A STUDY OF THE EFFECT OF CRYOGENIC TREATMENT ON THE PERFORMANCE OF HIGH SPEED STEEL TOOLS AND CARBIDE INSERTS

A THESIS SUBMITTED IN PARTIAL FULFILMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Technology

in

Mechanical Engineering

By

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Department of Mechanical Engineering

National Institute of Technology

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CERTIFICATE

This is to certify that thesis entitled, “A STUDY OF THE EFFECT OF CRYOGENIC TREATMENT ON THE PERFORMANCE OF HIGH SPEED STEEL TOOLS AND CARBIDE INSERTS**”** submitted by Ms. AMRITA PRIYADARSHINI in partial fulfillment of the requirements for the award of Master of Technology Degree in Mechanical Engineering with specialization in “Production Engineering” at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not been submitted to any other university/ institute for award of any Degree or Diploma.

Date: 21.05.07

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ABSTRACT

Cryogenic treatment has been acknowledged by some as means of extending tool life of many cutting tool materials, thus improving productivity significantly. However real mechanisms which guarantee better tool performance are still dubious. This implies the need of further investigations in order to control the technique more significantly. Studies on cryogenically treated HSS tools show microstructural changes in material that can influence tool lives. However little research has been done on other cutting tool materials. Cryogenic treatment of carbides has yet to be extensively studied. This work aims to study the effect of cryogenic treatment on M2 and S400 as well as Carbide inserts of SNMS120408 and SNMG120412MP grades. The tools were cryo-treated for 24 hours. The flank wear tests, sliding wear tests and hardness tests were conducted. In the process of ascertaining these findings, it was shown in this study that in flank wear tests cryogenically treated tool showed an increase in tool life. However in sliding wear test, weight loss in case of cryogenically treated tools was found to be more indicating the fact that the tool becomes more brittle after cryogenic treatment due to transformation of retained austenite to martensite as well as due to carbide refinement. Microstructural analysis and SEM analysis were done to support the results obtained.

Performance of cryogenically treated tools largely depends upon the cutting conditions. Hence design of experiment (DOE) was employed to study the effect of cutting parameters on tool wear and tool life equations were developed illustrating the significant factors that affect performance of cryogenically treated tools.

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CHAPTER 1

INTRODUCTION

1.1. BACKGROUND

Metal cutting process forms the basis of the engineering industry and is involved either directly or indirectly in the manufacture of nearly every product of our modern civilization. The cutting tool is one of the important elements in realizing the full potential out of any metal cutting operation. Over the years the demands of economic competition have motivated a lot of research in the field of metal cutting leading to the evolution of new tool materials of remarkable performance and vast potential for an impressive increase in productivity. Changes in work piece materials, manufacturing processes and even government regulations catalyze parallel advances in metal cutting tooling technology.

As manufacturers continually seek and apply new manufacturing materials that are lighter and stronger and therefore more fuel efficient it follows that cutting tools must be so developed that can machine new materials at the highest possible productivity. The most important elements in the design of cutting tools is the material construction and there judicious selection. The properties that a tool material must process are as follows:

- Capacity to retain form stability at elevated temperatures during high cutting speeds.
- Cost and ease of fabrication
- High resistance to brittle fracture
- Resistance to diffusion
- Resistance to thermal and mechanical shock

Developmental activities in the area of cutting tool materials are guided by the knowledge of the extreme conditions of stress and temperature produced at the tool-work piece interface. Tool wear occurs by one or more complex mechanisms which includes abrasive wear, chipping at the cutting edge, thermal cracking etc. Since most of these processes are greatly accelerated by increased temperatures, the more obvious requirements for tool materials are improvements in physical, mechanical and chemical properties at elevated temperature.

1.2. TECHNOLOGICAL DEVELOPMENT

Tool materials have improved rapidly during the last sixty years and in many instances, the development of new tool materials has necessitated a change in the design trend of machine tools to make full use of the potentialities of tool materials for high productivity. Progress from carbon tool steels, high speed steels and cast alloys to carbides and ceramics has facilitated the application of higher speeds at each stage of development. With the advent of carbides and ceramics radical changes have taken place in the design of tool holders and cutters and the concept of the throw away tipped tool where the insert is held mechanically and is discarded after use represents a major advance in the metal removing technology of modern times.

Till 1900 machining was performed by plain carbon tool steel, shortly after 1900 high speed steel was introduced which has undergone many modifications giving rise to several types of HSS. The next notable improvement came with the introduction of cobalt bonded sintered tungsten carbide. However shortage of tungsten has led to the development of many non-tungsten cutting tool materials. Ceramic tools exhibit very high hardness and wear resistance facilitating the use of higher cutting speeds. UCON a new tool material consisting of columbium, tungsten, titanium permits 60% increase in the cutting speed when compared with tungsten carbide. Cubic Boron Nitride with hardness next to diamond which is claimed to give speed 5 to 8 times that of carbide can be used to cut hardened materials.

Polycrystalline diamond bonded to tungsten carbide substrate has been successfully employed for machining non-ferrous materials.

But no single tool material has all the desired properties to withstand wide range of stresses, temperatures, abrasion and thermal shock to which a cutting tool is subjected during metal cutting. Each cutting tool has a unique combination of properties that are important to its performance. Hence by fine tuning combinations of tool material compositions, coatings and geometries tool makers enable users to make more parts faster and at reduced cost.

Traditional tool materials such as HSS continue to undergo substantial improvement in there properties through suitable modifications in their composition by optimizing the processing technique as well as incorporating various surface treatments. As a result of these technological advances HSS are still in use having surviving competition from carbides and ceramics. Carbide because of the ability to retain its strength and hardness at very high temperatures, to withstand cutting speeds 6 or more than 6 times higher than tools of HSS and the economical price has become a logical choice of many cutting industries. However with the incorporation of suitable surface treatments, its service life as well as its properties can be enhanced even more.

1.3. SURFACE TREATMENTS

Advances in manufacturing technologies (increased cutting speeds, dry machining, etc.) triggered the fast commercial growth of various surface treatments for cutting tools; on the other hand these surface coating technologies enabled these advances in manufacturing technologies. No single treatment will solve every problem and their use should be restricted to those operations where extra expense of the treatment can be justified by a substantial performance gain.

The processes of surface treatments more formally surface engineering tailor the surfaces of engineering materials to:

- Control friction and wear
- Improve corrosion resistance
- Change physical property
- Vary appearance
- Reduce cost

Ultimately the functions on service lines of the materials can be improved.

Common surface treatments can be divided into two major categories:

a. Treatments that cover surfaces
b. Treatments that alter surfaces

Treatments covering surfaces:

- Organic coatings such as paints, cements, laminates, fused powders, lubricants, or floor toppings on the surfaces of materials
- Inorganic coating such as electroplating, autocatalytic platings (electroless platings), conversion coatings, thermal sprayings, hot dippings, furnace fusing, or coat thin films on the surfaces of the materials (PVD and CVD)

Treatments altering surfaces:

- High energy treatments such as *i*on implantation, laser glazing/fusion, and electron beam treatment.
- Diffusion treatments include boronizing, and other high temperature reaction processes, e.g., TiC, VC.
- Hardenings such as flame, induction, laser or electron beam
- Heavy diffusion treatments include carburizing, nitriding, and carbonitriding
- Special treatments such as cryogenic, magnetic and sonic treatment

Cryogenic treatment is an inexpensive one time permanent treatment affecting the entire section or bulk of the component unlike coatings. The treatment is an add on process over conventional heat treatment in which the samples are cooled down to prescribed cryogenic temperature for a long time and then heated back to room temperature. It is believed that life of cutting tool get substantially extended due to cryogenic treatment. However, researchers have been skeptical about the process because it imparts no apparent visible change. Moreover mechanism is also unpredictable and research articles are also not sufficient to support the treatment. So in general cryogenic treatment is still in the dormant level.

