22	4
4	+	+	-	-	0,01	0,1	2,22	4
5	-	-	+	-	0,002	0	2,38	4
6	+	-	+	-	0,01	0	2,38	4
7	-	+	+	-	0,002	0,1	2,38	4
8	+	+	+	-	0,02	0,1	2,38	4
9	-	-	-	+	0,002	0	2,22	60
10	+	-	-	+	0,01	0	2,22	60
11	-	+	-	+	0,002	0,1	2,22	60
12	+	+	-	+	0,01	0,1	2,22	60
13	-	-	+	+	0,002	0	2,38	60
14	+	-	+	+	0,01	0	2,38	60
15	-	+	+	+	0,002	0,1	2,38	60
16	+	+	+	+	0,01	0,1	2,38	60

5.3 EXPERIMENTAL PROCEDURE

To obtain the required nitrogen and aluminium combinations, four steels compositions were required and these were manufactured in the vacuum induction furnace as 5 kg ingots, and rolled to 14 mm round bar, as described in Section 3.2.1.2. The steel analyses are given in Table 5.3. The rods were then turned to 11,5 mm and finished by centreless grinding to 10,5 mm, to ensure a defect-free, uniform, undecarburised surface.

TABLE 5.3 : CHEMICAL ANALYSES OF STEELS USED IN FACTORIAL DESIGN

Ingot No	Carbon %	Manganese %	Silicon %	Nitrogen %	Aluminium %
1	0,79	0,70	0,37	0,0015	0,000
2	0,82	0,66	0,30	0,0008	0,098
4	0,77	0,76	0,32	0,0082	0,001
6	0,81	0,63	0,28	0,0082	0,109

Patenting of the rods was carried out in the Haggie Rand Ltd works after butt-welding the rods together, as before, using standard conditions for this size material:

furnace no. FG1 : 950°C for 5,4 min; and
lead bath : 540°C for 75 s.

The patented rods were drawn to 4,05 mm, the feed size for the final wire drawing, on the single hole drawing machine in seven passes.

The small diameter wire lengths were then re-patented in the Haggie Rand Ltd works using identical conditions to ensure equivalence of the microstructures, and the conditions here are given in Table 5.4, which includes the mechanical properties for the as-patented material.

TABLE 5.4 : PATENTING OF 4,05 mm FEED MATERIAL

Furnace No. EF4 : 930 - 940°C, 3 min Lead Bath : 560 - 565°C, approx 40 s			
Steel No	Diameter mm	Av UTS MPa	Constriction %
1	4,010	1286	44,4
2	4,028	1293	40,4
4	4,010	1283	40,0
6	4,030	1277	42,0

The typical microstructures were all pearlitic, with a small amount of pro-eutectoid ferrite. The austenite grain size was similar for all the steels (whether aluminium was present or not) at about ASTM number 7.

Prior to drawing into wire according to the experimental design, the feed material was pickled and surface treated with phosphate and borax coatings, as normal. The wire was drawn to 1,32 and 1,22 mm diameter in a random order, in line with statistical principles. The work hardening rate during drawing of the wires was monitored, where sufficient sample material was available, and these data are represented in Figure 5.1.

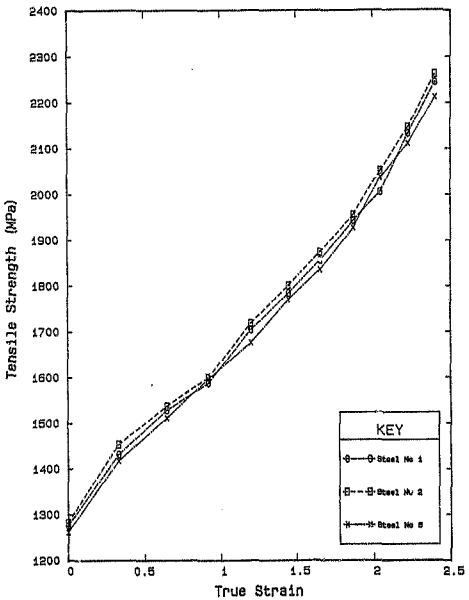


FIGURE 5.1 : WORK HARDENING CURVES FOR WIRES USED IN FACTORIAL EXPERIMENTS.

After drawing, the wire was divided into 84 equal lengths of 200 mm each, suitable for tensi. and shear testing. Seven groups of wires for ageing were then selected on a random basis, and were loosely bound. Samples were stored, as before, at -22°C in a domestic freezer.

Because of the requirement to achieve statistically significant results, the number of ageing treatments was reduced in favour of a larger number of samples per treatment. Thus twelve samples per group allowed for six tensile tests and six shear tests. The ageing treatments decided upon were designed to represent light and severe ageing treatments at representative temperatures. The wires were tested in the following conditions:
as-drawn;
aged 50°C : 2h, 500h, 1000h;
aged 80°C : 48h; and
aged 100°C : 2h, 24h.

Consideration was given to the need for even heating of the wires within the bundles, and between bundles, so for this reason, longer rather than shorter ageing times were selected. Ageing was carried out, as before, in the silicon oil bath maintained to within 1°C of the required temperature, except for the ageing at 50°C which was carried out in a laboratory convection oven maintained to within 3°C of the required temperature.

Previous work had shown (see section 4.3.3) tha. when a wire has severely embrittled, its greater tendency to break within the gripped length in the tensile test gave rather unreliable results, especially when relatively small wire diameters were used. In addition, problems were being experienced with the set of grips in use. It was therefore decided to measure the 0,2% proof stress as well as the UTS, and the programmes in Appendix A were used for this purpose.

All shear tests were carried out at the same test pressure, in spite of a difference in the wire sizes tested. The reasons for this were two-fold.

* The recommended test pressure for a 1,32 mm diameter wire was 3,0 MPa and for a 1,22 mm wire was 2,52 MPa. However, using the recommended test pressure for each wire size would not necessarily allow comparisons to be made between the different wire sizes, since this has not been shown to be valid.

* Samples from each experiment were tested in a random order, so that additional errors would have been introduced if the test pressures had to be adjusted continually (particularly since the pressure gauge on the test apparatus was accurate to not better than 0,1 MPa).

After testing, the results from each experiment were collated and averaged, so that only one replicate of each point was entered for the analysis. Analysis of the data was performed on a Hewlett-Packard (HP) model 9845B mini-computer at Haggie Rand Ltd, using appropriate proprietory HP software. This software could accept a maximum of four factors (satisfactory for this purpose), and the output consisted of all the main effect and interaction means, but did not provide the factor contrasts required for the error analysis. These were therefore calculated using a second specially created programme (Appendix B).

The method for testing the significance of the main effects and interactions, where only a single replication is available, has been described by Natrella (1979), and is given in Appendix B. This procedure uses the contrasts of the three- and four-way interaction effects (which would otherwise be discarded) to calculate a contrast error; these were estimated at the 80% and the 90% confidence limits for each ageing condition. The measured main effects and two-way interactions could then be compared against the contrast errors, to evaluate their statistical significance.

5.4 RESULTS AND DISCUSSION

The results from the factorial analysis will be discussed below, in terms of the effect on the ageing rate of the various factors as measured by the shear elongation and the change in the tensile properties.

It was found that all the recorded tensile properties (i.e. the UTS, the 0,2% proof stress (PS), the relative increase in the 0,2% PS etc) behaved in a similar manner during ageing. The absolute 0,2% PS was found to yield the largest effects, and therefore only these results are discussed here, for simplicity.

The averaged effects of each factor for each ageing treatment (derived using the HP 9845B software) are given in full in Appendix C, and the results from the Yates method of analysis are given in Appendix D.

The overall mean wire properties at each condition are shown in Table 5.5.

TABLE 5.5 : OVERALL MEAN WIRE PROPERTIES AFTER AGEING

Wire Condition	0,2% Proof Stress MPa	Shear Elongation mm
As Drawn	2002	0,47
50°C : 2h	2037	-
500h	2186	0,40
1000h	2229	0,37
80°C : 48h	2262	0,33
100°C : 2h	2206	0,37
24h	2336	0,18

It is interesting to note that ageing at 100°C for 2 hours appeared to give similar aged properties to a treatment at 50°C for 1000 hours (equal to about six weeks). This was a useful result, since it allowed the comparison of the ageing behaviour of wire at 100°C and at a temperature close to ambient, which is more relevant to practical conditions (i.e. during storage of the rope). The activation energy represented by this observation is about 124 $kJmol^{-1}$, which is similar to results previously obtained (see Chapter Four).

The main effect of a factor describes the single effect of that factor on the measured property. Thus the contrast, or main effect, is defined to be the difference between the response means for that factor at each of its two levels.

The two-way interaction contrasts can be described by the following example. Consider the interaction between factor A and factor B, each at two levels, 1 and 2. The combinations can be expressed by $A_i.B_j$ where i and j represent the levels of A and B respectively and the contrast is then defined to be the difference between the sum of all the responses where (i+j) is even (comparison level 1) and where (i+j) is odd (comparison level 2).

The main effects of each factor on the rate of ageing, measured by the 0,2% proof stress and the shear elongation, were derived. These results will be discussed below, following which the significant two-way interactions will be considered.

5.4.1 Drawing Speed

The recorded main effects (i.e. the response mean at each level) of the drawing speed on the properties of high strength steel wire are listed in Table 5.6, and are also graphically represented in Figures 5.2 and 5.3. The defining contrast is then the difference between the two level means; the contrast errors relevant to each ageing treatment, were estimated at two confidence levels, 80% and 90%, and these values are also given in the table.

TABLE 5.6 : MAIN EFFECT OF DRAWING SPEED

	Mean Increase in 0,2% PS /MPa				Mean Shear Elongation/mm			
Wire Treatment	Draw Speed Level 1	2	Error at Confidence 80%	90%	Draw Speed Level 1	2	Error at Confidence 80%	90%
As-Drawn (PS)	(2002) (MPa)	(1983) (MPa)	16	23	0,47	0,47	,019	,025
50°C 2h	38	33	12	16	–	–	–	–
500h	195	173	27	37	0,37	0,43	0,04	0,05
1000h	235	218	24	33	0,35	0,39	0,05	0,06
80°C 48h	265	256	17	23	0,31	0,35	0,04	0,06
100°C 2h	211	196	21	29	0,35	0,39	0,04	0,05
24h	324	338	19	26	0,16	0,20	0,03	0,04

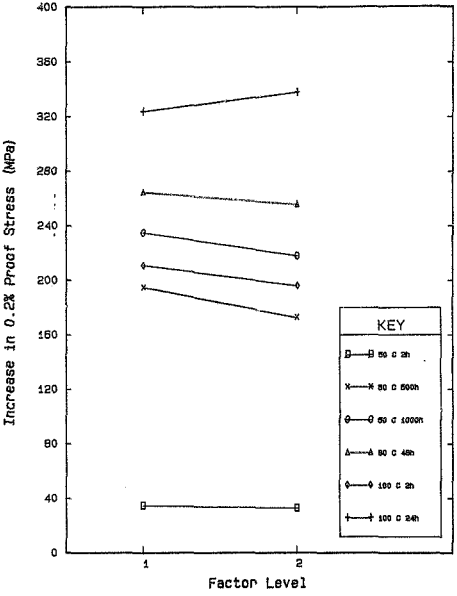


Fig 5.2 Main Effect of Drawing Speed on Proof Stress

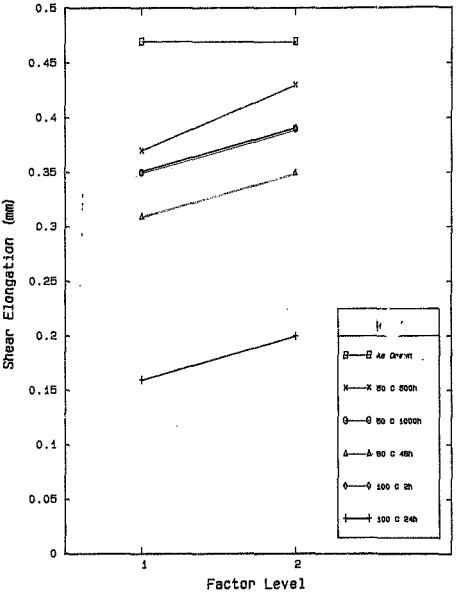


Fig 6.3 Main Effect of Drawing Speed on Shear Elongation

The drawing speed was selected as a factor because of its direct contribution to the strain rate, but it should be noted that an increased drawing speed will also affect other drawing parameters. For example, a higher speed enables the tendency towards hydrodynamic lubrication in drawing, leading to benefits in terms of reduced friction (and hence less heat generated) and possibly lower internal stress fields. The higher speed may also limit the amount of ageing which occurs during drawing (i.e. within or immediately beyond the die), assuming efficient water cooling, since the residence time of the wire at temperature is shortened, and the wire is quenched more rapidly from its peak temperature.

Generally, therefore, we might expect an increased drawing speed to yield better wire properties, both in the as-drawn condition and after ageing. An increased drawing speed was found to reduce the as-drawn 0,2% proof stress significantly, but no effect on the as-drawn shear elongation was measured.

Figure 5.3 clearly shows that, with regard to the shear properties after each ageing treatment, the higher speed was beneficial (measured by a higher shear elongation value); the effect was generally found to be significant at the 90% confidence level. The effect on the 0,2% proof stress (Figure 5.2) was, however, less clear.

5.4.2 Drawing Strain

The measured main effects of the total drawing strain are given in Table 5.7, and these results are also represented graphically by Figures 5.4 and 5.5. As expected, the amount of strain significantly increased the 0,2% proof stress in the as-drawn condition, but the measured shear properties were found to be unaffected (due regard being given to the difficulties in comparing two different wire sizes).

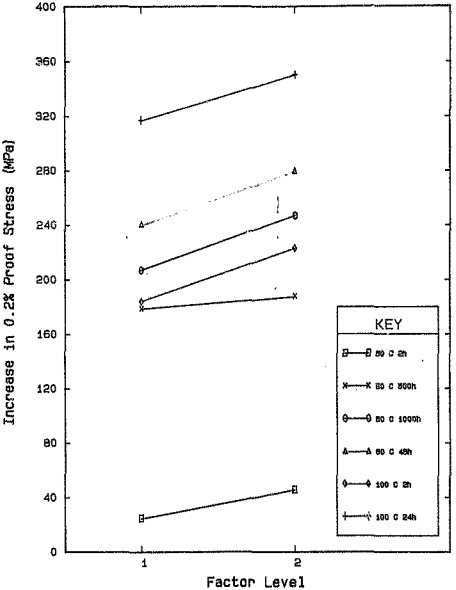


Fig 5.4 Main Effect of Drawing Strain on Proof Stress

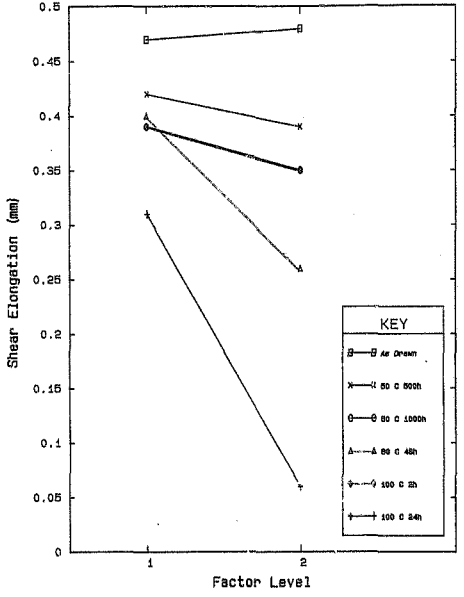


Fig 5.5 Main Effect of Drawing Strain on Shear Elongation

TABLE 5.7 : MAIN EFFECT OF TOTAL DRAWING STRAIN

	Mean Inc in 0,2% PS/MPa				Mean Shear Elongation/mm			
Wire Treatment	Drawing Strain Level 1	2	Error at Confidence 80%	90%	Drawing Strain Level 1	2	Error at Confidence 80%	90%
As-Drawn (PS)	(1960) (MPa)	(2044) (MPa)	16	23	0,47	0,48	,019	,025
50°C 2h	25	46	12	16	–	–	–	–
500h	179	188	27	37	0,42	0,39	0,04	0,05
1000h	207	247	24	33	0,39	0,35	0,05	0,06
80°C 48h	241	280	17	23	0,40	0,26	0,04	0,06
100°C 2h	184	223	21	29	0,39	0,35	0,04	0,05
24h	317	350	19	26	0,31	0,06	0,03	0,04

The increase in the proof stress after ageing was found to be consistently and significantly higher in the smaller wire, as would be expected from previous results (see Section 4.2.2).

The detrimental effect of a higher strain on the ageing response was more clearly evident in the shear properties. These showed a marked decrease at the higher strain after ageing, while the *lesser strained* wire maintained reasonable shear properties for a longer ageing period. The difference between the two samples increased steadily as the ageing treatment became more severe.

The influence of the higher drawing strain on the rate of ageing may be due to a more refined dislocation cell size, or to the reduction in the pearlite interlamellar spacing after drawing. Both of these factors may increase the kinetics of ageing by reducing the mean ferrite free path. However, the apparently sharp deterioration in the ageing properties may be a result of some degradation in the wire microstructure in the final pass (e.g. cementite fragmentation – see S[illegible],3.3.2).

5.4.3 Aluminium

The motivation for the addition of aluminium was to tie up free nitrogen and so to lessen its detrimental effect on ageing. On its own, aluminium might be expected to harden the ferrite considerably by solid solution strengthening, while it should also increase hardenability mildly.

With nitrogen, aluminium should refine the austenite grain size, by the precipitation of aluminium nitride particles at the grain boundaries. Table 5.4, however, clearly indicates that the addition of aluminium had no effect on the patented properties of the steels, and no reasons for this were apparent from the microstructures.

The influence of aluminium alone on the rate of ageing in drawn steel wire is illustrated in Table 5.8 and Figures 5.6 and 5.7. Except for a small detrimental effect in the shear test at severe ageing treatments (i.e. 100° for 24 hours), aluminium had no apparent observable effects on either of the mechanical properties, in both the as-drawn condition and after ageing.

TABLE 5.8 : MAIN EFFECT OF ALUMINIUM

	Mean Inc in 0,2% PS/MPa				Mean Shear Elongation/mm			
Wire Treatment	Aluminium Level 1	Aluminium Level 2	Error at Confidence 80%	Error at Confidence 90%	Aluminium Level 1	Aluminium Level 2	Error at Confidence 80%	Error at Confidence 90%
As-Drawn (PS)	(2002) (MPa)	(2002) (MPa)	16	23	0,47	0,47	,019	,025
50°C 2h	32	39	12	16	-	-	-	-
500h	179	189	27	37	0,41	0,40	0,04	0,05
1000h	223	231	24	33	0,37	0,37	0,05	0,06
80°C 48h	265	256	[illegible]	23	0,32	0,34	0,04	0,06
100°C 2h	204	202	21	29	0,38	0,36	0,04	0,05
24h	336	331	19	26	0,22	0,14	0,03	0,04

5.4.4 Nitrogen

Nitrogen has previously been shown to have a detrimental effect on the rate of ageing of a cold-drawn plain carbon steel (see Sections 4.2.5 and 4.3.3). The influence of nitrogen on the rate of ageing of steel wire in this study is shown in Table 5.9 and Figures 5.8 and 5.9.

The high nitrogen material was consistently found to have a lower strength than the low nitrogen material, although the shear elongation values were identical. This is difficult to rationalise on the basis of accepted theories of ageing, since one might expect that nitrogen, if present in reasonable

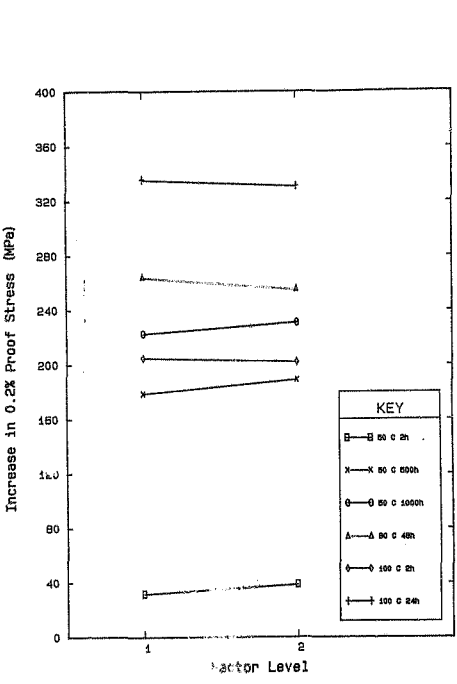


Fig 5.6 Main Effect of Aluminium on Proof Stress

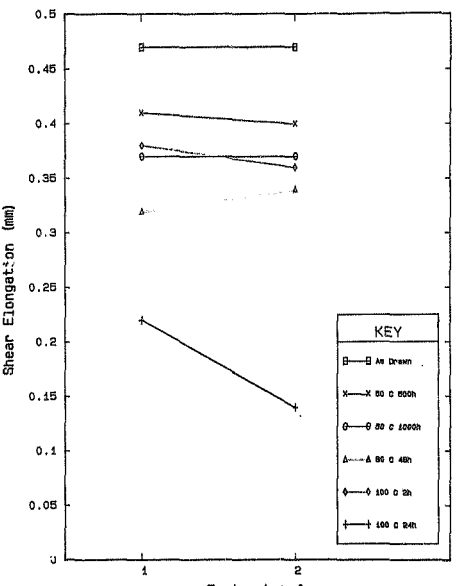


Fig 5.7 Main Effect of Aluminium on Shear Elongation

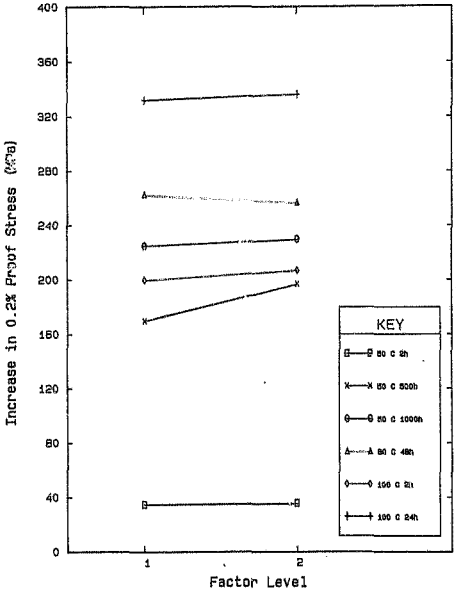


Fig 5.8 Main Effect of Nitrogen on Proof Stress

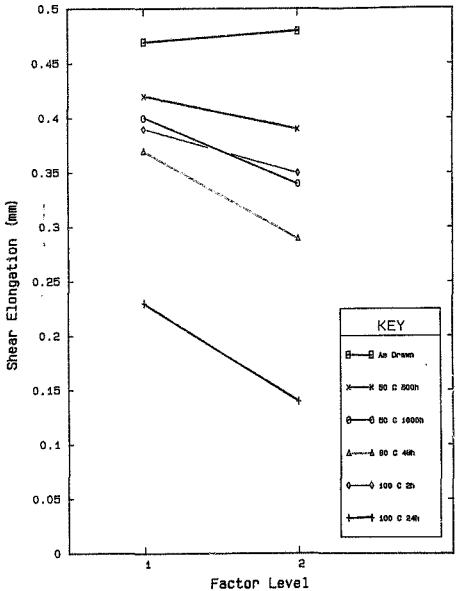


Fig 5.9 Main Effect of Nitrogen on Shear Elongation

quantities, might contribute to ageing during the drawing process, which would serve to increase the yield stress in the as-drawn condition. No obvious differences in chemical composition (see Table 5.4) could account for the phenomenon.

TABLE 5.9 : MAIN EFFECT OF NITROGEN

	Mean Inc in 0,2% PS/MPa				Mean Shear Elongation/mm			
Wire Treatment	Nitrogen Level 1	2	Error at Confidence 80%	90%	Nitrogen Level 1	2	Error at Confidence 80%	90%
As-Drawn (PS)	(2020) (MPa)	(1984) (MPa)	16	23	0,47	0,48	,019	,025
50°C 2h	35	36	12	16	-	-	-	-
500h	170	197	27	37	0,42	0,39	0,04	0,05
1000h	225	230	24	33	0,40	0,34	0,05	0,06
80°C 48h	263	257	17	23	0,37	0,29	0,04	0,06
100°C 2h	200	207	21	29	0,39	0,35	0,04	0,05
24h	332	336	19	26	0,23	0,14	0,03	0,04

The behaviour of the proof stress during ageing (Figure 5.8) did not reveal any significant effect of an increase in the nitrogen content, on the ageing rate. This result was unexpected in the light of the accepted dominant effect of nitrogen on the rate of ageing at these temperatures in lower carbon steels. It also appears to contradict the previous results from commercial steels (Section 4.2.5), which showed that an increased nitrogen content accelerated the reaction kinetics when the reaction rate was measured by the tensile properties.

The detrimental effect of nitrogen does emerge, however, when the shear test results are examined (Figure 5.9). Thus it is apparent that the presence of reasonable amounts of nitrogen is likely to lead rapidly to embrittlement of the wire; and this does support the results obtained previously.

5.4.5 Two-Way Interactions

As previously described, Yates' (1937) method of analysis provides a convenient method of identifying the significant interactions between the various factors in the design, in the same way that the significant main effects can be isolated.

The significant interactions identified by this method at the 80% confidence limit are given in Table 5.10. As an example, Table 5.10 shows that the only significant interaction (with 80% confidence) occurs between the aluminium content (b) and the drawing strain (c), on the effect of an ageing treatment of 1000 hours at 50°C on the 0,2% proof stress.

TABLE 5.10 : THE SIGNIFICANT TWO-WAY INTERACTIONS AT THE 80% CONFIDENCE LIMIT

Wire Condition	0,2% Proof Stress	Shear Elongation
As-Drawn	ab ac ad bc	bd cd
50°C 2h	ab ac cd	–
500h	bc cd	ac bc cd
1000h	bc	ac ad
80°C 48h	bc	ac bc ad cd
100°C 2h	bc	bc ad
24h	None	bd

a : Nitrogen Content
b : Aluminium Content
c : Drawing Strain
d : Drawing Speed

It is apparent that the two-way interactions are not clearly defined, and tend to vary with the ageing treatment applied. The mechanical property considered also has some influence on the results. Thus, for instance, after 1000 hours at 50°C, an interaction between the aluminium content and the drawing strain is apparent from the proof stress results, but not from the shear test results.

The significant interactions in Table 5.10 can probably be summarised as follows.

Tensile Properties: aluminium and drawing strain (general); drawing strain and drawing speed (light ageing treatments only).

Shear Properties: aluminium and drawing strain (general); nitrogen and drawing strain (light ageing treatments only); nitrogen and drawing speed (light ageing treatments only).

5.4.5.1 Aluminium and Nitrogen

No consistent interaction between the aluminium and nitrogen contents on the ageing rate was observed; this was unexpected, since the aluminium additions were made to tie up the free nitrogen as aluminium nitride and so remove the influence of nitrogen. This result is evident from Table 5.11, which shows the comparison level means (i.e. at the "even" level and at the "odd" level - see Section 5.4) for the interaction at each ageing treatment, and the estimated errors at the 80% confidence limit. As before, the defining contrast is the difference between the comparison level means.

TABLE 5.11 : DEFINING CONTRASTS FOR THE TWO-WAY INTERACTION - ALUMINIUM VS NITROGEN

	Mean Inc in 0,2% PS MPa			Mean She[illegible]tion		
	Comparison Level Means			Comparison [illegible]eans		
Wire Condition	Even (1)	Odd (2)	Contrast Error	Even (1)	Odd (2)	Contrast Error
As-Drawn (PS)	(1994) (MPa)	(2010) (MPa)	16	0,468	0,478	0,019
50°C 2h	43	28	12	-	-	-
500h	175	193	27	0,40	0,40	0,04
1000h	226	228	24	0,36	0,38	0,05
80°C 48h	261	260	17	0,33	0,33	0,04
100°C 2h	209	199	21	0,36	0,38	0,04
24h	330	338	19	0,19	0,17	0,03

If no interaction exists then aluminium additions would probably not serve any useful purpose in restricting ageing in commercial steels. However, it has been pointed out by Langenscheid (1979) (Section 2.2.5.2) that the precipitation of aluminium nitride from solution is most effective if the steel is held at about 750°C for a few minutes before cooling to room temperature. The precipitation rate at the normal pearlite transformation temperatures of about 550°C would be exceptionally slow, due to the restricted kinetics of the reaction. This factor would effectively explain the lack of any beneficial effect of aluminium in the nitrogen-charged steel.

Therefore, for aluminium to have any effect on ageing a separate heat treatment might have to be interposed in the normal production process: i.e. quenching to about 750°C from the austenitising temperature, and maintaining this temperature for a few minutes before the steel is again quenched to the isothermal transformation temperature.

5.4.5.2 Strain and Nitrogen

An interaction was noted between the drawing strain and the nitrogen content, although statistically the interaction is only significant for the shear test results at relatively light ageing treatments. The two-way interaction means at the two comparison levels are given in Table 5.12 and Figures 5.10 and 5.11. The interaction is readily apparent from the figures, where an interaction is identified by an inclined response line.

The results here imply that the nitrogen content and drawing strain have some synergistic effect on the rate of ageing, such that a combination of the two factors is greater than the sum of the individual effects. This would not be unexpected if some strain-enhanced diffusion process is present, since an increase in strain would therefore assist the diffusion of nitrogen.

TABLE 5.12 : DEFINING CONTRASTS FOR THE TWO-WAY INTERACTION - STRAIN VS NITROGEN

	Mean Inc in 0,2% PS MPa			Mean Shear Elongation mm		
	Comparison Level Means			Comparison Level Means		
Wire Condition	Even (1)	Odd (2)	Contrast Error	Even (1)	Odd (2)	Contrast Error
As-Drawn (PS)	(1991) (MPa)	(2014) (MPa)	16	0,474	0,472	0,019
50°C 2h	47	24	12	..	..	..
500h	201	166	27	0,38	0,43	0,04
1000h	233	223	24	0,34	0,40	0,05
80°C 48h	264	256	17	0,26	0,40	0,04
100°C 2h	213	198	21	0,36	0,37	0,04
24h	340	327	19	0,17	0,20	0,03

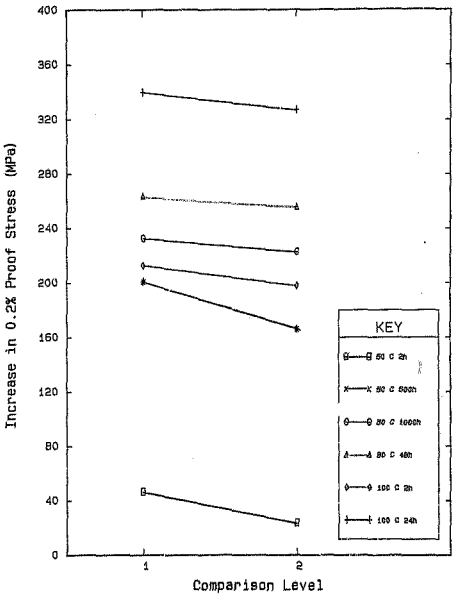


FIGURE 5.10 : MEAN INCREASE IN 0,2% P.S. AT EACH COMPARISON LEVEL FOR THE TWO-WAY INTERACTION BETWEEN DRAWING STRAIN AND NITROGEN CONTENT.

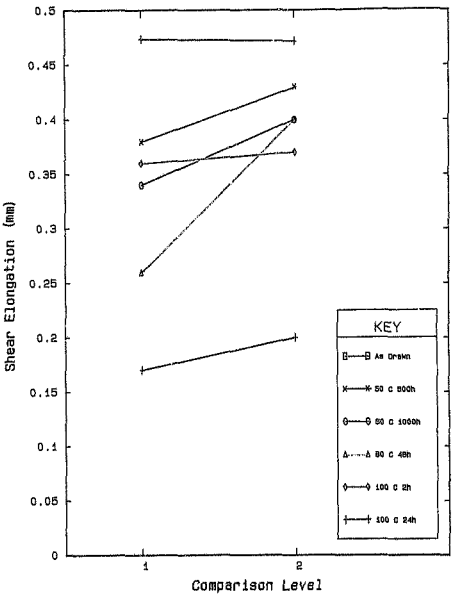


FIGURE 5.11 : MEAN SHEAR ELONGATION AT EACH COMPARISON LEVEL FOR THE TWO-WAY INTERACTION BETWEEN DRAWING STRAIN AND NITROGEN CONTENT.

5.4.5.3 Strain and Speed

An interaction between the drawing speed and the drawing strain was noted, although again the interaction was only significant at relatively light ageing treatments. The interaction means are shown in Table 5.13 and Figures 5.12 and 5.13.

TABLE 5.13 : DEFINING CONTRASTS FOR THE TWO-WAY INTERACTION - STRAIN VS SPEED

	Mean Inc in 0,2% PS MPa			Mean Shear Elongation mm		
	Comparison Level Means			Comparison Level Means		
Wire Condition	Even (1)	Odd (2)	Contrast Error	Even (1)	Odd (2)	Contrast Error
As-Drawn (PS)	(2010) (MPa)	(1994) (MPa)	16	0,486	0,460	0,019
50°C 2h	28	44	12			
500h	159	208	27	0,43	0,38	0,04
1000h	219	235	24	0,39	0,35	0,05
80°C 48h	256	265	17	0,36	0,30	0,04
100°C 2h	195	213	21	0,36	0,36	0,04
24h	327	341	19	0,17	0,20	0,03

The higher drawing speed effectively reduces the detrimental effect of the higher drawing strain, and the effect is quite dramatic in certain instances (e.g. at 80°C for 48 hours). It is not possible to say whether this is due to a beneficial effect of the higher strain rate, or due to the presumeably improved lubrication conditions within the die at the higher speed. If there is some destructive event which occurs in the last drawing stage to the higher strain of 2,4 (Section 5.4.2), then this might be retarded by the higher drawing speed, leading ultimately to an improved ageing response.

5.4.5.4 Aluminium and Strain

A generally significant interaction between the aluminium content and the drawing strain was determined, and the relevant data is shown in Table 5.14.

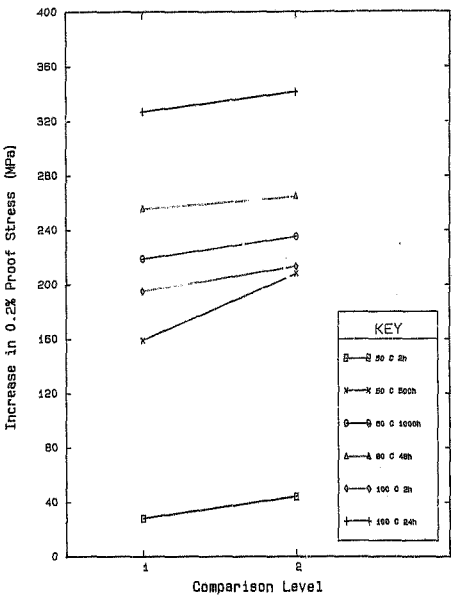


FIGURE 5.12 : MEAN INCREASE IN 0,2% P.S. AT EACH COMPARISON LEVEL FOR THE TWO-WAY INTERACTION BETWEEN DRAWING STRAIN AND DRAW SPEED.