Over the past few years there has been an increase in interest in the application of cryogenic temperature to different materials. Some literature says that the cryogenic treatment can improve the life span would depend a lot on the cutting conditions. Hence various research works are being carried out to study the effects of this treatment on the performance of various cutting tools so that it could be added to the regular heat treatment cycle for the components the production sector manufacture. However for evaluating the performance of the cutting tools it is very necessary to study the effect of cutting parameters (cutting speed, depth of cut and feed) on the tool wear. This necessitates planning experiments in advance so that maximum benefit can be derived from data obtained from organized sets of experiment. Designs of experiment (DOE) is one such approach that has proved to be a powerful technique in getting a quantitative relationship among the variables (in the form equations). One important benefit of DOE is that this not only evaluates the significant effect of each of the individual factors (parameters) but also determines the interaction effects among all the factors. When an interaction is large the corresponding main effect cease to have much meaning. Hence, it is very important to determine the interaction effects of various process variables to fully evaluate the performance of the tools.

1.4. OBJECTIVE

- To make a comparative study on the hardness and wear resistance of cryogenically treated HSS samples and carbide inserts with that of untreated tools.
- To study the effect of different cutting parameters on the tool life of cryogenically treated tool (HSS and carbides) and development of tool life equations employing design of experiment (DOE) technique.
- To study the microstructural changes.

CHAPTER 2

LITERATURE SURVEY

In recent decades, there has been an increase in interest in the application of cryogenic treatment to different materials. Research has shown that cryogenic treatment increases product life, and in most cases, provides additional qualities to the product, such as stress relieving. In the area of cutting tools, extensive study has been done on tool steels, which include high-speed steel (HSS) and medium carbon steels. It has been reported that cryogenic treatment can double the service life of HSS tools, and also increase hardness and toughness simultaneously [34][3]

Cryogenic treatment of cutting tool materials such as tungsten carbide, have yet to be extensively studied. Tungsten carbide has been proven to be much more efficient than HSS when machining hard materials such as steel itself. If cryogenic treatments can double the service life of HSS, it could probably do the same for tungsten carbide tools [31]. Unlike coatings that are only a superficial treatment, the cryogenic treatment is applied to the whole volume of the material, reaching the core of the tools. This guarantees maintenance of their properties even after regrinding or resharpening. One of the most prevalent claims in low-temperature treatment is an increase in wear resistance of certain steels [10][22][34] However, most researchers believe that cryogenic treatment promotes the complete transformation of retained austenite into martensite at cryogenic temperatures, which is attributed to improved wear resistance [14][32]. Others claim that cryogenic treatment facilitates the formation of fine carbides in the martensite, thus improving the wear resistance [22] [33]. However, the lack of common sense in the literature regarding to the metallurgical aspects that cryogenic treatment confers better wear resistance and consequently higher tool lives as well as contradictory results that are also encountered [3] [4][5] lead to many doubts and questions involving the practical application of this sort of treatment.

Several different cryogenic processes have been tested by researchers. These involve a combination of deep freezing and tempering cycles. Generally, they can be described as a controlled lowering of temperature from room temperature to the boiling point of liquid nitrogen (−196 °C), maintenance of the temperature for about twenty four hours, followed by a controlled raising of the temperature back to room temperature. Subsequent tempering processes may follow [30]. There are different levels of treatment temperatures. In order to avoid confusion, cryogenic treatment has been classified into shallow cryogenic treatment (SCT) and deep cryogenic treatment (DCT) depending upon the temperatures in which the material is treated [26].The common practice for shallow cryogenic treatment is to keep the specimens in a mechanical freezer at 193 K for 5 h and then exposed to room temperature. But in deep cryogenic treatment the materials are slowly brought down from room temperature to 77 K at 1.26 K/min, held at the same temperature for 24 h and subsequently brought back to room at 0.63 K/min. In order to achieve deep cold temperatures, materials cannot be directly kept in freezer at 77 K similar to that of shallow cryogenic treatment because the temperature difference is very high and fast cooling will lead to quench cracks.

The conventional heat treatment normally uses cooling conditions only until room temperature, which may leave some retained austenite on the microstructure. This fact must be considered during heat treatment of tool steels. This retained austenite is soft and unstable at lower temperatures that it is likely to transform into martensite under certain conducive conditions. It should be noted that freshly formed martensite is also brittle and only tempered martensite is acceptable. To further aggravate this problem the transformation of austenite to martensite yields a 4% volume expansion [29] causing distortion which cannot be ignored. Thus retained austenite should be alleviated to the maximum possible before any component or tool is put into service. The degree of undercooling decides the potential to transform retained austenite to martensite completely [15]. In this context cryogenic treatment is handy. It also causes the precipitation of finely dispersed carbides in the martensite. It would be the interest of researchers to quantify the benefits and also know the conditions at which the treatment derives maximum benefits. For instance in case of the eutectoid steel the M_f temperature is of approximately of −50 °C, therefore after quenching some percentage of retained austenite will be present [8]. Lately this structure can be transformed into martensite if the material is submitted to reheating or to a stress field, causing distortion on its body. This non-tempered martensite may cause cracks, particularly in complex shape tools made of highly alloyed steels [9]. The subzero treatment will transform a great deal of this retained austenite by reaching the M_f line, giving more dimensional stability in the tool microstructure.

The main variables during heat treatment have a great deal of influence on the results. A research done in steels equivalent to M2, varying the cryogenic cycles has quantified the precipitated particles and verified their influence onto the material properties [22]. Their research involved seven steel samples, each of them submitted to different heating and cooling (up to −70 °C) cycles. The microstructure was analyzed and the carbide particles quantified using SEM, X-ray difractometer, quantitative metallography and differential dilatometer. The results confirmed an increase in carbide precipitation (from 6.9% to 17.4%), a reduction of the retained austenite (from 42.6% to 0.9%) and an increase in the martensite content (from 66% to 81.7%). The machining tests carried out with bits in turning AISI 1050 steels showed a significant increase in tool lives of cryogenically treated tools. These results can be attributed to minimum quantity of retained austenite, higher amount of martensite content, higher density of fine carbides (smaller than 1 μm) and a more favourable distribution of the alloying elements among the carbide of the matrix.

When temperature was applied [6] in the range of −80 to −100 °C for periods of about 30 min–1 h, and the improvement on tool life was credited to the transformation of retained austenite (softer) into martensite (harder) and the production of a more stable structure. In general the addition of alloying elements lowers the M_s (temperature of the beginning of martensite transformation) and M_f (final transformation temperature) lines in a way that the latter dwells at subzero temperatures.

Barron [14] after cryogenically treating several materials including the M2 high speed steel at −84 °C (maintaining it at this temperature for 24 h) observed a significant improvement on the wear resistance in sliding abrasion tests [15] when compared to conventionally heat treated steel (quenched and tempered). When the temperature of the cryogenic treatment was reduced further to −196 °C, the wear resistance was increased even more. He has attributed the improvement of the wear resistance of these tools to another mechanism besides the transformation of the retained austenite into martensite. He verified that the tool steels submitted to conventional heat treatment presented only a small amount of retained austenite, but those submitted to cryogenic treatment showed better performance during machining. This new mechanism would be time and temperature dependent due to the long period (8 h or more) during which the tools would have to stay at cryogenic temperatures. Before the cryogenic treatment the microstructure showed relatively large carbides (20 μm) dispersed in the matrix. After the cryogenic treatment, carbide particles as small as 5 μm were found. The carbide refinement could in such a way contribute to the improvement of the wear resistance of the tool. Barron thus attributed this achievement both to austenite transformation and to the presence of hard and small carbide particles well distributed among the larger carbide particles within the martensite matrix [10].