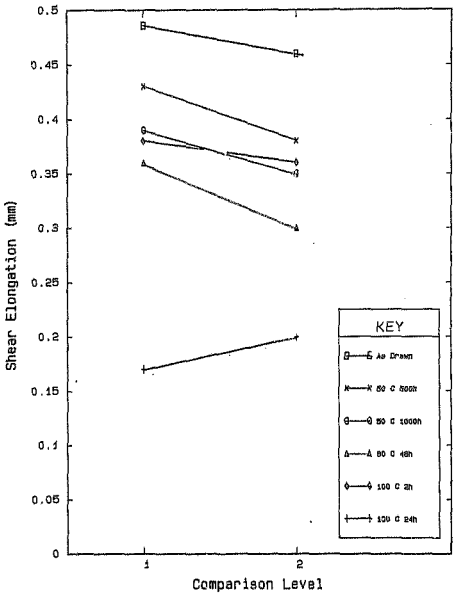


FIGURE 5.13 : MEAN SHEAR ELONGA* : AT EACH COMPARISON LEVEL FOR THE TWO-WAY INTERACTION BETWEEN *DRAWING STRAIN AND* DRAW SPEED.

TABLE 5.14 : DEFINING CONTRASTS FOR THE TWO-WAY INTERACTION - STRAIN VS ALUMINIUM

	Mean Inc in 0,2% PS MPa			Mean Shear Elongation mm		
	Comparison Level Means			Comparison Level Means		
Wire Condition	Even (1)	Odd (2)	Contrast Error	Even (1)	Odd (2)	Contrast Error
As-Drawn (PS)	(1989) (MPa)	(2015) (MPa)	16	0,473	0,472	0,019
50°C 2h	30	28	12	-	-	-
500h	188	193	27	0,38	0,43	0,04
1000h	213	241	24	0,34	0,40	0,05
80°C 48h	250	271	17	0,35	0,31	0,04
100°C 2h	191	216	21	0,35	0,39	0,04
24h	326	342	19	0,19	0,18	0,03

It is evident from Table 5.14 that the "direction" of the interaction is not constant, so that for some treatments a high aluminium content is beneficial to the rate of ageing, while in others the converse is true. The interaction can therefore be discounted, since it is probably an artifact of the testing procedure, and is therefore due to experimental errors.

5.5 SUMMARY

In summary, the factorial experiments have shown the following significant effects.

* An increased drawing speed (in the range considered) has a beneficial effect on the shear properties of aged wire, but no apparent effect on the as-drawn values. Conversely, the as-drawn proof stress was lowered by a higher drawing speed, but the aged properties were only slightly affected.

* A high level of drawing strain has a definite detrimental effect on the ageing rate of steel wire, in terms of the rate of increase of the 0,2% proof stress and the rate of decrease of the shear properties.

* Aluminium additions to high carbon steel wire processed in the usual way has no measurable effect on either the as-drawn or the ageing properties, whether or not nitrogen was present in large quantities (of about 0,01%).

* Nitrogen does not appear to have any effect on the ageing tendency of steel wire as measured by the tensile properties. However, the transverse properties measured by the shear test were adversely affected by high nitrogen contents.

* A synergistic effect of strain and nitrogen has been determined, such that the deleterious effect of a high drawing strain is exacerbated by a high nitrogen content.

* A high drawing speed seems to lessen the detrimental effect of high drawing strains. Thus, the beneficial effects of speed are more significant at high strains.

It is important to be aware that the relationships derived from this work cannot be described as definitive since only two levels of each factor were investigated.

CHAPTER SIX : SUMMARY AND CONCLUSIONS

6.1 INTRODUCTION

This chapter will attempt to summarise the work performed on this project, and to discuss the overall results obtained. Some comments on the results at each stage of the investigation have already been offered, so that the discussion presented here will be brief; further details will be found in the relevant sections of the text.

6.2 EXPERIMENTAL PROCEDURE

In any project, the quality of the experimental procedure determines the quality of the final results. With this work in particular, the response of a steel wire to ageing after drawing was found to be very sensitive to the manner in which the sample was prepared and tested, and, therefore, attention must be paid to the starting material, the drawing procedure and the testing procedure.

The work on the vacuum-melted steels (Sections 4.3 and 4.4) emphasised the requirement for consistent material properties along the length of the wire sample. In this work, a marked variance in the properties of the feed materials was evident after heat treating in the small-scale facilities available. This was partly due to small variations in the heat treatment conditions at different points in the wire length. This scatter was exacerbated in the drawn wire, due to different responses to drawing strain (i.e. different work hardening rates along the wire length, and across the wire cross-section).

Consideration must also be given to macro-segregation effects, especially in alloy steels, and also to the surface quality (i.e. rolling defects). Both of these factors accounted for the discard of a substantial proportion of the vacuum-melted material, and therefore restricted the number of tests which could be performed on any one steel batch. The ultimate effect of these factors on the scatter in the wire properties could not be ascertained.

The method of wire sample preparation from the starting material may have a significant effect on the as-drawn wire properties, and on the ageing characteristics. For instance, the effect of a small final reduction in the drafting schedule had little apparent effect on the as-drawn properties measured (Section 4.2.2), but did have a significant effect on the ageing properties (as measured by the shear test).

The slow drawing of wire on a tensile test machine, to control the temperatures in the wire during drawing, was investigated (Section 3.2.3), but no apparent advantage over conventional wire preparation was found in terms of the ageing behaviour. In addition, the wire produced was considered to be unrepresentative of production material, due to inferior lubrication.

A commercial, single-hole drawing machine capable of a wide range of drawing speeds was found to be adequate, as long as efficient direct wire cooling was used. The drawing speeds used here were generally about 2 to 4 $m.min^{-1}$, which were about two orders of magnitude higher than those available on the tensile machine, but were still low when compared to production machines. The lubrication conditions, however, appeared to be satisfactory on examination of the wire surface after drawing. Close control of the drawing conditions was maintained by changing the starting material size to 4 mm diameter.

Ageing treatments were carried out over a range of temperatures in order to determine the temperature dependence of the ageing process. However, the highest temperature used was just 150°C, which is representative of the wire temperatures encountered in the die during drawing.

It had been proposed that ageing at high temperatures might initiate some ageing mechanism not significant under normal conditions (i.e. "natural ageing" at ambient temperatures). However, the validity of using elevated temperatures to determine the rate of ageing after drawing was confirmed when comparative tests on wire aged at temperatures close to ambient showed no deviation from the temperature dependence for the reaction derived at higher temperatures.

The method of artificially ageing the wires in a silicon oil bath was found to be entirely satisfactory, and close control of the ageing temperatures was possible.

The method of detection of ageing proved to be a significant problem. Initial attempts to monitor the progress of ageing by measuring the change in the electrical resistivity had to be abandoned due to inadequacies in the test procedure, which gave unreliable results.

The increase in the specimen tensile strength was thus used for the primary measure of ageing, and was found to be satisfactory until problems arose with premature wire failures in the grips of the testing machine. This effectively restricted the use of the tensile strength to reasonably ductile wire which had not been severely aged.

The 0,2% proof stress was therefore determined regularly, and was found to give satisfactory results. The proof stress has a larger response to ageing than the UTS, although this was partially offset by the increased variability in this property. The proof stress determination therefore required more wire sample per "condition" to ensure reliable results.

The shear test was also used in later tests, after it was noted that there was no apparent correlation between the tensile test and the shear test. The tensile properties generally showed a gradual increase during ageing, up to the maximum level; the shear test, on the other hand, was found to be more markedly affected, if the wire was "susceptible" to ageing (i.e. embrittlement).

For instance, the effect of a low final reduction in the drafting schedule was only evident from the shear test results. This led to the conclusion that the transverse properties are affected in a different way to the tensile properties.

6.3 SUMMARY OF RESULTS AND DISCUSSION

6.3.1 Commercial Plain Carbon Steels

The initial work on the commercial plain carbon steels (Section 4.2) revealed the following results.

1. The temperature-dependence of the ageing rate, as measured by the UTS, can be characterised by an Arrhenius-type relationship, indicating that the reaction is thermally activated. This would be expected if the normal mechanism of strain ageing in steels (i.e. the migration of carbon atoms to dislocation sites) holds for eutectoid steels drawn to high strains. The derived activation energy for a small amount of ageing did not correspond with that accepted for conventional carbon diffusion in ferrite (i.e 84 $kJmol^{-1}$), although the results did correspond very well with Yamada's (1976) value for "second stage" ageing in high carbon steel wire, of 117 $kJmol^{-1}$ (see Section 2.4.2). No "first stage" ageing, attributed by Yamada to conventional "Cottrell" strain ageing, was apparent in any of the tests performed.

2. An empirical rate equation proposed by Johnson and Mehl (Section 4.2.1) was found to characterise the kinetics of the ageing reaction. The rate exponent in the Johnson-Mehl equation was derived for certain instances and was found to be between 0,25 and 0,30, depending on the temperature of ageing (as expected since it represents a frequency factor for the process). According to the literature (Kemp, 1987), this value of the rate exponent is equivalent to that for carbon diffusion in the ferrite to planar arrays of dislocations at the ferrite/cementite lamellar interface. This observation was based on a model proposed for the early stages of tempering (Lement and Cohen, 1956), for which an activation energy of 113 $kJmol^{-1}$ has been derived.

This mechanism is not, however, likely, since the diffusion behaviour of carbon in ferrite is known to be different to that for diffusion in martensite. Thus, the similarity between the data characterising each of the reactions, is probably coincidental.

3. The level of drawing strain (Section 4.2.2) was found to increase the kinetics of the ageing process. Tensile tests were performed at a variety of strains, from 1,17 to 2,16, after ageing at 100°C. The slopes of the response curves were similar, indicating that the rate constant was probably not influenced by the drawing strain.

4. The effect of the drawing schedule was noted on the shear properties (as has already been described), such that a low reduction in the final pass appeared to delay the onset of embrittlement as measured by this test. The effect was not observed in the tensile test results.

   There are two possible explanations for this effect: a lower final reduction is equivalent to a higher strain rate in the last die (and hence to an effective lower temperature of deformation), and also may give a modified internal stress field in the wire. It is possible that the wire behaviour in the shear test is itself dependent on the internal stress field, but this may also lead to a "differential ageing rate" across the wire section, if the ageing rate is strain-enhanced.

5. Tests on a steel wire containing 0,7% carbon showed rate exponents of 0,28 and 0,31 at two levels of drawing strain, results which were marginally higher than those obtained for the 0,8% carbon steel. This may infer that the reaction kinetics are increased by higher volume fractions of ferrite, but this is uncertain.

6. It was considered possible that the supersaturated carbon in the ferrite after heat treatment might be an important factor in post-draw ageing, and an attempt was made to reduce this by warming the steel prior to drawing, at 200°C for several hours.

A small increase in the feed material UTS was observed, but this was not reflected in the drawn wire, and no effect on the ageing behaviour was apparent. This result infers that the carbon held in supersaturation probably migrated to dislocation sites and precipitated there during the annealing treatment, thus accounting for the observed increase in the feed UTS. On drawing, the dislocations would be pulled away from these precipitates, and thus carbon would again effectively be in supersaturation. Thus, no beneficial effect on the rate of ageing would be apparent.

This observation was confirmed by the fact that at all times an activation energy for ageing was determined to be about 117 $kJmol^{-1}$, and this does not correspond with that for carbon diffusion in ferrite (84 $kJmol^{-1}$). Thus, conventional carbon diffusion is unlikely to be the controlling mechanism in the ageing reaction.

7. The effect of the nitrogen content on the rate of ageing was investigated in two steels, one containing 0,006% nitrogen, and the other 0,01%. No difference in the measured energies of activation was apparent, indicating that nitrogen does not affect the rate-controlling step in the reaction. However, the kinetics of the reaction were accelerated by an increase in the nitrogen content.

6.3.2 Vacuum-Melted Plain Carbon Steels

Some additional work was performed on specially prepared vacuum-melted steels, with controlled compositions. Here, the effect of nitrogen was investigated further, while an attempt was also made to establish the effect of the pearlite interlamellar spacing on the rate of ageing. This work was described in Section 4.3.

The two levels of nitrogen investigated here were 0,0015% and 0,034%. The study clearly demonstrated that high levels of nitrogen are detrimental to the rate of ageing in drawn steel wire, particularly in terms of the transverse ductility; the tensile properties were, however, little changed.

In Section 4.3.5.2, it was proposed that nitrogen affects the transverse ductility particularly because of a possible embrittling effect of the grain or colony boundaries or of the interfacial regions of the pearlite lamellae, resulting in "directional" embrittlement.

Nitrogen is noted for its general detrimental effect on steel ductility during strain ageing, especially in mild steels at temperatures below 100°C (Section 2.2.4). However, if the above mechanism does occur, it is apparent that it is not the rate-controlling step in the ageing process since the activation energy was unaffected by the nitrogen content, and therefore, another mechanism may be operating alongside "nitrogen ageing".

Leading on from this, the pearlite interlamellar spacing was found to have some influence on the rate of ageing: a finer interlamellar spacing prior to drawing was noted to yield a higher rate of increase in the UTS. The possible reasons for this are outlined below.

The pearlite interlamellar spacing affects the strength of the feed material, and of the drawn wire by affecting the work-hardening rate during drawing. Thus, the dislocation substructure generated during drawing is probably affected by the pearlite spacing, in terms of the dislocation density, the cell wall thickness and the mean cell size. If the ageing mechanism is controlled by carbon dissolution and migration from the cementite (as has been suggested by Yamada (1976) and others) then the rate of ageing (i.e. the kinetics) might be improved by shortening the mean ferrite free path for diffusion.

Alternatively, as proposed by Waugh et al (1981), the rate of increase in the tensile properties is due to refinement of the dislocation substructure. This is likely to be dependent to some extent on the pearlite spacing, and therefore ageing would be pearlite spacing dependent. It might be noted that Waugh et al did not detect any evidence of carbon migration and precipitation during ageing, using atom-probe techniques.

This mechanism might also account for the apparent marked effect of the residual stress level in the wire after drawing since the movement of dislocations requires the superimposition of a stress, such as can be supplied by internal stresses. The amount of redundant work during drawing might also affect the dislocation substructure.

This mechanism is similar to that proposed for second-stage creep (Reed-Hill, 1973). The activation energy for the migration of dislocations is a function of the activation energy for self-diffusion (about 250 $kJmol^{-1}$ for ferrite) and of the level of the applied stress. It is not known if the derived activation energies obtained in this work (of about 117 $kJmol^{-1}$) can be accounted for by this mechanism.

According to Kemp (1987), the Johnson-Mehl rate exponent for thermally activated dislocation movement is about 2, which is of course very different to that derived in this study (about 0,3).

6.3.3 Vacuum-Melted Steels Containing Silicon

The effect of limited additions of silicon to the steel was investigated by adding 0,5%, 1% and 2% silicon to a 0,8% carbon, 0,6% manganese steel base composition (Section 4.4). Initial tests were performed on wire heat-treated under factory conditions (i.e. isothermally transformed at 560°C), while subsequent tests were also performed on the 1% and 2% silicon steels transformed at 610°C.

It was apparent from the works-patented material that the addition of 0,5% silicon increased the rate of ageing in the drawn wire, when compared to the steel containing no silicon. A small, but possibly significant, reduction in the activation energy for ageing was measured with the addition of silicon, which may account for the increased ageing rate.

Silicon is known to increase the carbon activity in iron, so that carbon precipitation is encouraged. By Le Chatelier's principle, this will encourage cementite dissolution and therefore carbon atoms will be supplied for ageing (silicon is known to assist graphitisation in cast irons).

This factor may also account for Yamada's (1976) observation that the value of the binding energy of carbon to cementite which he derived was slightly lower than expected in plain carbon steels (Yamada attributed this observation to cementite fragmentation at high drawing strains). The normal level of silicon additions to commercial steels (of about 0,3%) is apparently sufficient to affect the activation energy for ageing in this way.

Silicon additions of 1% and higher appeared to have a beneficial effect on the ageing rate, such that a 1% addition showed similar behaviour to the 0% silicon steel, and a 2% addition showed improved ageing behaviour. This result is not definitive, since only a limited number of tests could be performed due to a shortage of sample.

It has been proposed (Smith, 1986) that the beneficial effect of silicon may be due to the lattice strain in the ferrite around a silicon atom in solution effectively trapping carbon atoms, and therefore restricting ageing.

6.3.4 *Factorial Experiments*

A series of experiments was designed to investigate the effects of four factors on the rate of ageing, in an effort to confirm the main effects and the interactions between the factors. These factors were; the drawing speed, the total drawing strain, the aluminium content and the nitrogen content. The factors were investigated at two levels only for a total of 16 experiments. These were analysed statistically, and the following results were obtained.

6.3.4.1 *Drawing Speed*

A high drawing speed was noted to be beneficial to the ageing properties, and in particular, to the wire behaviour in the shear test. The as-drawn properties were, however, apparently unaffected by the drawing speed.

The observed behaviour could be likened to that noted in a previous experiment, where a low final reduction of area in the drafting schedule was found to improve the ageing response, especially in the shear test.

It should be noted here that the test conditions were not representative of production conditions. The drawing speeds used were 4 $m.min^{-1}$ and 1 ms^{-1}, and these were kept constant at each pass. In addition, the wire temperature was reduced to ambient between each die by efficient water cooling.

There are three possible reasons for the beneficial effects of a high drawing speed on ageing. These are a higher strain rate; an improved internal stress field after drawing; and a reduced amount of ageing during the drawing process.

A higher strain rate can be considered to be equivalent to deformation at a lower temperature, and thus can be likened to drawing with more efficient cooling. This has been shown to be beneficial to the ageing properties (Section 2.5,2), by restricting ageing during drawing.

The improved lubrication conditions associated with a higher drawing speed are likely to affect the internal stress field remaining in the wire after drawing. This may have some effect on carbon diffusion or cementite dissolution, and will also affect dislocation migration, if this occurs during ageing.

The restriction of ageing during drawing has been proposed on the basis that the wire will enter the cooling water more rapidly at a higher drawing speed. This mechanism is perhaps the least likely, since this would be expected to show some influence on the as-drawn properties, and none was evident.

It is not certain which of the above mechanisms is valid, and, indeed, they may operate simultaneously. Thus, it appears that modifying the internal stress field and a higher strain rate may be beneficial, and both of these can also be achieved by lowering the reduction in area per drawing pass, and also by increasing the die angle.

6.3.4.2 Drawing Strain

The total strain applied to a wire, in combination with the as-patented steel strength, and the amount of ageing which occurs during drawing, effectively determines the strength of the finished product. In the effort to increase the strengths of cold-drawn plain carbon steel wire, higher values of strain have been utilised, and it is with these high strains that the problem of severe wire embrittlement due to ageing has become apparent (Shipley, 1985). A recommended maximum true strain has been set at about 2,27, based on experience at Haggie Rand Ltd.

The strain levels under investigation in this experiment were 2,22 and 2,38, which fall on either side of this limit. These strains correspond to tensile strengths of about 2150 MPa and 2250 MPa respectively, in the as-drawn wire.

The results of the investigation showed that a small difference in the drawing strain at this level can have a substantial effect on the ageing behaviour of plain carbon wire, especially as regards the transverse properties. The shear test does not generally show a gradual decrease in the transverse ductility of wire drawn to low strains. However, in wire susceptible to ageing, a gradual fall-off in the shear elongation with ageing occurs, followed by a dramatic fall to zero elongation over a short time interval after a certain time has elapsed. Therefore the higher strain advances the ageing rate such that the rapid deterioration in ductility occurs within a "reasonable" time (i.e. within the storage life of the rope); and the maximum recommended drawing strain of 2,27 may therefore be valid.

Earlier work has demonstrated, however (see Section 6.3.1 of this chapter), that the nominal total strain is not the only factor to be considered, since some interaction probably exists between factors such as the total strain, the drawing speed, the reduction in area per pass and the die angle, especially where the shear test ageing response is considered. It should also be noted that the total strain applied to the wire depends on the proportion of redundant work performed.

6.3.4.3 Aluminium Additions

Steels with aluminium additions of 0,1% were compared with steels containing no aluminium. On a stoichiometric basis, 0,1% additions were more than sufficient to tie up all the available nitrogen as aluminium nitride.

With no nitrogen present aluminium would be expected to strengthen the ferrite by solid solution strengthening, while with nitrogen present, aluminium would be expected to combine with the nitrogen to form AlN, and precipitate on the grain boundaries, where it would serve to restrict grain growth during austenitising.

No beneficial effects of aluminium on the strength properties could be observed, which was unexpected. Aluminium was also shown to have no beneficial effect on the ageing rate, whether nitrogen was present or not. It is likely, therefore, that aluminium nitrides were not formed, so the influence of nitrogen was not removed from the ageing process.

According to Langenscheid (1979), aluminium is not precipitated effectively at the normal isothermal transformation temperatures applied to steel wire, and an additional intermediate heat treatment at about 750°C for a few minutes would be required before quenching to the isothermal transformation temperature. This aspect was not investigated.

6.3.4.4 The Nitrogen Content

The detrimental effect of nitrogen on the ductility of steel wire was again shown by steels containing of 0,01% nitrogen, when compared to steels containing only 0,0015% nitrogen. This result generally confirmed earlier work on both vacuum-melted and commercial steels, and these results have been discussed in detail previously. A mechanism for the particular effect of nitrogen on the transverse ductility has been proposed.

6.3.4.5 Interactions Between Factors

Two possibly significant two-way interactions were identified by this study; strain with nitrogen and drawing speed with strain. All the higher order interactions were too prone to error for significance, and were not considered.

The drawing strain appeared to have a synergistic detrimental effect with a high nitrogen content on the ageing rate. This was not unexpected, since each factor has been previously shown to have a negative effect on the ageing rate.

A high drawing speed was found to reduce the detrimental effect of a high drawing strain, or, considered another way, the beneficial effects of a high drawing speed were more significant at the higher strain. The reasons for this are unclear.

6.4 CONCLUSIONS

The following conclusions regarding the ageing behaviour of cold-drawn pearlitic steel wire can be drawn from the results of this study.

1. The temperature dependence of the rate of ageing as measured by the increase in the tensile properties can be represented by an Arrhenius-type relationship, with an activation energy of about 117 $kJmol^{-1}$. This values agrees very well with literature values for strain ageing in high carbon steel wire.

2. A time exponent for the ageing kinetics was derived, and was found to be in the range of 0,25 to 0,30, depending on the temperature of ageing under consideration.

3. Reducing the level of carbon supersaturation in the ferrite before drawing, using a low temperature anneal, had no effect on the ageing rate after drawing. It is thought that carbon atoms will precipitate on dislocations during the anneal, and will therefore not effectively be removed from solid solution in the ferrite.

4. The drafting schedule was noted to affect the ageing rate of steel wire when measured by the shear test: a low final reduction in area was found to increase the time required for embrittlement. This was thought to be due either to a modified internal stress field, or to the influence of the strain rate. This result also illustrated the difference in the behaviour of aged wire in the tensile and shear tests.

5. Nitrogen was found to increase the ageing rate of steel wire, in particular if measured by the shear test. No measurable effect on the ageing mechanism was apparent.

6. The drawing strain was found to generally increase the rate of ageing. With regard to the shear properties, high strains can bring about embrittlement within the storage and service life of a rope. This appeared to occur at some strain between about 2,2 and 2,4. However, the drafting schedule also influences this behaviour (item 4).

7. A high drawing speed in the range up to 1 ms^{-1} was found to reduce the rate of deterioration of the wire ductility during ageing. This was due either to the less severe friction conditions within the die normally noted with high drawing speeds, or to the higher rate of strain.

8. A fine pearlite interlamellar spacing was found to increase the ageing rate. This could be attributed either to a shortened mean ferrite free path (for carbon diffusion), or to a modified dislocation substructure as a result of the effects of the pearlite spacing on the work hardening rate.

9. With small additions (of about 0,5%), silicon appears to increase the rate of ageing of a plain carbon steel, possibly by assisting the dissolution of carbon from the cementite. At larger silicon additions, from about 1% to 2%, the ageing rate was decreased, and this was attributed to the trapping of the carbon atoms at strained regions of the lattice where silicon atoms were present.

10. Aluminium additions were found not to remove the influence of nitrogen on the ageing rate. This was attributed to the heat treatment conditions used, which were not apparently suitable for the precipitation of aluminium nitride.

11. The drawing strain and the nitrogen content appeared to have a synergistic detrimental effect on the ageing rate. A high drawing speed was found to reduce the detrimental effect of a high drawing strain.

12. The statistical factorial design approach to experiments was found to be very useful for this application, where a large number of parameters may affect the results.

From these conclusions it is not possible to define the ageing mechanism(s) operating. Yamada attributed the rate controlling ageing step to carbon dissolution and migration from cementite, and the results presented here generally do not conflict with this theory. However, the overall process(es) is probably more complex, because of the particular effects of nitrogen and the drafting schedule on the wire ductility. It was proposed that these effects are related in some way to the dislocation substructure after drawing, and that ageing might therefore be influenced by the migration of dislocations.

An alternative theory, that the increase in the tensile properties during ageing is due a refinement of the dislocation substructure has been proposed in the literature, but not proved. However, the activation energy derived here for the ageing process may not conform to that for dislocation movement, although this is difficult to determine.

6.5 RECOMMENDATIONS FOR FUTURE WORK

This study has not been able to resolve the exact ageing mechanism operating in steel wire, and has left a number of questions unanswered. Further work is therefore required to develop a deeper insight into the ageing phenomenon, and some of these areas of investigation are outlined below.

1. Of particular importance commercially is the effect of ageing on the ductility of a wire in a roping application. Thus the shear test, which attempts to simulate these conditions, is a more valid test than the tensile test. This study demonstrated that the tensile properties and the shear properties are affected differently by ageing, and the reasons for this are not clear. The full characterisation of the shear test (i.e. the exact failure mechanism of the "shear" break), and the parameters which affect it (e.g. the residual stress level etc.) would therefore be very useful, although difficult to achieve.

2. A detailed investigation of the effects of the various drawing parameters (i.e. the drafting schedule, the strain rate, the amount of redundant work etc) would be beneficial.

3. A close investigation of the as-drawn structure, and in particular the pearlite spacing and the dislocation substructure should assist with identifying the ageing mechanism. In particular, the effect of strains greater than about 2,1 should be studied. Unfortunately, sufficient resolution for a detailed investigation may not be available with present instruments.

4. The effect of aluminium additions should be re-examined, with a view to selecting the correct heat treatment to precipitate the aluminium nitride, and hence to reduce the influence of nitrogen on the ageing process.

5. Alternative nitride-forming elements, such as vanadium, niobium, and boron could also be studied.

6. The small-scale heat treatment facilities which were required for the processing of alloy steels, and of plain carbon steels under special conditions, should be examined in an attempt to reduce the errors incurred by non-uniform heat treatments on long sample lengths.

APPENDIX A : COMPUTER PROGRAMME LISTINGS FOR THE ANALYSIS OF STRESS STRAIN DATA FROM THE TENSILE TEST (HP 85).

SSTEST
TO LOG THE DATA FROM A STRESS-STRAIN TEST ON THE INSTRON M/C

COMMENTS

```
  1 ! SSTEST - JULY 1986 NPWD
  5 CLEAR @ DISP @ DISP
 10 DISP "PROGRAMME TO READ LOAD
    -EXTENSION"
 20 DISP "DATA FROM THE STRESS/S
    TRAIN TEST" @ DISP
 30 WAIT 2000 @ DIM P(1000)
 32 DEFAULT ON
 33 IF FS(1)=0 THEN DISP "LOAD R
    ANGE NOT CALIBRATED"
 35 IF FS(2)=0 THEN DISP "STRAIN
     RANGE NOT CALIBRATED"
 40 DEFAULT OFF @ Y$=""
 50 IMAGE #,B
 60 SET TIMEOUT 7;1000
 70 ON TIMEOUT 7 GOTO 1370
 80 MASK -29920,1
 90 !
100 DISP "Enter Load-Cell Full-S
    cale (kN)" @ INPUT D1@ D1=D1
    *1000
120 DISP "USE THE 50mm GL EXTENS
    OMETER" @ DISP "WITH 10% MAX
     STRAIN"
130 DISP @ DISP "ENTER THE RANGE
     USED"
140 INPUT R@ D2=R/2
160 DISP "Enter Extensometer Gau
    se Length" @ INPUT G
180 DISP "Enter Wire Diameter (m
    m)" @ INPUT D
200 A1=D^2*PI/4 ! AREA
210 E1=D1/(FS(1)-ZR(1))
220 E2=D2/(FS(2)-ZR(2))
230 IF Y$="Y" THEN GOTO 350
235 CLEAR
240 DISP "TEST NUMBER ? (N.B. MAX
     6 Chars)"
270 DISP @ BEEP
280 DISP "**********************
    *********" @ DISP "  DATA TA
    PE MUST BE INSTALLED"
290 DISP "**********************
    *********"
295 ON ERROR GOTO 4000
300 INPUT K$
350 CLEAR @ DISP "Preload sample
    "
370 DISP @ DISP "Press 'CONT' to
     read load"
380 PAUSE
400 DISP @ DISP
410 DISP " ****** READING PRELOA
    D ****** "
420 GOSUB 1110
440 P1=E1*(L-ZR(1)) ! LOAD (N)
450 IMAGE /,4X,"Preload = ",20.2
    D,X,"kN",/
460 DISP USING 450 ; P1/1000
470 DISP "Value agrees with char
    t ? (Y/N)" @ INPUT A$
```

Check for calibration of load and strain ranges on Instron machine.

Set device timeout for handshake errors.

Enter relevant test data.

Enter test number.

Apply preload manually to sample (i.e. not under computer control).

Read value of preload for operator to check calibration of HP85 against Instron readout.

```
490 IF A$="N" THEN 1180
500 IF A$<>"Y" THEN 470
510 CLEAR
520 DISP @ DISP
530 DISP "OK - Attach Extensomet
    er and" @ DISP "zero strain"
550 DISP @ DISP "Recommend 2 mm/
    min X-Head Speed" @ DISP
580 DISP "Time interval between
    readings ?" @ INPUT T
610 ON KEY# 1,"START" GOTO 700
630 N=1 @ CLEAR @ DISP
640 KEY LABEL
650 DISP "START TEST WHEN READY"
670 DISP @ DISP "Press k1 to sta
    rt taking LOAD" @ DISP "and
    STRAIN readings"
690 GOTO 690
700 ON KEY# 1,"STOP" GOTO 1290
705 SETTIME 0,1
710 CLEAR
720 KEY LABEL
725 L2=P1/A1
730 GOSUB 1110
740 L1=E1*(L-ZR(1)) ! Load (N)
750 S1=E2*(S-ZR(2)) ! Ext (mm)
760 IF N/5<>INT(N/5) THEN GOTO 7
    95
770 IF N/50=INT(N/50) THEN CLEAR
    @ KEY LABEL
780 IMAGE "POINT No ",3D,3X,DD.D
    D,"kN",3X,D.DD,"mm"
790 DISP USING 780 ; N,L1/1000,S
    1
795 L2=MAX(L1,L2)
800 L1=IP(L1*10/A1)
802 S1=IP(S1*100000/G)/10000
805 P(N)=L1+S1
810 N=N+1
820 IF L1>L2*.5 THEN GOTO 830 EL
    SE 835
830 IF N<1000 THEN GOTO 730
835 L3=IP(L2/A1) ! MAX STRESS
836 L2=IP(1000*L2+.5)/1000
840 GOSUB 1290
845 PRINT @ PRINT "Test No : ";K
    $
850 PRINT "READINGS TAKEN =";N
852 PRINT "MAX LOAD       =";IP(
    L2)/1000;"kN"
855 PRINT "ESTIMATED UTS  =";L3;
    "MPa" @ PRINT @ PRINT
860 GOSUB 5000
890 BEEP @ DISP "DATA HAS BEEN S
    TORED " @ DISP
940 DISP "Another Test (Y/N) ?"
950 INPUT G$
960 IF G$="Y" THEN GOTO 5100
970 IF G$<>"N" THEN GOTO 940
980 CLEAR
```

Go-ahead to attach extensometer.

Set time interval between reading data readings (in ms)

Start test - i.e. start crosshead. When ready (i.e. after grips have bedded in) start recording data points.

Convert load and strain signals from mV to kN and strain units.

Monitor end of test by fall-off in load.

Test results summary.

Another test (yes or no)

```
990 CPOS 2,6,"SAMPLING COMPLETE"
1020 DISP @ DISP "DO YOU WANT TO
     ANALYSE DATA ? "
1030 DISP "Enter (Y)es OR (N)o"
1040 INPUT H$
1050 IF H$="Y" THEN GOTO 1540
1060 IF H$<>"N" THEN GOTO 1020
1070 CLEAR
1080 CPOS 3,6,"PROGRAMME ABANDON
     ED"
1090 WAIT 2000 @ CLEAR
1100 END
1110 ! SBR TO READ L @ S
1120 CONTROL 7,16 ; 3,5,0,0
1130 MASK 2^5,1
1140 SAMP E,W0,L,S
1150 LOCAL 7
1160 WAIT T
1170 RETURN
1180 !
1190 CLEAR
1200 DISP "CHECK LOAD-CELL RANGE
     "
1210 DISP "CHECK ZERO" @ DISP
1220 DISP "IF YOU WANT TO RESTAR
     T,ENTER'Y';  OTHERWISE ENTE
     R 'N'"
1230 INPUT Y$
1240 IF Y$="Y" THEN GOTO 90
1250 IF Y$<>"N" THEN GOTO 1220
1260 DISP @ DISP
1270 DISP "      ABORTING TEST"
1280 END
1290 ! SBR TO STOP INSTRON
1300 REMOTE 700
1310 TRIGGER 700
1320 OUTPUT 700 USING 50 ; 0,0,6
     4
1330 LOCAL 7
1340 BEEP @ DISP "STOP"
1350 IF F3=1 THEN GOTO 7000
1360 RETURN
1370 ! TIMEOUT SBR
1380 CLEAR @ BEEP
1390 DISP "      INTERFACE ERROR"
1400 RESET 7
1410 LOCAL 7
1420 DISP @ DISP
1430 DISP "       TEST ABORTED"
1440 END
1540 !
1550 BEEP @ DISP "ENSURE SOFTWAR
     E TAPE IS LOADED" @ DISP
1560 DISP "   WHEN READY PRESS '
     CONT' "
1570 PAUSE
1580 CLEAR
1590 CPOS 5,6,"PLEASE WAIT WHILE
     "
1600 CPOS 5,8,"PROGRAM IS LOADED
     "
```

Communication with A/D card to read load and strain signals.

Error trapping subroutines.