Dong et al. [29] did a detailed study on the effects of varying the deep freezing and tempering cycles on high speed steel and confirmed that in tool steels, this treatment affects the material in two ways. Firstly, it eliminates retained austenite, and hence increases the hardness of the material. Secondly, this treatment initiates nucleation sites for precipitation of large numbers of very fine carbide particles, resulting in an increase in wear resistance.

Popandopulo and Zhukova [11] carried out dilatometry studies and microstructure analysis during cryogenic treatment. They observed volume reduction of the specimen at the temperature range of −90 to +20 °C. This behaviour was attributed to partial decomposition of the martensite and precipitation of carbon atoms at dislocation lines and formation of ultramicroscopic carbides.

Paulin [2] also verified the presence of fine precipitated carbide particles and their importance to the material properties. The precipitated carbides reduce internal tension of the martensite and minimize micro cracks susceptibility, while the uniform distribution of fine carbides of high hardness enhances the wear resistance. Huang et al. [12] confirmed that cryogenic treatment not only facilitate the carbide formation but can also make the carbide distribution more homogeneous.

Yun et al. [17] verified changes in the microstructure of M2 high speed steel when this material was submitted to different cycles of cryogenic treatment at −196 °C. Comparing the conventional quenching cycle with other cryogenic cycles it was observed increases of 11.5% in the bending strength, 43% in the toughness and changes in the room temperature and hot hardness. The results were again attributed to transformation of the retained austenite into martensite and precipitation of ultra-fine carbides, with this latter being considered the key point for the changes in the properties.

Molinari [18] found out that the deep cryogenic treatment (−196°C) of quenched and tempered high speed steel tools improves their properties; in particular, it increases the hardness and improves the hardness homogeneity, reduces the tool consumption and the down time for the equipments set up, thus leading to about 50% cost reduction [30].The greatest improvement in properties is obtained by carrying out the deep cryogenic treatment between quenching and tempering. However, a significant improvement can be obtained even by treating the tools at the end of the usual heat treatment cycle, i.e. the finished tools. This last solution is more flexible than the other one and can extend the use of the treatment to many practical applications [17]

Mohan Lal et al[19]., made a comparative study on wear resistance improvement of cryogenically treated samples with standard heat-treated samples through flank wear test and sliding wear test. Untempered samples when cryogenically treated yield 3%, 10% and 10.6% extra life over tempered and cryogenically treated T1, M2 and D3 samples, respectively. Hence it is suggested to cryogenically treat without tempering. Tempered samples when cryogenically treated at 133 K for 24 h yielded negative results, but when cryogenically treated at 93 K for 24 h the results were favourable. Hence tempered samples if treated at still lower temperatures may yield still better results on par with untempered cryotreated samples. This also suggested to conclude that the stabilization of phases that would take place during tempering requires sufficient degree of undercooling and time to get transformed to stable harder/tougher phases that offer better wear resistance. Cryogenic treatment done at 93 K as per the prescribed cycle yields 20% extra life as compared to the maximum life achieved through cold treatment. Cryogenic treatment at 93K for 24 hours [8] is superior to TiN coatings also. The effect of cryotreatment on TiN coating is not favourable which may be because of uneven contraction of the coating material and the substrate leading to incipient cracks at the interface. Hence cryotreatment should not follow TiN coating [14].

Meng and Tagashira [28] studied the wear resistance and microstructure of Fe–12Cr–Mo–V-1.4C tool steel both with and without cryogenic treatment. The study reveals that cryogenically treated samples show improvement from 110% to 600% through sliding wear test.

The conventionally heat-treated and cryogenically treated specimens showed the largest and smallest wear volume at all sliding distance, respectively. From the microstructure of the steel it is reported that the improvement in wear resistance after cryogenic treatment can be attributed to η-carbide precipitates.

It was found that wear resistance has been improved by 85% for shallow cryogenic treatment and 372% for deep cryogenic treatment over conventional heat treatment and also the wear resistance improvement of deep cryogenic treatment is 152% over shallow cryogenic treatment. Wear is found to increase linearly with load at constant sliding speeds and with sliding speed at constant loads [2]. Studies [10] show that the wear improvement of samples treated at 83 K (close to DCT) was approximately 2.6 times higher than the wear resistance of sample treated at 188 K (close to SCT). Also it was found that the improvement of wear resistance for the above alloys when treated at 188 K ranges by factors from 1.2 to 2.0 whereas the same alloys when treated at 83 K improves the wear resistance by factors ranging from 2.0 to 6.6.

Seah et al. [31] did some study on the effect of cryogenic treatment on tungsten carbide and found that such treatment increases its wear resistance. They attributed this to an increase in the number of η-phase particles after cryogenic treatment, a theory which he supported with photographs taken using a scanning electron microscope (SEM). The experimental procedures that were used to perform the cutting on the workpiece were "repeated turning operations". Such "repeated turning operations" refers to using the same cutting edge for subsequent cutting operations, instead of switching to a brand new cutting edge for each new cut. By doing so, they managed to show that cryogenically treated tungsten carbide tools had a much greater resistance to chipping compared to the untreated ones. In addition, the cryogenically treated tools also performed better than the untreated tools at higher cutting speeds.

So far, few researchers have proposed other mechanisms that explain the effect of cryogenic treatment on tungsten carbide. Bryson [32] attributes the wear resistance, and hence the increase in tool life, of carbide tools to the improvement in the holding strength of the binder after cryogenic treatment. He believes that cryogenic treatment also acts to relieve the stresses introduced during the sintering process under which carbide tools are produced. However, Bryson also warned that under certain conditions, cryogenic treatment would have little or no effect on carbide tools, such as when reprocessed carbides are used.

In a more recent work [36] it was verified that cryogenic treatment no doubt improves the resistance to chipping of tools and to a less significant extent, improves flank wear resistance but however, under certain conditions, such as prolonged exposure to high temperatures during long continuous cutting operations, cryogenically treated tools can lose their superior properties. In light of the fact that cryogenically treated tools perform best when the tool temperature is kept low, their effectiveness can be extended if coolants or suitable methods of cooling are used to keep the tool temperatures low. Hence, the validity of claims that cryogenic treatment can improve the lifespan of cutting tools would depend a lot on the cutting conditions. Tools under mild cutting conditions stand to gain from cryogenic treatment, but heavy duty cutting operations with long periods of heating of the cutting tool will not benefit from it.

The real mechanisms which guarantee better tool performance after cryogenic treatment are still dubious. This implies in the need of further investigation in order to control the technique more scientifically.

CHAPTER 3

CRYOGENIC TREATMENT

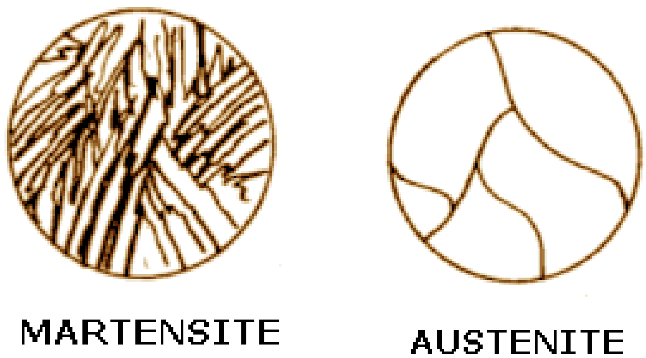
3.1. INTRODUCTION

Cryogenics is defined as the branches of physics and engineering that study very low temperatures, how to produce them, and how materials behave at those temperatures. Rather than the familiar temperature scales of Fahrenheit and Celsius, cryogenicists use the Kelvin and Rankine scales.

The word cryogenics literally means "the production of icy cold"; however the term is used today as a synonym for the low-temperature state. It is not well-defined at what point on the temperature scale refrigeration ends and cryogenics begins. The workers at the National Institute of Standards and Technology at Boulder, Colorado have chosen to consider the field of cryogenics as that involving temperatures below –180 °C (93.15 K). This is a logical dividing line, since the normal boiling points of the so-called permanent gases (such as helium, hydrogen, neon, nitrogen, oxygen, and normal air) lie below -180 °C while the Freon refrigerants, hydrogen sulfide, and other common refrigerants have boiling points above -180 °C. Cryogenic temperatures are achieved either by the rapid evaporation of volatile liquids or by the expansion of gases confined initially at pressures of 150 to 200 atmospheres. The expansion may be simple, that is, through a valve to a region of lower pressure, or it may occur in the cylinder of a reciprocating engine, with the gas driving the piston of the engine. The second method is more efficient but is also more difficult to apply.

Cryogenic treatment is a one-time permanent treatment process and it affects the entire cross-section of the material usually done at the end of conventional heat treatment process but before tempering. Also it is not a substitute process but rather a supplement to conventional heat treatment process. It is believed to improve wear resistance as well the surface hardness and thermal stability of various materials.

This treatment is done to make sure there is no retained austenite during quenching. When steel is at the hardening temperature, there is a solid solution of Carbon and Iron, known as Austenite. The amount of martensite formed at quenching is a function of the lowest temperature encountered. At any given temperature of quenching there is a certain amount of martensite and the balance is untransformed austenite. This untransformed austenite is very brittle and can cause loss of strength or hardness, dimensional instability, or cracking. Fig 3.1 shows the structure of austenite and martensite.



(a) (b)

Fig. 3.1. Structure of austenite and martensite

Quenches are usually done to room temperature. Most medium carbon steels and low alloy steels undergo transformation to 100 % martensite at room temperature. However, high carbon and high alloy steels have retained Austenite at room temperature. To eliminate retained Austenite, the temperature has to be lowered.

Liquefied gases, such as liquid nitrogen and liquid helium, are used in many cryogenic applications. Liquid nitrogen is the most commonly used element in cryogenics and is legally purchasable around the world. Liquid helium is also commonly used and allows for the lowest attainable temperatures to be reached. These gases are held in either special containers known as Dewar flasks, which are generally about six feet tall (1.8 m) and three feet (91.5 cm) in diameter, or giant tanks in larger commercial operations. Cryogenic transfer pumps are the pumps used on LNG piers to transfer Liquefied Natural Gas from LNG Carriers to LNG storage tanks.

3.2 THE MAKING OF LIQUID NITROGEN

A common method for production of liquid nitrogen is the liquefaction of air. Liquefaction is the phase change of a substance from the gaseous phase to the liquid phase. In the liquid nitrogen compressors or generators, air is compressed, expanded and cooled via the Joule-Thompson's effect as depicted in fig3.2 and fig. 3.3. **Fig.3.4** shows **the set up for making nitrogen.** Since nitrogen boils at a different temperature than oxygen, the nitrogen can be distilled out of the liquid air, recompressed and re-liquefied. Once liquid nitrogen is removed from the distillation chamber it is stored in a pressurized tank or a well insulated deewar flask.Liquid nitrogen is converted to a gas before it enters the chamber so that at no time does liquid nitrogen come in to contact with the parts assuring that the dangers of cracking from too rapid cooling are eliminated.