Carry on to analyse data.

```
1610 WAIT 2000
1620 CHAIN "SSPLOT"
1630 CLEAR
4000 BEEP
4002 IF ERRN#63 THEN GOTO 4500          More error trapping subroutines.
4005 CLEAR
4010 DISP " ERROR IN FILENAME"
4020 DISP @ DISP "FILE ";K$;" AL
     READY EXISTS"
4030 DISP @ DISP
4040 DISP " PURGE THIS FILE (P)
     or " @ DISP " ENTER NEW NAM
     E  (N) ?"
4050 INPUT N$
4060 IF N$="N" THEN GOTO 240
4070 IF N$="P" THEN GOTO 4100
4080 GOTO 4040
4100 DISP "PURGING FILE ";K$
4110 DISP @ DISP
4120 DISP "PLEASE WAIT"
4125 WAIT 1000
4130 PURGE K$
4140 BEEP @ CLEAR
4150 DISP "FILE ";K$;" PURGED"
4160 DISP @ DISP
4170 DISP "RECREATING NEW FILE "
     ;K$
4175 WAIT 1000
4180 GOTO 5012
4500 DISP "ERROR NUMBER ";ERRN
4505 DISP "ERROR ON LINE";ERRL
4510 DISP @ DISP
4521 IF ERRN=65 THEN DISP "TAPE
     IS FULL !" @ GOTO 4600
4522 IF ERRN=61 THEN DISP "TOO M
     ANY FILES ! (i.e. > 42)" @
     GOTO 4600
4525 DISP
4530 DISP "ENDING PROGRAMME"
4540 WAIT 2000
4545 COPY
4550 CLEAR
4560 END
4600 DISP "LOAD A NEW TAPE - WHE
     N READY " @ DISP "PRESS 'CO
     NT'" @ PAUSE
4610 GOTO 860
5000 ! DATA STORAGE SBR
5010 CLEAR @ DISP "STORING TEST     Store data from test onto tape.
     DATA - PLEASE WAIT"
5012 R1=IP(N%8/256+.5)+1
5015 CREATE K$,R1
5017 ASSIGN# 1 TO K$
5018 PRINT# 1 ; D,R1,G
5020 FOR I=1 TO N-1
5030 PRINT# 1 ; P(I) @ NEXT I
5040 PRINT# 1 ; N,L2
5050 ASSIGN# 1 TO *
5060 RETURN
5100 !
```

```
5110 DISP @ DISP "ARE TEST PARAM
     ETERS THE SAME ?" @ INPUT Y
     $
5120 IF Y$="Y" THEN GOTO 235
5130 IF Y$<>"N" THEN GOTO 5110
5140 GOTO 90
7000 DISP "STOP - DO YOU WISH TO
      SAVE       CURRENT DATA (Y/
     N) ?"
7010 INPUT A$
7020 IF A$="Y" THEN GOSUB 5000
7025 DISP "NEW TEST ..." @ GOTO
     5100
7030 GOTO 5110
```

```
SSSEQ
TO CALCULATE THE 0.2%PS FROM THE
DATA COLLECTED BY SSTEST


  5 CLEAR
 20 DISP "  AUTOPLOT STRESS/STRA
    IN DATA"
 40 OPTION BASE 0
 50 DIM C1(10),C2(10),C3(10)
 60 DISP @ DISP
 70 DISP "Please ensure that the
     DATA " @ DISP "STORAGE TAPE
     is installed"
 80 DISP @ DISP "Press 'CONT' wh
    en ready"
 90 BEEP @ PAUSE
 91 CLEAR @ DISP "Default values
     are A=5 and B=75"
 92 ON ERROR GOTO 8000
 93 DISP "Enter lower bound for
    E-mod calc" @ INPUT A
 94 ON ERROR GOTO 8010
 95 DISP "Enter upper bound for
    E-Mod calc" @ INPUT B
100 DIM X(900,2)
110 CLEAR @ GOSUB 9000
115 FOR Q=0 TO S-1
120 ON Q+1 GOSUB 5500,5510,5520,
    5530,5540,5550,5560,5570,538
    0,5590
121 FOR J=C1(Q) TO C2(Q)
122 G$[C3(Q)+13]=VAL$(J)
125 DISP "LOADING ";G$
128 ON ERROR GOTO 6000
130 ASSIGN# 1 TO G$
140 READ# 1 ; D,A,G
150 I=0 @ N=0 @ U1=0 @ U=0 @ U2=
    0
160 I=I+1 @ READ# 1 ; P
165 U=IP(P)/10 ! ENG STRESS
170 X(I,1)=U*(FP(P)/10+1) ! TRUE
    STRESS
180 X(I,2)=LOG(FP(P)/10+1) ! TRU
    E STRAIN
192 U1=MAX(X(I,1),U1)
195 U2=MAX(U2,U)
200 IF FP(P)#0 THEN GOTO 160
205 N=P
210 ASSIGN# 1 TO *
220 PRINT "DATA SUMMARY FOR ";G$
    @ PRINT
238 PRINT "Wire Diameter (mm)";D
240 PRINT "Gauge Length  (mm)";G
250 PRINT "U.T.S (Eng)  (MPa)";I
    P(U2)
255 PRINT "U.T.S (True) (MPa)";I
    P(U1)
260 PRINT "No of Readings    ";N
270 PRINT @ PRINT
275 IF N>1000 THEN PRINT "DATA
    STORAGE ERROR" @ PRINT @ PRI
    NT @ PRINT @ GOTO 925
280 P=1 @ K=1
370 GOSUB 2140
```

Set start and end points for Young's modulus calculation in terms of the data points recorded in the test.

Select data file to be analysed.

Load data file.

Print data summary for test.

```
470 DISP "CORRECTING STRAIN VALU
    ES"
490 FOR I=1 TO N
500 X(I,2)=X(I,2)-E0 @ NEXT I
510 OFF ERROR
540 DISP "CALCULATING ELASTIC ST
    RAIN"
550 I=10
560 IF X(I,2) <(I,1)/M).0002 THE
    N GOTO 586
570 I=I+1 @ GOTO 560
580 II=I
585 ON ERROR GOTO 7000
590 PRINT "PROPORTIONAL LIMIT "
    @ PRINT @ PRINT USING "Point No.";
    II
600 IMAGE "STRAIN",2X,DD.3D," %"
    ,/,"STRESS",3X,5D," MPa",/
610 PRINT USING 600 ; X(I1,2)*10
    0,X(II,1)
612 ON ERROR GOTO 7020
615 PRINT @ PRINT "ELASTIC SLOPE
    "
620 IMAGE /,"E-mod ",4X,4D," GPa
    ",/,"Std Error",2X,3D," MPa"
630 PRINT USING 620 ; M/1000,E
635 OFF ERROR
640 DISP "CALCULATING PROOF STRE
    SS"
650 I=50 @ F2=0 @ F3=1
655 IF F2=1 THEN GOTO 668
658 H2=X(I,1)-M*(X(I,2)-.001)
660 IF ABS(H2)<10 THEN F2=1 @ X1
    =X(I,1) @ Y1=X(I,2)*100
668 H=X(I,1)-M*(X(I,2)-.002)
670 IF ABS(H)<10 THEN GOTO 681
675 IF I=N-1 THEN F3=0 @ GOTO 68
    1
680 I=I+1 @ GOTO 655
681 IF F2=0 THEN PRINT @ PRINT "
    CAN'T DO 0.1% P.S. - SORRY"
    @ GOTO 685
682 IMAGE /,"0,1% P.S.= ",4D," M
    Pa"
683 PRINT USING 682 ; X1
685 IF F3=0 THEN PRINT @ PRINT "
    CAN'T DO 0.2% P.S. - SORRY"
    @ GOTO 704
690 IMAGE /,"0,2% P.S.= ",4D," M
    Pa"
700 X2=X(I,1) @ Y2=X(I,2)*100
702 PRINT USING 690 ; X2
704 PRINT @ PRINT @ PRINT
705 GOSUB 1060
920 BEEP
922 NEXT J
925 NEXT Q
930 WAIT 1000 @ DISP "MORE DATA-
    FILES (Y/N)?"
935 BEEP 40,1000 @ BEEP 50,1000
```

Corrects strain values to zero strain at zero load (by extrapolating backwards to zero load to find the offset). Calculate elastic strain (point at which curve deviates from the elastic slope by more than 0.0002 strain)

Print estimated limit of proportionality

Print estimated elstic slope and associated error.

Calculate 0,1 and 0,2% proof stress.

```
940 INPUT Y1$
950 IF Y1$#"Y" AND Y1$#"N" THEN
    GOTO 930
960 IF Y1$="Y" THEN GOTO 110
980 BEEP
1030 CLEAR
1040 END
1050 ! ********************
1060 ! STRESS/STRAIN PLOT
1070 ! ********************
1080 PEN 1 @ GCLEAR
1090 SCALE -.35,2.2,-150,2500
1100 AXES .1,.1,0,2.2
1110 AXES .1,100,0,2500
1120 REMOP
1130 FOR I=1 TO N
1140 PLOT X(I,2)*100,X(I,1)
1150 PENUP @ NEXT I
1160 LDIR 0
1180 MOVE 1.50 @ LABEL "TRUE STR
     AIN %"
1200 MOVE .9,500 @ LABEL "Test N
     o. "
1220 MOVE 1.6,500 @ LABEL G$
1240 LDIR 90
1250 MOVE .15,686 @ LABEL "TRUE
     STRESS MPa"
1270 LDIR 0
1280 MOVE -.35,-30 @ LABEL "   0
     "
1300 MOVE -.35,470 @ LABEL " 500
     "
1320 MOVE -.35,970 @ LABEL "1000
     "
1340 MOVE -.35,1470 @ LABEL "150
     0"
1360 MOVE -.35,1970 @ LABEL "200
     0"
1380 !
1390 MOVE 0,-150 @ LABEL "0"
1410 MOVE .45,-150 @ LABEL ".5"
1430 MOVE .9,-150 @ LABEL "1.0"
1450 MOVE 1.4,-150 @ LABEL "1.5"
1470 MOVE 1.9,-150 @ LABEL "2.0"
1500 MOVE 0,0 @ DRAW 250000/M,25
     00
1510 MOVE .1,0 @ DRAW 250000/M+.
     1,2500
1520 MOVE .2,0 @ DRAW 250000/M+.
     2,2500
1530 IF F3=1 THEN MOVE 0,X2 @ DR
     AW Y2,X2
1535 IF F2=1 THEN MOVE 0,X1 @ DR
     AW Y1,X1
1550 COPY @ PRINT @ PRINT @ PRIN
     T @ PRINT @ PRINT
1560 RETURN
2140 ! ********************
2150 ! STATS ON STRESS/STRAIN
2160 ! ********************
```

Plot stress/strain curve.

Superimpose the elastic slope, and the proof stresses.

```
2380 CLEAR @ DISP "  PLEASE WAIT
     "
2390 DISP "  COMPUTING DATA..."
2400 X1=0 @ X2=0 @ X3=0
2410 Y1=0 @ Y2=0
2420 S1=0
2430 ! CALCULATIONS                    Linear regression calculations for
2440 FOR I=A TO B                      elastic slope.
2450 Y1=Y1+X(I,1) ! SY
2460 X1=X1+X(I,2) ! SX
2470 X2=X2+X(I,2)^2 ! SSX
2480 S1=S1+X(I,2)*X(I,1) ! SXY
2490 Y2=Y2+X(I,1)^2 ! SSY
2500 NEXT I
2510 X3=X1^2 ! SXS
2520 N1=B-A+1
2530 M=(N1*S1-X1*Y1)/(N1*X2-X3)
     ! SLOPE
2540 C=(Y1-M*X1)/N1 ! INTERCEPT
2550 E=((Y2-C*Y1-M*S1)/(N1-2))^.
     5 ! STANDARD ERROR
2680 E0=-(C/M)
2810 RETURN
5500 G$=S0$ @ RETURN                   Datafile name storage (temporary).
5510 G$=S1$ @ RETURN
5520 G$=S2$ @ RETURN
5530 G$=S3$ @ RETURN
5540 G$=S4$ @ RETURN
5550 G$=S5$ @ RETURN
5560 G$=S6$ @ RETURN
5570 G$=S7$ @ RETURN
5580 G$=S8$ @ RETURN
5590 G$=S9$ @ RETURN
6000 ! ERROR SBR                       Error trapping subroutines.
6010 BEEP 50,1000
6020 PRINT "ERROR SOMEWHERE IN F
     ILE ";G$ @ PRINT @ PRINT @
     OFF ERROR @ GOTO 925
7000 !
7005 OFF ERROR
7010 PRINT "ERROR IN PROP LIMIT"
      @ GOTO 612
7020 !
7025 OFF ERROR
7030 PRINT "ERROR IN E-MOD CALCN
     " @ GOTO 640
8000 OFF ERROR @ IF ERRN=43 THEN
      A=5 @ GOTO 94
8010 OFF ERROR @ IF ERRN=43 THEN
      B=75 @ GOTO 100
9000 ! SEQUENTIAL FILE INPUT           Datafile name input (in this case based
9010 DISP "HOW MANY TEST SETS (M       on a sequence of datafile names with
     AX 10)?" @ INPUT S                common roots).
9015 IF S>10 THEN BEEP @ GOTO 90
     10
9017 FOR I=0 TO S-1
9018 DISP "No ";I+1;"BATCH NAME
     (MAX 5 CHARS) ?" @ INPUT L$
9019 C3(I)=LEN(L$) @ IF C3(I)>5
     THEN BEEP @ GOTO 9018
```

```
9020 DISP "START No, END No" @ I
     NPUT C1(I),C2(I)
9025 ON I+1 GOSUB 9500,9510,9520
     ,9530,9540,9550,9560,9570,9
     580,9590
9027 DISP @ DISP
9030 NEXT I
9040 CLEAR @ RETURN
9500 S0$=L$ @ RETURN
9510 S1$=L$ @ RETURN
9520 S2$=L$ @ RETURN
9530 S3$=L$ @ RETURN
9540 S4$=L$ @ RETURN
9550 S5$=L$ @ RETURN
9560 S6$=L$ @ RETURN
9570 S7$=L$ @ RETURN
9580 S8$=L$ @ RETURN
9590 S9$=L$ @ RETURN
9598 RETURN
```

APPENDIX B : YATES' METHOD OF ANALYSIS FOR A 2^4 FACTORIAL DESIGN.

B1 INTRODUCTION

The following is a description of the Yates method of analysis for the estimation of the main effects and interactions from a factorial experiment, and is due to Natrella (1979). The method as given here applies to factorials, blocked factorials and fractional factorials for which 2^n observations have been made.

A procedure for testing the significance of the main effects and the interactions, using the upper order interactions, is also presented.

B2 ESTIMATION OF THE MAIN EFFECTS AND INTERACTIONS

In the Yates method, each experiment is identified with a symbol which represents the treatment combinations for each experiment. Thus, the symbol "a" represents factor A at its level 2, and all the other factors at their level 1. Similarly, "bcd" represents the experiment where factors B, C, and D were at their level 2, and factor A was at its level 1.

The first step in the analysis method is to arrange the data for the experiment (i.e. the responses) into a standard order. For a 2^4 design, this order is as follows: (1), a, b, ab, c, ac, bc, abc, d, ad, bd, abd, cd, acd, bcd, abcd.

A table is then constructed with n+3 columns where n is the number of factors in the experiment (4 in this case). The treatment combinations are listed in column 1, and the response (e.g. the increase in the 0,2% proof stress) is listed in column two. As an example, see Table B1, which shows the as-drawn proof stress results from the factorial experiment in this study. The values in the rest of the table are calculated as follows.

In the top half of column K2, enter, in order, the sums of consecutive pairs of entries in column 2. In the bottom half of the column, enter, in order, the differences between the same consecutive pairs of entries, i.e. second entry minus first entry, fourth entry minus third entry, etc.

TABLE B1 : YATES METHOD OF ANALYSIS USING ACTUAL DATA FROM THE DESIGN (AS-DRAWN 0,2% PROOF STRESS RESULTS).

Treatment Combination	Response (0,2% PS)	K2	K3	K4	G	Error G/8
(1)	1996	3992	7943	16165	32033	4004.1
a	1996	3951	8222	15868	-285	-35.6
b	1979	4060	7733	-29	3	0.4
ab	1972	4162	8135	-256	-131	-16.4
c	2022	3897	-7	61	681	85.1
ac	2038	3836	-22	-58	-185	-23.1
bc	2100	4066	-43	-61	207	25.9
abc	2062	4069	-213	-70	-99	-12.4
d	1957	0	-41	279	-297	-37.1
ad	1940	-7	102	402	-227	-28.4
bd	1931	16	-61	-15	-119	-14.9
abd	1902	-38	3	-170	-9	-1.1
cd	2071	-17	-7	143	123	15.4
acd	1995	-26	-54	64	-155	-19.4
bcd	2103	-76	-9	-47	-73	-9.2
abcd	1966	-137	-61	-52	-5	-0.6

$\Sigma G^2 = 1,027 \times 10^9$

Σ (Response) = 32 033

Σ (Response)^2 = 64 185 095

Obtain the entries in the next three columns (up to the column labelled G) in the same manner as for column K2, i.e. by obtaining in each case the sums and differences of the pairs in the preceeding column in the manner described above. The final column (labelled G/8), is obtained by dividing the entries in column G by 8 (2^{n-1}). This final column gives the estimates of the main effects and interactions for each treatment combination, while the first entry gives twice the overall mean.

To check the computation, the following steps may be performed.

1. The sum of all the 2^n individual responses (i.e. the proof stress data) should equal the first entry in the "G" column.

2. The sum of squares of the individual responses should equal the sum of squares of the entries in the "G" column, divided by 2^n.

3. For any main effect, the entry in the "G" column equals the sum of the responses in which that factor is at its first level, minus the sum of the responses at which that factor is at its second level.

83 PROCEDURE FOR TESTING THE SIGNIFICANCE OF THE MAIN EFFECTS AND INTERACTIONS

The following steps should be performed.

1. Select a level of significance, α.
2. Find the sum of squares of G's corresponding to interactions of three or more factors.
3. To obtain s^2 (the variance), divide the sum of squares obtained in step 2 by $2^n.v$, where v is the number of interactions included.
4. Look up $t_{(1-\alpha/2)}$ for degrees of freedom in standard t-tables.
5. Compute $w = (2^n)(t)(s)$
6. For any main effect or interaction X, if the absolute value of G_X is greater than w, conclude that X is different from zero. Otherwise, there is no reason to believe that X is different from zero.

Example

1. Let α=0,1.

2. From Table B1;
$G^2_{(abc)} + G^2_{(abd)} + G^2_{(acd)} + G^2_{(bcd)} + G^2_{(abcd)} = 40\ 173.$

3. $n = 4$; $[v] = 5$; $2^n.v = 16.(5) = 80.$
$s^2 = 40\ 173/80 = 502,2$; $s = 22,4$.

4. $t_{(0,95)}$ for 5 df = 2,015.

5. $w = 4.(2,015).(22,4) = 180,6$
Estimated error = $w/8 = 22,577$ MPa.

Comparing this value of w with the values of "G" in Table B1, the following main effects and interactions are significant.

$[G_a]$; $[G_c]$; $[G_d]$; $[G_{ac}]$; $[G_{bc}]$; $[G_{cd}]$.

B4 COMPUTER PROGRAMME FOR THE PROCESSING OF RESULTS OF FACTORIAL DESIGN BY YATES' METHOD

The following routine was written for the *IBM PC, in Microsoft* Basic.

```
10 ' Yates Analysis Programme
20 ' N P W Davies 1987
30 DIM B$(16),K1(16),K2(16),K3(16),K4(16),B(16)
40 INPUT"Heading ";H$
50 PRINT"Enter data list "
60 FOR I=1 TO 16:PRINT I;:INPUT K1(I):NEXT
70 FOR I=1 TO 8:K2(I)=K1(2*I)+K1(2*I-1):K2(8+I)=K1(2*I)-K1(2*I-1):NEXT I
80 FOR I=1 TO 8:K3(I)=K2(2*I)+K2(2*I-1):K3(8+I)=K2(2*I)-K2(2*I-1):NEXT I
90 FOR I=1 TO 8:K4(I)=K3(2*I)+K3(2*I-1):K4(8+I)=K3(2*I)-K3(2*I-1):NEXT I
100 FOR I=1 TO 8:G(I)=K4(2*I)+K4(2*I-1):G(8+I)=K4(2*I)-K4(2*I-1):NEXT I
110 SUMG=0
120 V=5 'No of interactions included
130 N=4 'No of factors
140 NV=V*2^N
150 SUMG=G(8)^2+G(12)^2+G(14)^2+G(15)^2+G(16)^2
160 SSQ=SUMG/NV
170 T90=2.015:T80=1.476 't(1-alpha/2)
180 G$(1)="i":G$(2)="a":G$(3)="b":G$(4)="ab"
190 G$(5)="c":G$(6)="ac":G$(7)="bc":G$(8)="abc"
200 G$(9)="d":G$(10)="ad":G$(11)="bd":G$(12)="abd"
210 G$(13)="cd":G$(14)="acd":G$(15)="bcd":G$(16)="abcd"
220 LPRINT CHR$(27);"l";CHR$(10);
230 LPRINT"_____
__________":LPRINT H$:LPRINT
240 LPRINT" a = Nitrogen  b = Aluminium  c = Strain  d = Speed":LPRINT
250 LPRINT       " Expt No  Treatment Response      K2          K3          K4
 G          G/8"
260 LPRINT"_____
_____":
270 S=SSQ*.5
280 FOR I=1 TO 16
290 LPRINT USING"   ##       \      \    ####.### ######.##" ######.### #####.### ####
#.###  ####.###";I,G$(I),K1(I),K2(I),K3(I),K4(I),G(I),G(I)/8
300 NEXT
310 LPRINT:LPRINT USING"Sum G Squared (for three and four way interactions = ###
##.### ";SUMG
320 LPRINT USING"t(95%) = #.###     t(90%) = #.###     s = ###.###";T90,T80,S
330 LPRINT USING "Estimated Error at 90% Confidence Limit = ###.###";T90*S/2
340 LPRINT USING "Estimated Error at 80% Confidence Limit = ###.###";T80*S/2
350 LPRINT"_____
_____":
360 LPRINT:LPRINT
370 END
380 DATA 4.2,3.1,4.5,2.9,3.9,2.8,4.6,3.2,4.0,3.0,5.0,2.5,4.0,2.5,5.0,2.3
```

APPENDIX C : MAIN EFFECT AND INTRACTION MEANS FROM THE HP 9845 ANALYSIS OF DATA FROM THE FACTORIAL EXPERIMENTS.

```
*****************************************************************************
*                        FACTORIAL ANALYSIS OF VARIANCE                     *
*****************************************************************************
                EXPT 8043 - As Drawn Wire - 0.2% Proof Stress

DESIGN
------
    Number of factors =  4
      No. of levels of Factor A =  2
      No. of levels of Factor B =  2
      No. of levels of Factor C =  2
      No. of levels of Factor D =  2
      No. of major replications (blocks) =  1
      No. of minor replications (samples) =  5

Subfiles will be ignored
Response variable(s) are :
Variable no.  1      1
Variable no.  2      2
Variable no.  3      3
Variable no.  4      4
Variable no.  5      5

MEANS
-----
* Overall mean =          2001.9


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
         2020.3          1983.5
Factor B - STRAIN   Levels ( 1 - 2 ) :
         1959.6          2044.2
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
         2001.9          2001.9
Factor D - NITROGEN   Levels ( 1 - 2 ) :
         2019.9          1984.0


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                    1               2
    1               1985.8          2054.8
    2               1933.3          2033.6

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                    1               2
    1               2013.1          2027.5
    2               1990.7          1976.2

Factor A - SPEED  down and  Factor D - NITROGEN  across
                    1               2
    1               2024.1          2016.5
    2               2015.5          1951.4

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                    1               2
    1               1972.5          1946.6
    2               2031.3          2057.1

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                    1               2
    1               1965.8          1953.3
    2               2073.8          2014.6

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                    1               2
```

1	2011.4	1992.4
2	2028.2	1975.6

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	1996.3	1975.9
2	2029.9	2079.7

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	1948.7	1917.9
2	2032.7	2034.5

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	1987.4	1984.2
2	2060.7	2048.8

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	1944.2	1922.4
2	2086.8	1980.4

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	2008.8	2017.3
2	2039.3	2015.7

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	2014.0	1967.4
2	2017.0	1935.4

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	1976.9	1968.2
2	1954.8	1938.4

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	2046.0	2016.5
2	2101.5	2012.7

* Four Way Interaction Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	1996.2	1996.4
2	1978.6	1972.0

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	2021.4	2038.2
2	2100.6	2059.4

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1

Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	1957.4	1940.8
2	193'.0	1984.8

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	2070.6	1994.8
2	2183.8	1965.8

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)	df	Sums of Squares	Mean Square	F Ratio	F-Prob
Total	79	286899.5	3631.6		
A SPEED	1	27121.6	27121.6	73.780	~.0000
B STRAIN	1	143227.8	143227.8	389.630	~.0000
C ALUMINIUM	1	.0	.0	.000	.9955
D NITROGEN	1	25668.6	25668.6	69.828	.0000
AB	1	4914.1	4914.1	13.368	.0005
AC	1	4190.5	4190.5	11.400	.0013
AD	1	15989.5	15989.5	43.497	.0000
BC	1	13390.3	13390.3	36.425	.0000
BD	1	10881.1	10881.1	29.603	.0000
CD	1	5628.0	5628.0	15.310	.0002
ABC	1	1833.6	1833.6	4.988	.0290
ABD	1	7201.0	7201.0	19.589	.0000
ACD	1	10.5	10.5	.029	.8662
BCD	1	3315.3	3315.3	9.019	.0038
ABCD	1	1.0	1.0	.003	.9589
Sampling Error	64	23526.4	367.6		

NOTE: F tests assume that all factors are fixed

```
**********************************************************************
*                  FACTORIAL ANALYSIS OF VARIANCE                    *
**********************************************************************
                As Drawn Wire - Shear Elongation Results

DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1      Shear El

MEANS
-----
* Overall mean =            .473


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
        .472              .473
Factor B - STRAIN   Levels ( 1 - 2 ) :
        .489              .457
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
        .474              .472
Factor D - NITROGEN   Levels ( 1 - 2 ) :
        .486              .488


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                  1                 2
    1              .481              .463
    2              .457              .490

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                  1                 2
    1              .491              .454
    2              .457              .490

Factor A - SPEED  down and  Factor D - NITROGEN  across
                  1                 2
    1              .489              .495
    2              .473              .474

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                  1                 2
    1              .471              .467
    2              .477              .477

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                  1                 2
    1              .483              .475
    2              .489              .485

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                  1                 2
    1              .481              .486
    2              .470              .474
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.493	.469
2	.489	.438

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.449	.465
2	.465	.515

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.465	.497
2	.453	.474

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.460	.454
2	.485	.495

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.463	.519
2	.455	.452

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.460	.454
2	.485	.495

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.483	.489
2	.473	.462

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.470	.484
2	.468	.486

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.455	.530
2	.475	.463

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.470	.507
2	.435	.441

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.450	.447
2	.470	.468

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.470	.460
2	.500	.530

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	.013	.001
A	SPEED	1	.000	.000
B	STRAIN	1	.000	.000
C	ALUMINIUM	1	.000	.000
D	NITROGEN	1	.001	.001
AB		1	.003	.003
AC		1	.005	.005
AD		1	.001	.001
BC		1	.000	.000
BD		1	.000	.000
CD		1	.000	.000
ABC		1	.001	.001
ABD		1	.000	.000
ACD		1	.001	.001
BCD		1	.001	.001
ABCD		1	.000	.000

```
*************************************************************************
*                    FACTORIAL ANALYSIS OF VARIANCE                     *
*************************************************************************
                    AGED WIRE - 50 C - 2 hours


DESIGN
------
    Number of factors =  4
     No. of levels of factor A =  2
     No. of levels of factor B =  2
     No. of levels of factor C =  2
     No. of levels of factor D =  2
     No. of major replications (blocks) =  1
     No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1       delta .2PS

MEANS
-----
* Overall mean =              35.44


* Main Effect Means :

Factor A - SPEED    Levels ( 1 - 2 ) :
          37.50               33.38
Factor B - STRAIN   Levels ( 1 - 2 ) :
          24.88               46.00
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
          31.75               39.13
Factor D - NITROGEN    Levels ( 1 - 2 ) :
          35.13               35.75


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                      1                   2
     1                19.25               55.75
     2                30.50               36.25

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                      1                   2
     1                34.00               41.00
     2                29.50               37.25

Factor A - SPEED  down and  Factor D - NITROGEN  across
                      1                   2
     1                41.00               34.00
     2                29.25               37.50

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                      1                   2
     1                16.00               33.75
     2                47.50               44.50

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                      1                   2
     1                36.00               13.75
     2                34.25               57.75

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                      1                   2
     1                38.75               24.75
     2                31.50               46.75
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	12.00	26.50
2	56.00	55.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	20.00	41.00
2	39.00	33.50

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	29.50	9.00
2	52.50	59.00

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	42.50	18.50
2	16.00	56.50

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	50.00	16.00
2	32.00	50.00

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	27.50	31.50
2	31.00	43.50

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	29.50	2.50
2	42.50	25.00

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	48.00	47.00
2	20.50	68.50

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	29.00	-5.00
2	30.00	23.00

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	71.00	41.00
2	54.00	77.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	30.00	16.00
2	55.00	27.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM' down and Factor D - NITROGEN across

	1	2
1	25.00	53.00
2	7.00	60.00

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	7845.94	523.06
A	SPEED	1	68.06	68.06
B	STRAIN	1	1785.06	1785.06
C	*ALUMINIUM*	1	217.56	217.56
D	NITROGEN	1	1.56	1.56
AB		1	945.56	945.56
AC		1	.56	.56
AD		1	232.56	232.56
BC		1	430.56	430.56
BD		1	2093.06	2093.06
CD		1	855.56	855.56
ABC		1	33.06	33.06
ABD		1	351.56	351.56
ACD		1	430.56	430.56
BCD		1	390.06	390.06
ABCD		1	10.56	10.56

```
*****************************************************************************
*                        FACTORIAL ANALYSIS OF VARIANCE                     *
*****************************************************************************
                        AGED WIRE - 50 C - 500 hours


DESIGN
------
   Number of factors =  4
    No. of levels of Factor A =  2
    No. of levels of Factor B =  2
    No. of levels of Factor C =  2
    No. of levels of Factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1       delta .2PS

MEANS
-----
* Overall mean =            183.69


* Main Effect Means :

Factor A - SPEED    Levels ( 1 - 2 ) :
          194.88             172.50
Factor B - STRAIN   Levels ( 1 - 2 ) :
          179.00             188.38
Factor C - ALUMINIUM    Levels ( 1 - 2 ) :
          176.75             188.63
Factor D - NITROGEN    Levels ( 1 - 2 ) :
          170.25             197.13


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                      1                 2
    1               165.50            224.25
    2               192.50            152.50

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                      1                 2
    1               204.50            185.25
    2               153.00            192.00

Factor A - SPEED  down and  Factor D - NITROGEN  across
                      1                 2
    1               188.50            201.25
    2               152.00            193.00

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                      1                 2
    1               179.25            179.75
    2               179.25            197.50

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                      1                 2
    1               182.75            175.25
    2               157.75            219.00

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                      1                 2
    1               155.75            201.75
    2               184.75            192.50
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	165.00	166.00
2	244.00	264.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	191.50	193.50
2	114.50	198.50

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	157.00	174.00
2	228.00	229.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	208.50	176.50
2	95.50	209.50

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	204.00	205.00
2	173.00	197.50

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	127.50	196.50
2	196.50	187.50

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	183.50	173.00
2	182.00	177.50

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	128.00	230.50
2	187.50	207.50

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	170.00	168.00
2	144.00	188.00

Factor A - SPEED, Level 1 Fac[illegible], Level 2
Factor C - ALUMINIUM down and Factor [illegible] across

	1	2
1	238.00	250.00
2	282.00	207.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	197.00	186.00
2	228.00	167.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	18.00	211.00
2	173.00	288.00

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	41251.44	2750.10
A	SPEED	1	2002.56	2002.56
B	STRAIN	1	351.56	351.56
C	ALUMINIUM	1	390.06	390.06
D	NITROGEN	1	2889.06	2889.06
AB		1	9751.56	9751.56
AC		1	3393.06	3393.06
AD		1	798.06	798.06
BC		1	280.56	280.56
BD		1	4726.56	4726.56
CD		1	1463.06	1463.06
ABC		1	3277.56	3277.56
ABD		1	5967.56	5967.56
ACD		1	3813.06	3813.06
BCD		1	1958.06	1958.06
ABCD		1	189.06	189.06

```
**********************************************************************
*          -  FACTORIAL ANALYSIS OF VARIANCE                         *
**********************************************************************
                    AGED WIRE - 50 C - 500 hours


DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  4       SHEAR EL.

MEANS
-----
* Overall mean =              .403


* Main Effect Means :

Factor A - SPEED    Levels ( 1 - 2 ) :
          .374                .431
Factor B - STRAIN   Levels ( 1 - 2 ) :
          .415                .390
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
          .406                .399
Factor D - NITROGEN   Levels ( 1 - 2 ) :
          .416                .389


* Two Way Interaction Means :

Factor A - SPEED    up and  Factor B - STRAIN  across
                    1                   2
     1                .418                .330
     2                .420                .443

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                    1                   2
     1                .375                .373
     2                .438                .425

Factor A - SPEED  down and  Factor D - NITROGEN  across
                    1                   2
     1                .393                .355
     2                .440                .423

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                    1                   2
     1                .390                .440
     2                .423                .358

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                    1                   2
     1                .403                .428
     2                .430                .350

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                    1                   2
     1                .423                .390
     2                .410                .388
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.380	.440
2	.370	.305

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.400	.440
2	.475	.410

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.400	.420
2	.385	.290

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.405	.435
2	.475	.418

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.370	.388
2	.415	.330

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.475	.400
2	.405	.445

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.395	.395
2	.420	.460

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.460	.385
2	.400	.315

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.360	.400
2	.440	.440

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.380	.360
2	.380	.320

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.410	.390
2	.400	.480

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.648	.418
2	.418	.418

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)	df	Sums of Squares	Mean Square
Total	15	.066	.004
A SPEED	1	.013	.013
B STRAIN	1	.003	.003
C ALUMINIUM	1	.008	.008
D NITROGEN	1	.003	.003
AB	1	.009	.009
AC	1	.000	.000
AD	1	.000	.000
BC	1	.013	.013
BD	1	.011	.011
CD	1	.000	.000
ABC	1	.000	.000
ABD	1	.000	.000
ACD	1	.011	.011
BCD	1	.000	.000
ABCD	1	.001	.001

```
*************************************************************************
*                    FACTORIAL ANALYSIS OF VARIANCE                     *
*************************************************************************
                        AGED WIRE   50 C - 1000 Hours

DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1       delta .2PS

MEANS
-----
* Overall mean =          226.88


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
         236.38           217.38
Factor B - STRAIN   Levels ( 1 - 2 ) :
         207.13           246.63
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
         223.88           230.75
Factor D - NITROGEN   Levels ( 1 - 2 ) :
         224.50           229.25


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                      1                2
    1               206.75           264.00
    2               205.50           229.25

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                      1                2
    1               232.00           240.75
    2               214.00           220.75

Factor A - SPEED  down and  Factor D - NITROGEN  across
                      1                2
    1               222.75           250.00
    2               226.25           208.50

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                      1                2
    1               189.00           225.25
    2               257.00           236.25

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                      1                2
    1               208.25           206.00
    2               240.75           252.50

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                      1                2
    1               219.75           226.25
    2               229.25           232.25
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	187.50	230.00
2	276.50	251.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	198.50	220.50
2	237.50	221.00

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	186.50	231.00
2	259.00	269.00

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	238.00	181.00
2	222.50	236.00

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	238.50	233.50
2	215.00	266.50

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	289.00	219.00
2	243.50	198.00

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	187.50	190.50
2	229.00	221.50

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	252.00	262.00
2	229.50	243.00

* Four Way Interaction Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	180.00	195.00
2	193.00	267.00

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	281.00	272.00
2	237.00	266.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	195.00	186.00
2	265.00	176.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across
1 2

1	223.00	252.00
2	222.00	228.00

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	20815.75	1334.38
A	SPEED	1	1444.00	1444.00
B	STRAIN	1	6241.00	6241.00
C	ALUMINIUM	1	240.25	240.25
D	NITROGEN	1	90.25	90.25
AB		1	992.25	992.25
AC		1	4.00	4.00
AD		1	2025.00	2025.00
BC		1	3249.00	3249.00
BD		1	196.00	196.00
CD		1	12.25	12.25
ABC		1	110.25	110.25
ABD		1	2352.25	2352.25
ACD		1	2704.00	2704.00
BCD		1	49.00	49.00
ABCD		1	306.25	306.25

```
*************************************************************************
*                      FACTORIAL ANALYSIS OF VARIANCE                   *
*************************************************************************
                      AGED WIRE - 50 C - 1000 hours

DESIGN
------
   Number of factors =  4
     No. of levels of factor A =  2
     No. of levels of factor B =  2
     No. of levels of factor C =  2
     No. of levels of factor D =  2
     No. of major replications (blocks) =  1
     No. of minor replications (samples) =  1

Sub-files will be ignored
Response variable(s) are :
Variable no.  4       SHEAR EL.