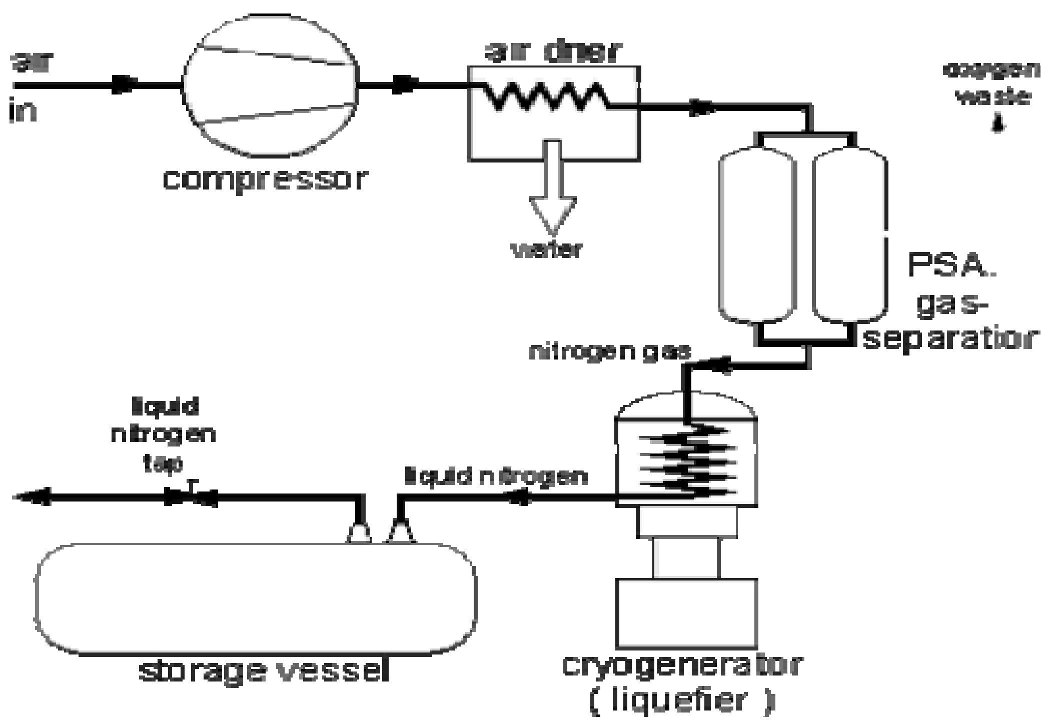


Fig. 3.2. Making of liquefied nitrogen

Fig. 3.4. Set up for nitrogen making

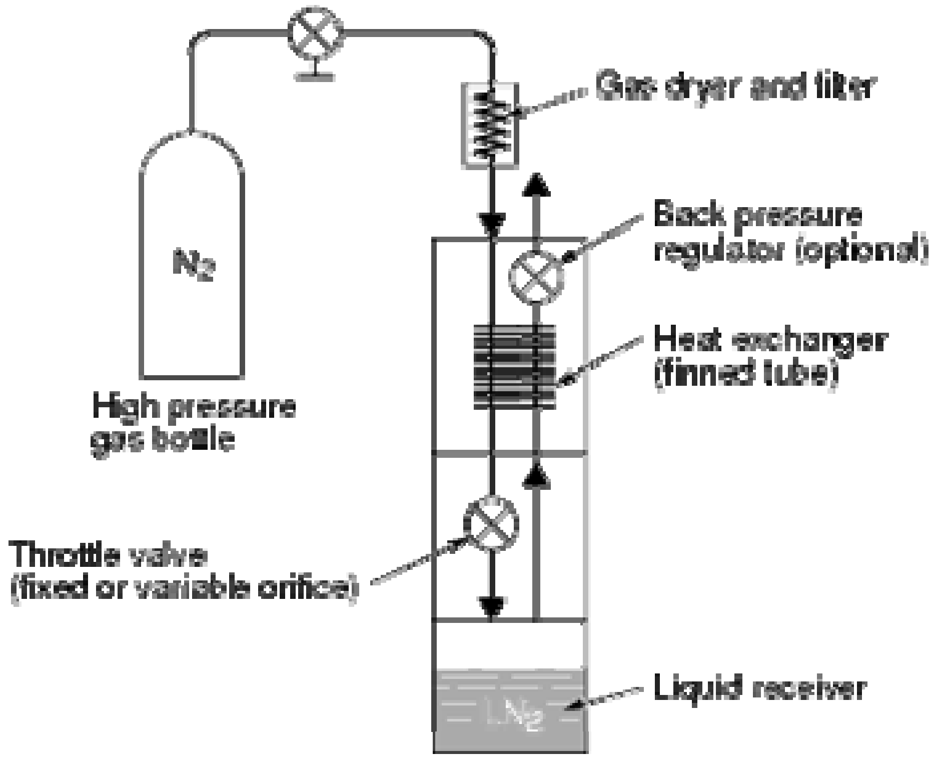


Fig. 3.3. Use of a Joule-Thomson system to generate a liquid cryogen.

3.3. CRYOGENIC TREATMENT PROCEDURE

The liquid nitrogen as generated from the nitogen plant is stored in storage vessels.With help of transfer lines, it is directed to a closed vacuum evacuated chamber called cryogenic freezer through a nozzle.The supply of liquid nitrogen into the cryo-freezer is operated with the help of soleniod valves.Inside the chamber gradual cooling occurs at a rate of 2º C /min from the room temperature to a temperature of -196º C.Once the sub zero temperature is reached, specimens are transferred to the nitrogen chamber or soaking chamber where in they are are stored for 24 hours with continiuos supply of liquid nitrogen. **Fig. 3.5** illustrates the **entire set up for cryogenic treatment**. The entire process is schematically shown in fig. 3.6.

Fig. 3.5. Photograph of the cryogenic treatment set up

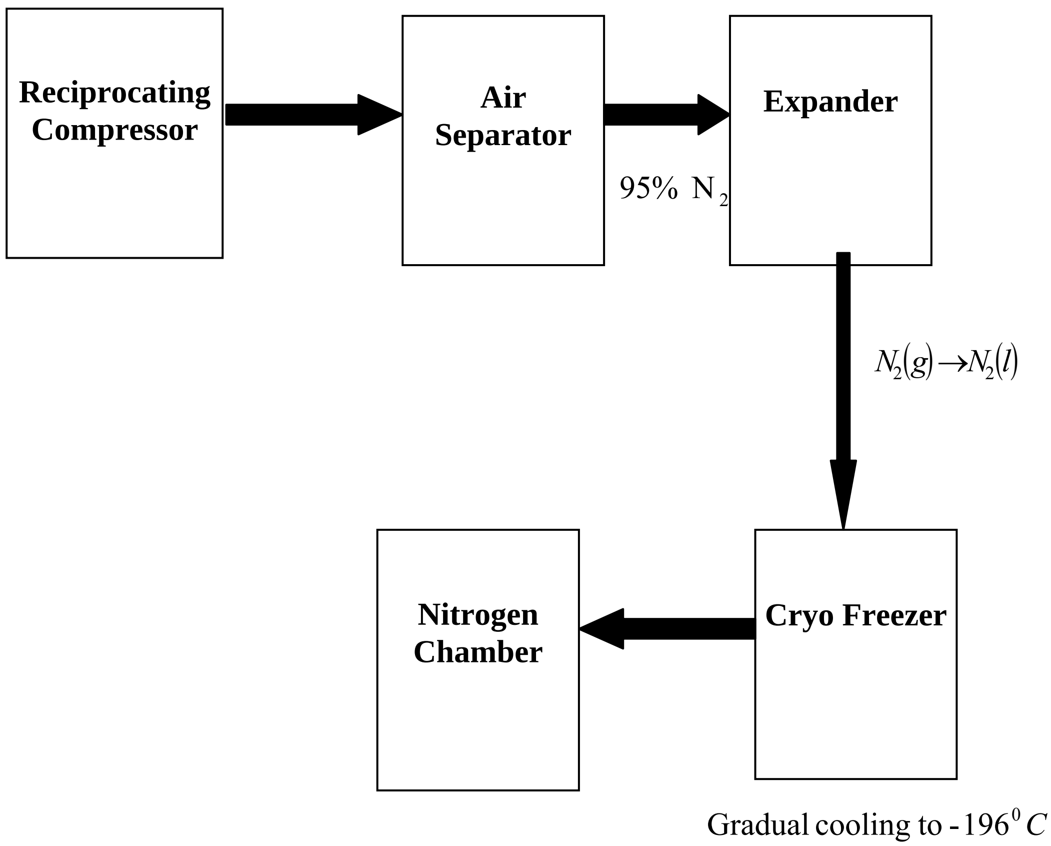


Fig. 3.5. Schematic representation of cryogenic treatment procedure

CHAPTER 4

DESIGN OF EXPERIMENT

4.1. INTRODUCTION

Design of Experiments is a statistical method of planning experiments in advance so that maximum benefit can be derived from data obtained from organized sets of experiments. Compared to single factor experiments where one parameter is varied at a time keeping other parameters constant, statistical design of experiment permits variation of several parameters at a time in a predetermined manner. This leads to reducing the number of experiments to the least possible for getting a quantitative relationship among the variables (in the form of equations) thereby saving considerable amount of time, money and material. Finally an important benefit of statistical design of experiment is its ability to determine the interaction effects of various process variables at their different levels, which are otherwise difficult to be obtained through single factor experiments since a single factor experiment is likely to provide only a number of disconnected pieces of information that cannot be easily put together.

In order to conduct an experiment on a single factor A, some decision must be taken on the levels of other factors viz., B, C, D, that are to be used in the experiment. The experiment reveals the effect of A on the particular combination of B, C and D, but no information is provided for predicting the effects of A with any other combination of B, C and D.

With a factorial approach, on the other hand, the effects of A are examined for every combination of B, C and D that is included in the experiment. Thus a great deal of information is accumulated both about the effects of the factors and their inter-relationship (interactions).

4.2. FACTORS

Factors are experimental variables that are controlled by the investigator. In order to investigate/optimize a desired parameter/property (called response), it is important to identify the probable factors that may influence the property. There may be a number of variables which may influence the response, but the magnitudes with which these factors affect the response are not the same. Consequently, less important factors may be kept at constant level while other important factors may be included in the study. This is critical, since when an important factor is fixed at a certain level, a false idea of the optimum is obtained and there is no guarantee that the fixed level is the optimal one. On the other hand if factors are increased to a large number, huge trials will be necessary. Therefore, when the number of factors is large it is necessary to resort to methods of eliminating less important factors.

Factors may be independent, i.e., the level of one factor can be varied independently of the levels of other factors. However, two or more factors may interact with one another, i.e., the effect on the response of one variable depends on the levels of the other variables. Interactions between the factors are obtained by varying the factors simultaneously in a statistically predetermined way rather than varying one factor at a time. Fig. 4.1 illustrates the different types of behavior between factors (x_1 and x_2) and the response (y).