MEANS
-----
* Overall mean =              .378


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
             .359              .389
Factor B - STRAIN   Levels ( 1 - 2 ) :
             .393              .346
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
             .372              .369
Factor D - NITROGEN   Levels ( 1 - 2 ) :
             .399              .346


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                    1                 2
    1                .391              .310
    2                .395              .383

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                    1                 2
    1                .346              .355
    2                .398              .380

Factor A - SPEED  down and  Factor D - NITROGEN  across
                    1                 2
    1                .403              .298
    2                .395              .383

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                    1                 2
    1                .391              .395
    2                .353              .340

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                    1                 2
    1                .393              .393
    2                .405              .286

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                    1                 2
    1                .395              .348
    2                .403              .333
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.406	.375
2	.285	.335

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.375	.415
2	.420	.345

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.400	.381
2	.405	.215

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.385	.405
2	.405	.360

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.405	.286
2	.400	.310

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.385	.410
2	.405	.355

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.380	.401
2	.405	.385

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.410	.295
2	.400	.280

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.400	.412
2	.400	.350

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.410	.160
2	.400	.270

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.360	.390
2	.410	.420

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.410	.438
2	.400	.290

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	.078	.005
A	SPEED	1	.006	.006
B	STRAIN	1	.009	.009
C	ALUMINIUM	1	.000	.000
D	NITROGEN	1	.014	.014
AB		1	.005	.005
AC		1	.001	.001
AD		1	.008	.008
BC		1	.000	.000
BD		1	.014	.014
CD		1	.001	.001
ABC		1	.010	.010
ABD		1	.003	.003
ACD		1	.003	.003
BCD		1	.000	.000
ABCD		1	.005	.005

```
*****************************************************************************
*                    FACTORIAL ANALYSIS OF VARIANCE                         *
*****************************************************************************
                        AGED WIRE - 80 C - 48 hours


DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1       delta .2PS

MEANS
-----
* Overall mean =           260.19


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
         264.75           255.63
Factor B - STRAIN   Levels ( 1 - 2 ) :
         240.75           279.63
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
         265.25           255.13
Factor D - NITROGEN   Levels ( 1 - 2 ) :
         263.25           257.13


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                      1                2
    1                240.75           288.75
    2                240.75           270.50

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                      1                2
    1                272.25           257.25
    2                258.25           253.00

Factor A - SPEED  down and  Factor D - NITROGEN  across
                      1                2
    1                273.00           256.50
    2                253.50           257.75

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                      1                2
    1                235.25           246.25
    2                295.25           264.00

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                      1                2
    1                248.25           233.25
    2                278.25           281.00

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                      1                2
    1                268.50           262.00
    2                258.00           252.25
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	239.00	242.50
2	305.50	272.00

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	231.50	250.00
2	285.00	256.00

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	242.00	239.50
2	304.00	273.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	254.50	227.00
2	252.50	286.50

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	284.00	268.50
2	262.00	313.50

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	253.00	263.50
2	254.00	252.00

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	242.50	228.00
2	254.00	238.50

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	294.50	296.00
2	262.00	266.00

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	248.00	230.00
2	336.00	249.00

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	320.00	291.00
2	288.00	256.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	237.00	226.00
2	272.00	228.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM, Down and Factor D - NITROGEN across

	1	2
1	275.00	381.88
2	296.38	276.00

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	12548.44	836.56
A	SPEED	1	333.06	333.06
B	STRAIN	1	6045.06	6045.06
C	ALUMINIUM	1	410.06	410.06
D	NITROGEN	1	150.06	150.06
AB		1	333.06	333.06
AC		1	95.06	95.06
AD		1	430.56	430.56
BC		1	1785.06	1785.06
BD		1	315.06	315.06
CD		1	.56	.56
ABC		1	27.56	27.56
ABD		1	2093.06	2093.06
ACD		1	175.56	175.56
BCD		1	3.06	3.06
ABCD		1	351.56	351.56

```
*************************************************************************
*                        FACTORIAL ANALYSIS OF VARIANCE                 *
*************************************************************************
                        AGED WIRE - 80 C - 48 hours

DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  4       SHEAR EL

MEANS
-----
* Overall mean =              .329

* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
            .308              .350
Factor B - STRAIN   Levels ( 1 - 2 ) :
            .398              .260
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
            .318              .340
Factor D - NITROGEN   Levels ( 1 - 2 ) :
            .365              .293

* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                  1                 2
    1              .410              .205
    2              .385              .315

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                  1                 2
    1              .278              .338
    2              .358              .343

Factor A - SPEED  down and  Factor D - NITROGEN  across
                  1                 2
    1              .370              .245
    2              .360              .340

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                  1                 2
    1              .410              .385
    2              .225              .295

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                  1                 2
    1              .358              .438
    2              .363               58

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                  1                 2
    1              .348              .288
    2              .383              .298
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.400	.420
2	.155	.255

Factor A - SPEED, Level 2
Factor B - STRAIN down and actor C - ALUMINIUM across

	1	2
1	.420	.350
2	.295	.395

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.395	.430
2	.350	.060

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.345	.425
2	.375	.255

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.350	.205
2	.390	.285

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.345	.370
2	.375	.310

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.390	.430
2	.345	.425

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.305	.145
2	.420	.170

* Four Way Interaction Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.390	.410
2	.390	.450

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.310	0.000
2	.390	.120

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.390	.450
2	.380	.400

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.300	.290
2	.450	.220

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	.238	.016
A	SPEED	1	.007	.007
B	STRAIN	1	.076	.076
C	ALUMINIUM	1	.002	.002
D	NITROGEN	1	.021	.021
AB		1	.018	.018
AC		1	.006	.006
AD		1	.011	.011
BC		1	.009	.009
BD		1	.070	.070
CD		1	.001	.001
ABC		1	.000	.000
ABD		1	.004	.004
ACD		1	.004	.004
BCD		1	.004	.004
ABCD		1	.004	.004

```
*************************************************************************
*                    FACTORIAL ANALYSIS OF VARIANCE                     *
*************************************************************************
                        AGED WIRE - 100 C - 2 hours


DESIGN
------
   Number of factors =  4
     No. of levels of factor A =  2
     No. of levels of factor B =  2
     No. of levels of factor C =  2
     No. of levels of factor D =  2
     No. of major replications (blocks) =  1
     No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1      delta .2PS

MEANS
-----
* Overall mean =            203.50


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
          211.13             195.88
Factor B - STRAIN   Levels ( 1 - 2 ) :
          184.00             223.00
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
          204.89             202.13
Factor D - NITROGEN   Levels ( 1 - 2 ) :
          208.13             206.88


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                        1                  2
    1                 182.75             239.50
    2                 185.25             206.50

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                        1                  2
    1                 207.50             214.75
    2                 202.25             189.50

Factor A - SPEED  down and  Factor D - NITROGEN  across
                        1                  2
    1                 211.50             210.75
    2                 188.75             203.00

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                        1                  2
    1                 172.75             195.25
    2                 237.00             209.00

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                        1                  2
    1                 186.50             181.50
    2                 213.75             232.25

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                        1                  2
    1                 206.75             203.00
    2                 193.50             210.75
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	165.50	200.00
2	249.50	229.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	180.00	190.50
2	224.50	188.50

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	186.50	185.00
2	242.50	236.50

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	192.50	178.00
2	185.00	228.00

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	225.50	189.50
2	197.50	232.00

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	188.00	216.50
2	189.50	189.50

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	175.00	170.50
2	198.00	192.50

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	238.50	235.50
2	189.00	229.00

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	175.00	156.00
2	186.00	214.00

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	276.00	223.00
2	289.00	250.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	175.00	185.00
2	210.00	171.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	281.00	248.00
2	169.00	288.00

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)	df	Sums of Squares	Mean Square
Total	15	16764.00	1117.60
A SPEED	1	930.25	930.25
B STRAIN	1	5084.00	5084.00
C ALUMINIUM	1	30.25	30.25
D NITROGEN	1	182.25	182.25
AB	1	1260.25	1260.25
AC	1	400.00	400.00
AD	1	225.00	225.00
BC	1	2550.25	2550.25
BD	1	552.25	552.25
CD	1	441.00	441.00
ABC	1	16.00	16.00
ABD	1	1156.00	1156.00
ACD	1	2450.25	2450.25
BCD	1	484.00	484.00
ABCD	1	2.25	2.25

```
*****************************************************************************
*                        FACTORIAL ANALYSIS OF VARIANCE                     *
*****************************************************************************
                          AGED WIRE - 100 C - 2 hours

DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  4       SHEAR EL

MEANS
-----
* Overall mean =              .369


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
          .354              .385
Factor B - STRAIN   Levels ( 1 - 2 ) :
          .366              .353
Factor C - ALUMINIUM    Levels ( 1 - 2 ) :
          .378              .361
Factor D - NITROGEN   Levels ( 1 - 2 ) :
          .385              .354


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                    1                 2
    1                .378              .330
    2                .395              .375

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                    1                 2
    1                .375              .333
    2                .380              .390

Factor A - SPEED  down and  Factor D - NITROGEN  across
                    1                 2
    1                .393              .315
    2                .378              .393

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                    1                 2
    1                .373              .400
    2                .383              .323

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                    1                 2
    1                .393              .380
    2                .378              .328

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                    1                 2
    1                .385              .370
    2                .385              .338
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.365	.390
2	.385	.275

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.380	.410
2	.380	.370

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.395	.360
2	.390	.270

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.392	.400
2	.365	.385

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.385	.365
2	.400	.265

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.385	375
2	.370	.410

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.375	.370
2	.410	.390

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.395	.370
2	.360	.285

* Four Way Interacion Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.370	.360
2	.420	.360

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.400	.370
2	.380	.170

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.380	.380
2	.400	.420

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.390	.370
2	.340	.400

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)	df	Sums of Squares	Mean Square
Total	15	.049	.003
A SPEED	1	.004	.004
B STRAIN	1	.005	.005
C ALUMINIUM	1	.001	.001
D NITROGEN	1	.004	.004
AB	1	.001	.001
AC	1	.003	.003
AD	1	.009	.009
BC	1	.008	.008
BD	1	.001	.001
CD	1	.001	.001
ABC	1	.002	.002
ABD	1	.002	.002
ACD	1	.007	.007
BCD	1	.000	.000
ABCD	1	.002	.002

```
*****************************************************************************
*                        FACTORIAL ANALYSIS OF VARIANCE                     *
*****************************************************************************
                        AGED WIRE - 100 C - 24 hours


DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  1      delta .2PS

MEANS
-----
* Overall mean =          333.63


* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
        329.25          338.00
Factor B - STRAIN   Levels ( 1 - 2 ) :
        316.75          350.50
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
        336.25          331.00
Factor D - NITROGEN   Levels ( 1 - 2 ) :
        331.63          335.63


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                1               2
    1          305.75          352.75
    2          327.75          348.25

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                1               2
    1          328.00          330.50
    2          344.50          331.50

Factor A - SPEED  down and  Factor D - NITROGEN  across
                1               2
    1          326.25          332.25
    2          337.00          339.00

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                1               2
    1          311.25          322.25
    2          361.25          339.75

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                1               2
    1          321.25          312.25
    2          342.00          359.00

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                1               2
    1          338.25          342.25
    2          333.88          329.00
```

* Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	292.50	319.00
2	363.50	342.00

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	338.00	325.50
2	359.00	337.50

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	299.00	312.50
2	353.50	352.00

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	343.50	312.00
2	330.50	366.00

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	330.00	326.00
2	322.50	338.50

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	338.50	358.50
2	343.50	319.50

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	310.50	312.00
2	332.00	312.50

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	350.00	372.50
2	334.00	343.50

* Four Way Interaction Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	288.00	297.00
2	310.00	328.00

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	372.00	355.00
2	335.00	349.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	333.00	327.00
2	354.00	297.00

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	328.00	390.00
2	333.00	342.00

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	11261.75	750.78
A	SPEED	1	306.25	306.25
B	STRAIN	1	4556.25	4556.25
C	ALUMINIUM	1	110.25	110.25
D	NITROGEN	1	64.00	64.00
AB		1	702.25	702.25
AC		1	240.25	240.25
AD		1	16.00	16.00
BC		1	1056.25	1056.25
BD		1	676.00	676.00
CD		1	256.00	256.00
ABC		1	240.25	240.25
ABD		1	1681.00	1681.00
ACD		1	1296.00	1296.00
BCD		1	25.00	25.00
ABCD		1	36.00	36.00

```
*****************************************************************************
*                      FACTORIAL ANALYSIS OF VARIANCE                       *
*****************************************************************************
                      AGED WIRE - 100 C - 24 hours

DESIGN
------
   Number of factors =  4
    No. of levels of factor A =  2
    No. of levels of factor B =  2
    No. of levels of factor C =  2
    No. of levels of factor D =  2
    No. of major replications (blocks) =  1
    No. of minor replications (samples) =  1

Subfiles will be ignored
Response variable(s) are :
Variable no.  4       SHEAR EL

MEANS
-----
* Overall mean =              .184

* Main Effect Means :

Factor A - SPEED   Levels ( 1 - 2 ) :
          .164               .204
Factor B - STRAIN   Levels ( 1 - 2 ) :
          .310               .058
Factor C - ALUMINIUM   Levels ( 1 - 2 ) :
          .224               .144
Factor D - NITROGEN   Levels ( 1 - 2 ) :
          .229               .139


* Two Way Interaction Means :

Factor A - SPEED  down and  Factor B - STRAIN  across
                  1                  2
    1              .278               .050
    2              .343               .065

Factor A - SPEED  down and  Factor C - ALUMINIUM  across
                  1                  2
    1              .223               .105
    2              .225               .183

Factor A - SPEED  down and  Factor D - NITROGEN  across
                  1                  2
    1              .198               .130
    2              .268               .148

Factor B - STRAIN  down and  Factor C - ALUMINIUM  across
                  1                  2
    1              .358               .263
    2              .090               .025

Factor B - STRAIN  down and  Factor D - NITROGEN  across
                  1                  2
    1              .343               .278
    2              .115              0.000

Factor C - ALUMINIUM  down and  Factor D - NITROGEN  across
                  1                  2
    1              .293               .155
    2              .175               .113
```

Three Way Interaction Means :

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.345	.210
2	.100	0.000

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor C - ALUMINIUM across

	1	2
1	.370	.315
2	.080	.050

Factor A - SPEED, Level 1
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.295	.260
2	.100	0.000

Factor A - SPEED, Level 2
Factor B - STRAIN down and Factor D - NITROGEN across

	1	2
1	.390	.295
2	.130	0.000

Factor A - SPEED, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.205	.160
2	.110	.100

Factor A - SPEED, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.280	.170
2	.240	.125

Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.385	.330
2	.300	.225

Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.180	0.000
2	.050	0.000

Four Way Interaction Means :

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.370	.320
2	.220	.200

Factor A - SPEED, Level 1 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.200	0.000
2	0.000	0.000

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 1
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.400	.340
2	.380	.250

Factor A - SPEED, Level 2 Factor B - STRAIN, Level 2
Factor C - ALUMINIUM down and Factor D - NITROGEN across

	1	2
1	.160	0.000
2	.100	0.000

ANOVA TABLE

Factorial Analysis of Variance

Source (Name)		df	Sums of Squares	Mean Square
Total		15	.346	.023
A	SPEED	1	.006	.006
B	STRAIN	1	.255	.255
C	ALUMINIUM	1	.026	.026
D	NITROGEN	1	.032	.032
AB		1	.003	.003
AC		1	.006	.006
AD		1	.002	.002
BC		1	.001	.001
BD		1	.003	.003
CD		1	.003	.003
ABC		1	.000	.000
ABD		1	.000	.000
ACD		1	.004	.004
BCD		1	.006	.006
ABCD		1	.000	.000

APPENDIX D : RESULTS OF YATES' ANALYSIS OF FACTORIAL EXPERIMENTS.