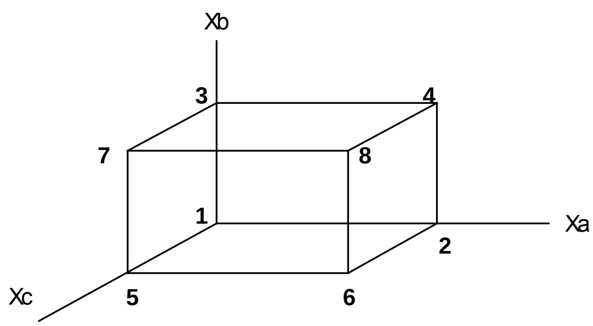


Fig. 4.1. Three factors at two levels

4.3. FACTORIAL DESIGN OF THE TYPE P^n

For the present work it is necessary to elucidate the effect of each factor and the possible ways in which each factor is modified by the variation of the others. In the design of P^n factorial experiments, n is a positive integer which denotes the number of factors varied at a time and P is the number of levels in which each factor is varied.

The simplest and the most common type of factorial design is of the type 2^n, where each factor is varied at two levels. There are two advantages of factorial design of the type 2^n experiments.

First, the number of experimental runs is reduced. Second, the computational method for this is easy and simple. The disadvantage of this type of design with two levels is that it takes care of linear order effects only and does not account for the higher order effects. To obliviate this difficulty a sufficiently small interval of variation is often chosen within which the response surface is almost planer in nature.

For a 2^3 design factors are varied between two levels; the higher level being denoted by +1 and the lower level by -1. A particular treatment combination of factors is written using the special Yates notation and is shown in Table 4.1 for a 2^3 type factorial design. The presence of a lowercase letter (representing a factor) indicates the factor is at a higher level while the absence of a letter indicates that the factor is at a lower level. The treatment combination (1) denotes all the factors (x_1, x_2, x_3 etc.) are at the lower level (i.e., at –1). For a 2^3 factorial design (no. of levels P=2, no. of factors n=3), there are 2^3=8 possible combinations of factors and hence the number of experimental runs required is 8.

Treatment combinations	Run No.	Level of Factor		
(Yates std. order)		X_1	X_2	X_3
(1)	1	-1	-1	-1
x_1	2	+1	-1	-1
x_2	3	-1	+1	-1
$x_1\ x_2$*	4	+1	+1	-1
x_3	5	-1	-1	+1
$x_1\ x_3$*	6	+1	-1	+1
$x_2\ x_3$*	7	-1	+1	+1
$x_1\ x_2 x_3{}^+$	8	+1	+1	+1

* First order interaction, $^+$ Second order interaction

+1 indicates higher level, -1 indicates lower level

Table 4.1 Three factor 2^3 Factorial Design

The main effect of a factor is the change in response produced by a change in the level of the factor. So, when a factor is examined at two levels only, the effect is simply the difference between the average response of all trials carried out at the higher level of the factor and that of all trials at the lower level. This average effect of the factor has to be an average overall level of other factors.

The main of a factor is given by the following expression:

$$\text{Main effect of } x_i = \frac{\sum \text{responses at high } x_i \; - \; \sum \text{ responses at low } x_i}{\text{half the no. of experimental runs}}$$

If the effect of one factor is different at different levels of another factor the two factors are said to interact. The interaction effect is the difference between the effects of changing a factor from its lower level to higher level in one case with the other factors at lower level and in another case with the other factors at higher level.

When an interaction is large the corresponding main effects cease to have much meaning. The existence of a large interaction means that the effect of one factor is markedly dependent on the level of the other. A large interaction coefficient signifies that the levels of the factors are too widely spaced and further experimental work at intermediate levels is necessary.

4.4. ANALYSIS OF VARIANCE (ANOVA)

ANOVA is a basic step in the Design Of Experiment (DOE), which is a powerful statistical tool aimed at statistically quantifying interactions between independent variables through their methodical modifications to determine their impact on the predicted variables.

The ANOVA pre requires the following assumptions:

- the treatment data must be normally distributed,
- the variance must be the same for all treatments,
- all samples are randomly selected
- and all the samples are independent

In the analysis of Variance, the total variance is subdivided into two independent variances: the variance due to the treatment and the variance due to random error. The computation of the ANOVA is done through the Sums of squares of the treatments, the error and their total. Total sum of square is given by:

Total SS = SSk + SSE

SSk measures the variations between factors; it represents the sum of square of the columns that generate the sum of square between treatments. The SSE is the sum of Square for errors measures the within- treatment variations.

The one-way ANOVA table is shown in table 4.2

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F-Statistic
Between Treatments	SS k	k-1	MS *k* = SS *k* /(k-1)	F = MS *k* /MSE
Error	SSE	N-k	MSE = SSE/(N-k)	
Total	TSS	N-1		

Table 4.2. The one-way ANOVA table

SS k = sum of squares between treatments

SSE = sum of squares due to error
SSE = TSS - SST
TSS = total sum of squares

MS k = mean square for treatments
MSE = mean square for error
t = number of treatment levels
n = number of runs at a particular level
N = total number of runs
F = the calculated *F* statistic with t -1 and *N* -t degrees of freedom

CHAPTER 5

EXPERIMENTAL DETAILS

5.1. CUTTING TOOLS

5.1.1. High speed steel

High speed steels owe their name to the fact that they were originally developed for high speed metal cutting. The properties of high resistance to wear and heat high initial hardness of about 60 to 65 RC at service temperature of 600 to 650 ºC and the economical price of HSS have made them a logical choice of many cutting industries. This finds applications as turning tools, twist drills, counter bores, taps and dies, reamers, broaches, milling cutters, hobs, saws, etc.The perfect combination of alloying elements and the domain of heat treatment processes confers excellent hardness and wear resistance properties allied to good toughness[35]

The HSS tool samples considered in this work are M2 and S400 steels procured from Miranda (ISO – 9002 company) with dimensions 12.70 x 152.40 mm. Some of the HSS tool bit blanks were made into single point cutting tools for turning with standard tool signature as given in table 5.1 while other tool blanks were used for micro structure analysis and sliding wear tests.

Back rake angle	0º
Side Clearance Angle	10º
Side Rake Angle	10º
Principal Cutting edge Angle	90º

Table 5.1.Description of single point HSS tools

5.1.2. Tungsten carbide

Tungsten carbide has been proved to be much more efficient than HSS when machining hard materials such as steel itself. Due to its extreme hardness, tungsten carbide is largely used in the manufacture of cutting tools as cheaper and more heat resistant alternative to diamond. This is useful when machining tough materials and may leave better surface finish on the parts.

The cutting tools used were squares inserts with cheap breakers of two different grades: SNMG120412MP (Kennametal) and SNMS12048 (Kennametal). The inserts were clamped onto a tool holder with a designation of PSBNR2020K12D5L (WIDAX).

5.2. LABORATORY TESTS

5.2.1. Flank wear tests

Tool wear is almost always used as a tool life criterion because it is easy to determine quantitatively. Various types of tool wear are shown in fig. 5.1

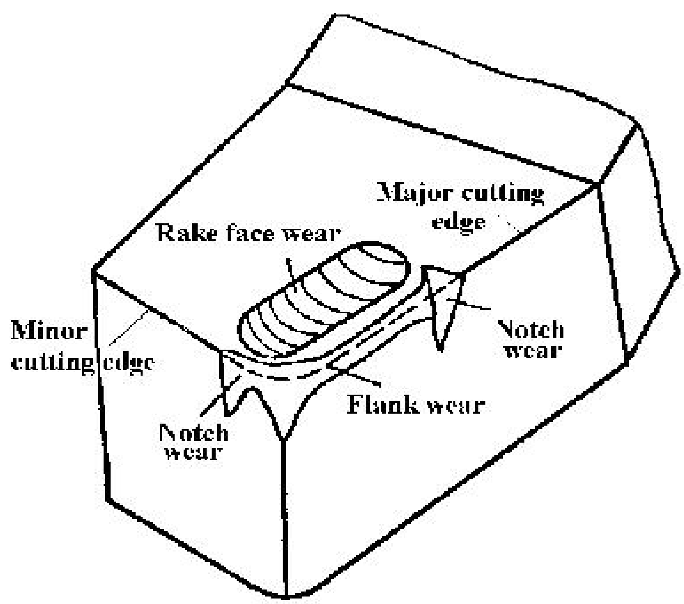


Fig. 5.1 Tool wear phenomena

The amount of flank wear is often used as a criterion because it is the flank wear that influences work material surface roughness and accuracy. A standard tool life is the time to develop a flank wear land of recommended size based on the material and operation as depicted in table 5.2.