As-Drawn Wire - 0.2% Proof Stress /MPa

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	1996.000	3992.000	7943.000	16165.000	32033.000	4004.125
2	a	1956.000	3951.000	8222.000	15868.000	-285.000	-35.625
3	b	1979.000	4060.000	7733.000	-29.000	3.000	0.375
4	ab	1972.000	4162.000	8135.000	-256.000	-131.000	-16.375
5	c	2022.000	3897.000	-7.000	61.000	681.000	85.125
6	ac	2038.000	3836.000	-22.000	-58.000	-185.000	-23.125
7	bc	4100.000	4066.000	-43.000	-61.000	207.000	25.875
8	abc	2062.000	4069.000	-213.000	-70.000	-99.000	-12.375
9	d	1957.000	0.000	-61.000	279.000	-297.000	-37.125
10	ad	1940.000	-7.000	102.000	402.000	-227.000	-28.375
11	bd	1931.000	16.000	-61.000	-15.000	-119.000	-14.875
12	abd	1965.000	-39.000	3.000	-170.000	-9.000	-1.125
13	cd	2071.000	-17.000	-7.000	143.000	123.000	15.375
14	acd	1995.000	-26.000	-54.000	64.000	-155.000	-19.375
15	bcd	2103.000	-76.000	-9.000	-47.000	-79.000	-9.875
16	abcd	1986.000	-137.000	-61.000	-52.000	-5.000	-0.625

Sum B Squared (for three and four way interactions = 40173.000
t(95%) = 2.015 t(90%) = 1.476 s = 22.409
Estimated Error at 90% Confidence Limit = 22.577
Estimated Error at 80% Confidence Limit = 16.538

As-Drawn Wire - Shear Elongation /m

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	0.455	0.905	1.823	3.774	7.543	0.943
2	a	0.450	0.938	1.853	3.787	0.113	0.014
3	b	0.475	0.977	1.827	0.106	-0.015	-0.002
4	ab	0.463	0.876	1.960	0.007	-0.085	-0.011
5	c	0.470	0.897	0.063	-0.148	0.063	0.008
6	ac	0.507	0.930	0.043	0.133	0.013	0.002
7	bc	0.435	0.930	-0.013	-0.118	0.013	0.002
8	abc	0.441	1.030	0.020	0.033	0.103	0.013
9	d	0.450	0.075	-0.047	-0.070	0.011	0.001
10	ad	0.447	-0.012	-0.101	0.133	-0.099	-0.012
11	bd	0.470	0.037	0.033	-0.020	0.281	0.035
12	abd	0.460	0.006	0.100	0.053	0.151	0.019
13	cd	0.470	-0.003	-0.087	-0.054	0.203	0.025
14	acd	0.460	-0.010	-0.031	0.067	0.053	0.007
15	bcd	0.500	-0.010	-0.007	0.056	0.121	0.015
16	abcd	0.530	0.030	0.040	0.047	-0.009	-0.001

Sum B Squared (for three and four way interactions = 0.051
t(95%) = 2.015 t(90%) = 1.476 s = 0.025
Estimated Error at 90% Confidence Limit = 0.025
Estimated Error at 80% Confidence Limit = 0.019

Wire Aged at 50°C for 2h - *Increase in 0.2% Proof Stress /MPa*

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	S	S/B
1	1	29.000	24.000	77.000	301.000	568.000	71.000
2	a	-5.000	53.000	224.000	267.000	6.000	0.750
3	b	30.000	112.000	122.000	-27.000	60.000	7.500
4	ab	23.000	112.000	148.000	33.000	118.000	14.750
5	c	71.000	49.000	-41.000	29.000	170.000	21.250
6	ac	41.000	82.000	14.000	31.000	184.000	23.000
7	bc	34.000	76.000	-48.000	101.000	-82.000	-10.250
8	abc	78.000	67.000	81.000	17.000	80.000	10.000
9	d	30.000	-34.000	29.000	147.000	-34.000	-4.250
10	ad	18.000	-7.000	0.000	23.000	60.000	7.500
11	bd	55.000	-30.000	42.000	55.000	2.000	0.250
12	abd	27.000	44.000	-11.000	129.000	-84.000	-10.500
13	cd	25.000	-20.000	27.000	-29.000	-124.000	-15.500
14	acd	53.000	-28.000	74.000	-53.000	74.000	9.250
15	bcd	7.000	28.000	-8.000	47.000	-24.000	-3.000
16	abcd	60.000	53.000	25.000	33.000	-14.000	-1.750

Sum S Squared (for three and four way interactions = 19704.000

t(95%) = 2.015 t(90%) = 1.476 s = 15.694

Estimated Error at 90% Confidence Limit = 15.812

Estimated Error at 80% Confidence Limit = 11.582

Wire Aged at 50°C for 500h - Increase in 0.2% Proof Stress

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	170.000	330.000	662.000	1649.000	3199.000	399.875
2	a	160.000	332.000	987.000	1550.000	-45.000	-5.625
3	b	144.000	578.000	770.000	-39.000	-181.000	-22.625
4	ab	188.000	409.000	780.000	-6.000	107.000	13.375
5	c	328.000	383.000	34.000	-167.000	335.000	41.875
6	ac	250.000	387.000	-73.000	-14.000	15.000	1.875
7	bc	202.000	398.000	-64.000	137.000	-193.000	-24.125
8	abc	207.000	381.000	58.000	-30.000	83.000	10.375
9	d	197.000	-10.000	2.000	325.000	-99.000	-12.375
10	ad	165.000	44.000	-169.000	10.000	33.000	4.125
11	bd	220.000	-78.000	4.000	-167.000	153.000	19.125
12	abd	167.000	5.000	-18.000	122.000	-167.000	-20.875
13	cd	168.000	-11.000	54.000	-171.000	-315.000	-39.375
14	acd	211.000	-53.000	83.000	-22.000	229.000	28.625
15	bcd	173.000	23.000	-42.000	29.000	149.000	18.625
16	abcd	208.000	35.000	12.000	54.000	25.000	3.125

Sum B Squared (for three and four way interactions = %110045.000
t(95%) = 2.015 t(90%) = 1.476 s = 37.089
Estimated Error at 90% Confidence Limit = 37.367
Estimated Error at 80% Confidence Limit = 27.371

Wire Aged at 50°C for 500h - Shear Elongation /mm

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	0.360	0.760	1.440	2.990	6.440	0.805
2	a	0.400	0.880	1.350	3.450	-0.220	-0.028
3	b	0.440	0.740	1.680	-0.150	-0.060	-0.008
4	ab	0.440	0.610	1.770	-0.070	0.040	0.005
5	c	0.380	0.800	0.040	-0.010	-0.200	-0.025
6	ac	0.360	0.880	-0.190	-0.050	-0.420	-0.052
7	bc	0.390	0.950	0.060	-0.190	-0.460	-0.058
8	abc	0.220	0.820	-0.130	0.230	-0.080	-0.010
9	d	0.410	0.040	0.120	-0.290	0.460	0.057
10	ad	0.390	0.000	-0.130	0.090	0.080	0.010
11	bd	0.400	-0.020	0.080	-0.230	-0.040	-0.005
12	abd	0.480	-0.170	-0.130	-0.190	0.420	0.053
13	cd	0.540	-0.020	-0.040	-0.250	0.380	0.048
14	acd	0.410	0.080	-0.150	-0.210	0.040	0.005
15	bcd	0.410	-0.130	0.100	-0.110	0.040	0.005
16	abcd	0.410	0.000	0.130	0.030	0.140	0.018

Sum B Squared (for three and four way interactions = 0.206
t(95%) = 2.015 t(90%) = 1.476 s = 0.051
Estimated Error at 90% Confidence Limit = 0.051
Estimated Error at 80% Confidence Limit = 0.037

Wire Aged at 50°C for 1000h - Increase in 0.2% Proof Stress

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	G	G/G
1	1	180.000	375.000	835.000	1891.000	3630.000	453.750
2	a	195.000	460.000	1056.000	1739.000	38.000	4.750
3	b	193.000	553.000	822.000	109.000	62.000	7.750
4	ab	267.000	503.000	917.000	-71.000	-14.000	-1.750
5	c	281.000	381.000	89.000	35.000	316.000	39.500
6	ac	272.000	441.000	20.000	27.000	56.000	7.000
7	bc	237.000	475.000	-98.000	97.000	-226.000	-28.500
8	abc	266.000	442.000	27.000	-111.000	28.000	3.500
9	d	195.000	15.000	85.000	221.000	-152.000	-19.000
10	ad	185.000	74.000	-50.000	95.000	-180.000	-22.500
11	bd	265.000	-9.000	80.000	-69.000	-8.000	-1.000
12	abd	176.000	29.000	-33.000	125.000	-208.000	-26.000
13	cd	223.000	-9.000	59.000	-135.000	-126.000	-15.750
14	acd	252.000	-89.000	38.000	-93.000	194.000	24.250
15	bcd	222.000	29.000	-80.000	-21.000	42.000	5.250
16	abcd	220.000	-2.000	-31.000	49.000	70.000	8.750

Sum G Squared (for three and four way interactions = 88348.000
t(95%) = 2.015 t(90%) = 1.476 s = 33.232
Estimated Error at 90% Confidence Limit = 33.481
Estimated Error at 80% Confidence Limit = 24.525

Wire Aged at 50°C for 1000h - Shear Elongation /mm

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	G	G/G
1	1	0.400	0.812	1.567	2.807	5.917	0.740
2	a	0.412	0.755	1.240	3.110	-0.473	-0.059
3	b	0.405	0.570	1.580	-0.423	-0.027	-0.003
4	ab	0.350	0.670	1.530	-0.050	-0.097	-0.012
5	c	0.410	0.750	-0.043	0.043	-0.377	-0.047
6	ac	0.160	0.830	-0.380	-0.070	-0.467	-0.058
7	bc	0.400	0.840	0.040	0.053	-0.073	-0.009
8	abc	0.270	0.690	-0.090	-0.150	0.077	0.010
9	d	0.340	0.012	-0.057	-0.327	0.303	0.038
10	ad	0.390	-0.055	0.100	-0.050	0.373	0.047
11	bd	0.410	-0.250	0.080	-0.337	-0.113	-0.014
12	abd	0.420	-0.130	-0.150	-0.130	-0.203	-0.025
13	cd	0.410	0.030	-0.067	0.157	0.277	0.035
14	acd	0.430	0.010	0.120	-0.230	0.207	0.026
15	bcd	0.400	0.020	-0.020	0.187	-0.387	-0.048
16	abcd	0.290	-0.110	-0.130	-0.110	-0.297	-0.037

Sum G Squared (for three and four way interactions = 0.328
t(95%) = 2.015 t(90%) = 1.476 s = 0.064
Estimated Error at 90% Confidence Limit = 0.065
Estimated Error at 80% Confidence Limit = 0.047

Wire Aged at 80°C for 48h - Increase in 0.2% Proof Stress

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	G	G/8
1	1	251.000	481.000	986.000	2121.000	4166.000	520.750
2	a	230.000	485.000	1155.000	2045.000	-52.000	-6.500
3	b	236.000	611.000	963.000	-69.000	-84.000	-10.500
4	ab	249.000	544.000	1082.000	17.000	6.000	0.750
5	c	320.000	463.000	-8.000	-65.000	308.000	38.500
6	ac	291.000	500.000	-61.000	-21.000	74.000	9.250
7	bc	288.000	570.000	-55.000	31.000	-166.000	-20.750
8	abc	256.000	542.000	72.000	-25.000	4.000	0.500
9	d	237.000	-21.000	4.000	189.000	-76.000	-9.500
10	ad	226.000	13.000	-67.000	119.000	96.000	12.750
11	bd	272.000	-29.000	37.000	-53.000	42.300	5.250
12	abd	228.000	-32.000	-58.000	127.000	-56.000	-7.000
13	cd	269.000	-11.000	34.000	-71.000	-70.000	-8.750
14	acd	301.000	-44.000	-3.000	-95.000	180.000	22.500
15	bcd	236.000	32.000	-33.000	-37.000	-24.000	-3.000
16	abcd	276.000	40.000	8.000	41.000	78.000	9.750

Sum G Squared (for three and four way interactions = 42212.000
t(95%) = 2.015 t(90%) = 1.476 s = 22.971
Estimated Error at 90% Confidence Limit = 23.143
Estimated Error at 80% Confidence Limit = 16.952

Wire Aged at 80°C for 48h - Shear Elongation /mm

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	G	G/8
1	1	0.390	0.800	1.840	2.460	3.260	0.658
2	a	0.410	0.840	0.820	2.800	-0.580	-0.073
3	b	0.390	0.310	1.540	-0.500	0.180	0.023
4	ab	0.450	0.510	1.260	-0.080	-0.100	-0.012
5	c	0.310	0.840	0.080	0.240	-1.100	-0.137
6	ac	0.000	0.700	-0.580	-0.060	-1.060	-0.132
7	bc	0.350	0.590	0.160	0.080	0.380	0.047
8	abc	0.120	0.670	-0.240	-0.180	-0.260	-0.032
9	d	0.390	0.020	0.040	-0.820	0.340	0.043
10	ad	0.450	0.060	0.200	-0.280	0.420	0.052
11	bd	0.300	-0.310	-0.140	-0.660	-0.300	-0.038
12	abd	0.400	-0.270	0.080	-0.400	-0.260	-0.033
13	cd	0.320	0.060	0.040	0.160	0.540	0.067
14	acd	0.290	0.100	0.040	0.220	0.260	0.032
15	bcd	0.450	-0.010	0.040	0.000	0.060	0.007
16	abcd	0.220	-0.230	-0.220	-0.260	-0.260	-0.033

Sum G Squared (for three and four way interactions = 0.274
t(95%) = 2.015 t(90%) = 1.476 s = 0.059
Estimated Error at 90% Confidence Limit = 0.059
Estimated Error at 80% Confidence Limit = 0.043

Wire Aged at 100°C for 2h - Increase in 0.2% Proof Stress

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	178.000	331.000	731.000	1692.000	3259.000	407.375
2	a	156.000	400.000	961.000	1567.000	57.000	7.125
3	b	186.000	499.000	741.000	0.000	-19.000	-2.375
4	ab	214.000	462.000	826.000	57.000	87.000	10.875
5	c	276.000	360.000	9.000	32.000	315.000	39.375
6	ac	223.000	381.000	-9.000	-51.000	97.000	12.125
7	bc	209.000	449.000	-29.000	144.000	-199.000	-24.875
8	abc	253.000	377.000	86.000	-37.000	91.000	11.375
9	d	172.000	-19.000	69.000	230.000	-125.000	-15.625
10	ad	185.000	28.000	-27.000	85.000	57.000	7.125
11	bd	210.000	-53.000	21.000	-18.000	-83.000	-10.375
12	abd	171.000	44.000	-72.000	115.000	-201.000	-25.125
13	cd	291.000	10.000	47.000	-108.000	-145.000	-18.125
14	acd	248.000	-39.000	97.000	-93.000	133.000	16.625
15	bcd	169.000	47.000	-49.000	50.000	13.000	1.625
16	abcd	208.000	39.000	-8.000	41.000	-9.000	-1.125

Sum B Squared (for three and four way interactions = 66621.000
t(95%) = 2.015 t(90%) = 1.476 s = 26.858
Estimated Error at 90% Confidence Limit = 29.074
Estimated Error at 80% Confidence Limit = 21.297

Wire Aged at 100°C - Shear Elongation /em

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	0.370	0.730	1.510	2.830	5.970	0.739
2	a	0.360	0.780	1.320	3.080	-0.250	-0.031
3	b	0.420	0.770	1.580	-0.310	-0.130	-0.016
4	ab	0.360	0.550	1.500	0.060	-0.130	-0.016
5	c	0.400	0.760	-0.070	-0.170	-0.270	-0.034
6	ac	0.370	0.820	-0.240	0.040	-0.150	-0.019
7	bc	0.380	0.760	0.020	-0.230	-0.350	-0.044
8	abc	0.170	0.740	0.040	0.100	-0.070	-0.009
9	d	0.380	-0.010	0.050	-0.190	0.250	0.031
10	ad	0.380	-0.060	-0.220	-0.080	0.370	0.046
11	bd	0.400	-0.030	0.060	-0.170	0.210	0.026
12	abd	0.420	-0.210	-0.020	0.020	0.330	0.041
13	cd	0.390	0.000	-0.050	-0.270	0.110	0.014
14	acd	0.370	0.020	-0.180	-0.080	0.190	0.024
15	bcd	0.340	-0.020	0.020	-0.130	0.190	0.024
16	abcd	0.400	0.060	0.080	0.060	0.190	0.024

Sum B Squared (for three and four way interactions = 0.222
t(95%) = 2.015 t(90%) = 1.476 s = 0.053
Estimated Error at 90% Confidence Limit = 0.053
Estimated Error at 80% Confidence Limit = 0.039

Wire Aged at 100°C for 24h - Increase in 0.2% Proof Stress /MPa

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	291.000	588.000	1226.000	2444.000	5348.000	668.500
2	a	297.000	638.000	1418.000	2704.000	32.000	4.000
3	b	310.000	729.000	1311.000	74.000	-42.000	-5.250
4	ab	328.000	689.000	1393.000	8.000	-54.000	-6.750
5	c	374.000	660.000	24.000	10.000	274.000	34.250
6	ac	355.000	651.000	0.000	-52.000	110.000	13.750
7	bc	335.000	718.000	-53.000	50.000	-124.000	-15.500
8	abc	354.000	675.000	71.000	-104.000	24.000	3.000
9	d	333.000	6.000	30.000	192.000	60.000	7.500
10	ad	327.000	18.000	-80.000	82.000	-16.000	-2.000
11	bd	354.000	-19.000	-9.000	-24.000	-62.000	-7.750
12	abd	297.000	19.000	-42.000	134.000	-154.000	-19.250
13	cd	328.000	-6.000	12.000	-90.000	-110.000	-13.750
14	acd	390.000	-57.000	36.000	-34.000	158.000	19.750
15	bcd	333.000	62.000	-51.000	26.000	56.000	7.000
16	abcd	342.000	9.000	-53.000	-2.000	-28.000	-3.500

Sum S Squared (for three and four way interactions = 53176.000
t(95%) = 2.015 t(90%) = 1.476 s = 25.782
Estimated Error at 90% Confidence Limit = 25.975
Estimated Error at 80% Confidence Limit = 19.027

Wire Aged at 100°C for 24h - Shear Elongation /mm

a = Nitrogen b = Aluminium c = Strain d = Speed

Expt No	Treatment	Response	K2	K3	K4	B	B/8
1	1	0.370	0.690	1.110	1.310	2.940	0.368
2	a	0.320	0.420	0.200	1.630	-0.720	-0.090
3	b	0.220	0.200	1.370	-0.270	-0.640	-0.080
4	ab	0.200	0.000	0.260	-0.450	0.220	0.028
5	c	0.200	0.740	-0.070	-0.470	-2.020	-0.253
6	ac	0.000	0.630	-0.200	-0.170	-0.200	-0.025
7	bc	0.000	0.160	-0.190	0.230	0.120	0.015
8	abc	0.000	0.100	-0.260	-0.010	0.300	0.037
9	d	0.400	-0.050	-0.270	-0.910	0.320	0.040
10	ad	0.340	-0.020	-0.200	-1.110	-0.180	-0.022
11	bd	0.380	-0.200	-0.110	-0.130	0.300	0.037
12	abd	0.250	0.000	-0.060	-0.070	-0.240	-0.030
13	cd	0.160	-0.060	0.030	0.070	-0.200	-0.025
14	acd	0.000	-0.130	0.200	0.050	0.060	0.008
15	bcd	0.100	-0.160	-0.070	0.170	-0.020	-0.002
16	abcd	0.000	-0.100	0.060	0.130	-0.040	-0.005

Sum S Squared (for three and four way interactions = 0.153
t(95%) = 2.015 t(90%) = 1.476 s = 0.044
Estimated Error at 90% Confidence Limit = 0.044
Estimated Error at 80% Confidence Limit = 0.032

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