Wear (in)	Tool Material	Remarks
0.030 (0.76 mm)	Carbide	Roughing passes
0.010-0.015 (0.25-0.38 mm)	Carbide	Finishing passes
0.060 or total destruction(1.25 mm)	H.S.S.	Roughing passes
0.010-0.015 (0.25-0.38 mm)	H.S.S.	Finishing passes
0.010-0.015 (0.25-0.38 mm)	Cemented oxides	Roughing and finishing passes

Table 5.2. Recommended size of flank wear

Fig. 5.2 shows the typical stages of tool wear as well as illustrates the method to evaluate tool life from flank wear graphically.

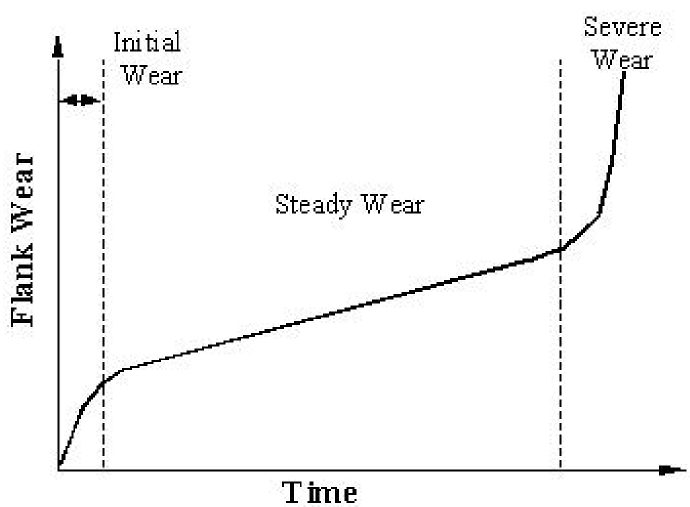


Fig. 5.2. Typical stages of tool wear in normal cutting situation

In the present work, the tool samples were subjected to turning operation in a high speed lathe (HMT NL26) with a maximum spindle speed of 1200 RPM. As soon as lathe was started, stop watch was switched on to note down the machining time. At the end of each run, flank wear was measured in a tool maker’s microscope. The flank wear was normally observed at every 2 minutes interval. The total machining time before reaching a minimum of 0.3 mm flank wear was considered to be the tool life of the sample.

5.2.2. Sliding wear tests

The materials considered for this were the cryogenically treated as well as untreated S400 and M2 grade HSS samples with dimensions 20 x 16 x 16 mm. The test was conducted on a machine called **disc and pinion** (make: SD scientific industries) as shown in **fig.5.3**. The sample was mounted perpendicularly on a stationary vice such that its one of the face is forced to press against the abrasive that is fixed on the revolving disc. Hence it is the abrasive paper that tends to wear the surface of the samples. When the disc rotates for a particular period of time the sample can be loaded at the top to press against the disc with the help of a lever mechanism.

The speed of revolution can also be varied and thus the test can be conducted with the following parameters-

(1) Load (2) Speed (3) Time

In the present experimental work, speed and time wear kept constant while the load was varied from 0 to 1.2 kg. Parameters that remained constant through out all the experiments are given in table (3).

RPM	300
Time	1hr
Type of abrasive paper	Emery

Table 5.3. Parameters taken constant in sliding wear test

For each of the sample, test was conducted for 5 times and the average of all the samples was taken as the observed values in each case.

Once the parameter is set and work piece is mounted, the test is carried on for the desired time. The wear track so formed on the rotating disc is a circle. After each test only the mass loss of the specimen was considered as the wear.

Fig.5.3. Disc and pinion apparatus

The wear rate of each sample was calculated from the weight loss, the amount of wear is determined by weighing the specimen before and after the test using precession electronic weighing machine with accuracy 0.0001 gm. Since the mass loss is measured it is converted to volume loss using the density of the specimen. Hence wear rate and wear resistance can be calculated from equation 5.1 and equation 5.2 respectively.

$$\mathrm{w} = \frac{V_w}{D} \qquad \text{... (5.1)}$$

$$W = \frac{1}{w} \qquad \text{... (5.2)}$$

Where w= Wear rate

W=Wear resistance

Vw = Wear volume

D = Distance traveled

A comparison has been made to identify effects of cryogenic treatment on wear improvement on S400 and M2 grade HSS samples. The test was conducted for 5 times for each of the samples.

5.2.3. Hardness test

Rockwell hardness testing (the **apparatus** being shown in **fig. 5.4**) is a general method for measuring the bulk hardness of metallic and polymer materials. Although hardness testing does not give a direct measurement of any performance properties, hardness correlates with strength, wear resistance, and other properties. Hardness testing is widely used for material evaluation due to its simplicity and low cost relative to direct measurement of many properties. This method consists of indenting the test material with a diamond cone or hardened steel ball indenter. The indenter is forced into the test material under a preliminary minor load *F0* (Fig. 5.5 A) usually 10 kgf. When equilibrium has been reached, an indicating device, which follows the movements of the indenter and so responds to changes in depth of penetration of the indenter, is set to a datum position. While the preliminary minor load is still applied an additional major load is applied with resulting increase in penetration (Fig. 5.5 B). When equilibrium has again been reach, the

Fig. 5.4. Rockwell hardness measuring machine

additional major load is removed but the preliminary minor load is still maintained. Removal of the additional major load allows a partial recovery, so reducing the depth of penetration (Fig. 5.5 C). The permanent increase in depth of penetration, resulting from the application and removal of the additional major load is used to calculate the Rockwell hardness number.

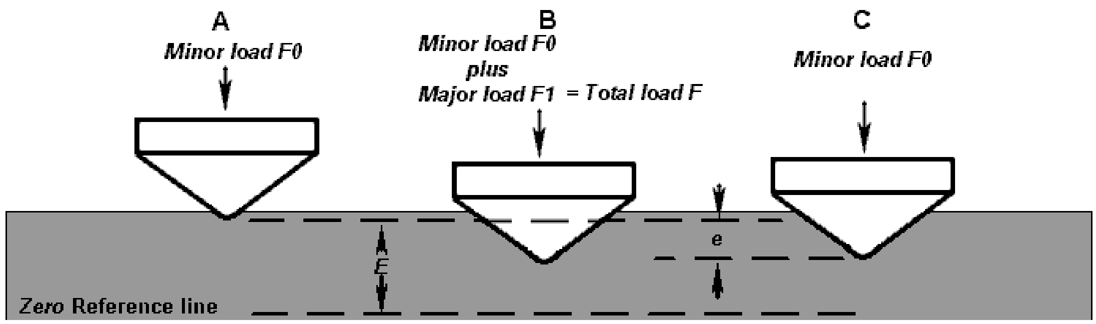


Fig. 5.5.Rockwell Principle

In the present experimental work Rockwell Hardness was measured on cryogenically treated and untreated S400 and M2 grade HSS samples with a minimum of four indentations in each. The average of these measurements was considered for comparison.

5.2.4. Metallographic examination

This part of the work had the objective of analyzing the changes that occurred in the micro structure of the S400 and M2 high speed steel after the cryogenic treatment. Metallographic study basically includes the following:

Optical micro- (micro structure) examination

This is defined as the method of studying microstructure constituents (grains, phases, micro pores, etc) by means of a metallurgical microscope. In order to carry the analysis first the samples were polished using emery paper of four different grits. This was followed by mirror finishing by polishing the samples on velvet cloth which is mounted on a rotating disc. After this these samples were etched with 2% nital and dried in air. Microstructure examination was carried out using an optical microscope

Scanning Electron Microscope (SEM) examination

Scanning electron microscope creates images by using electrons instead of light waves while conventional microscopes use a series of lenses to bend light waves and create magnified image. The SEM shows very detailed three dimensional images at much higher magnification. The images obtained from this are black and white only as this does not work on the principles of light waves.

X-Ray Diffraction (XRD) analysis

It is a versatile non destructive technique that reveals detailed information about the chemical composition and crystallographic structures of natural and manufactured materials.

5.3. IMPLEMENTATION OF DOE FOR WEAR BEHAVIOR AND TOOL LIFE PREDICTION

The goal of this experimental work was to investigate the effects of cutting parameters on tool wear and to establish a correlation between them. In order for this cutting velocity, depth of cut and feed rate were chosen as process parameters. The work material was mild steel. The turning tests were conducted on HMT lathe having maximum spindle speed of 1020 RPM.The cutting tools used were:

(1) cryogenically treated S400 HSS single point cutting tools

(2) cryogenically treated carbide inserts

For each of the tools a 2^3 Factorial design was selected. This indicates two levels were specified for each of the three parameters. The parameter levels were chosen within the intervals recommended by the cutting tool manufacturer. Three process parameter at two levels lead to a total of 8 tests. Flank wear was measured at regular intervals of two minutes with the help of tool maker's microscope.

Significant factors affecting the tool wear were found using DOE and tool life equations were developed illustrating the significant factors.

CHAPTER 6

RESULTS AND DISCUSSION

6.1. LABORATORY TESTS

6.1.1 Flank wear tests

Single point M2 grade HSS tools as well as carbide inserts were subjected to turning operation in HMT NL26 lathe according to the machining specifications given in table 6.1.

	M2 HSS	Carbide
Cutting velocity (m/min)	46.7	67.8
Depth of cut (mm)	0.5	0.5
Feed (mm/rev)	0.05	0.05
Cutting condition	Dry	Dry
Work piece material	Mild steel	Mild steel

Table 6.1. Machining specifications for turning HSS tools and carbide inserts

Results of Flank wear test for both cryogenically treated and untreated samples are shown in table 6.2 and table table 6.3 respectively.

Sl. No.	Time (min)	Flank wear (mm)
1	10	0.245
2	20	0.270
3	30	0.290
4	40	0.320
5	50	0.375
6	60	0.405

Table6.2. Results of flank wear test for untreated HSS tools

Sl. No.	Time (min)	Flank wear (mm)
1	10	0.140
2	20	0.165
3	30	0.180
4	40	0.245
5	50	0.275
6	60	0.380

Table 6.3. Results of flank wear test for treated HSS tools

Fig 6.1 illustrates the graph that are plotted to evaluate the tool life in each of the cases so as to make necessary comparison.

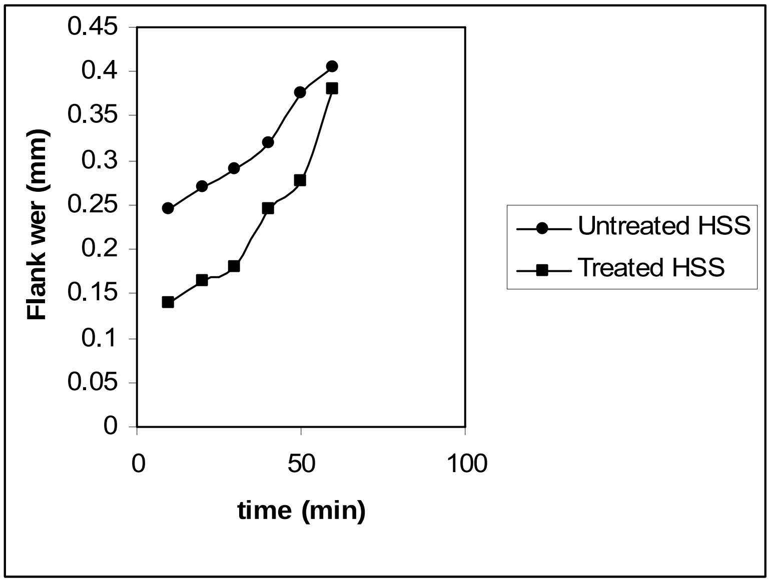


Fig.6.1.Flank wear development in HSS tools

From the graph it was observed that cryogenically treated HSS tools showed slightly higher value of tool life.

Carbide inserts were tested by performing orthogonal turning on mild steels on HMT lathe. Machining specifications as well as the results of the turning tests for both cryogenically treated and untreated tool inserts are given in table 6.4 and table 6.5 respectively.

Sl. No.	Time (min)	Flank wear (mm)
1	10	0.066
2	20	0.076
3	30	0.078
4	40	0.080
5	50	0.125
6	60	0.280

Table 6.4 Results of flank wear tests for untreated carbides

Sl. No.	Time (min)	Flank wear (mm)
1	10	0.060
2	20	0.070
3	30	0.072
4	40	0.074
5	50	0.105
6	60	0.184

Table 6.5 Results of flank wear tests for treated carbides

Graphs as shown in fig 6.2 were plotted to compare the tool lives in both the cases.

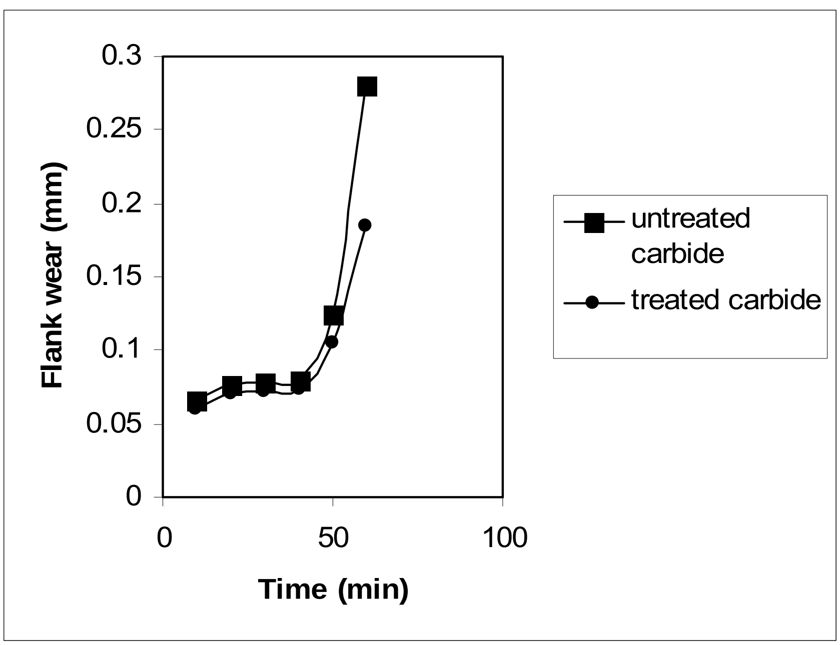


Fig. 6.2. Flank wear development in carbide inserts

Cryogenically treated carbide inserts presented longer tool lives as compared to the untreated.

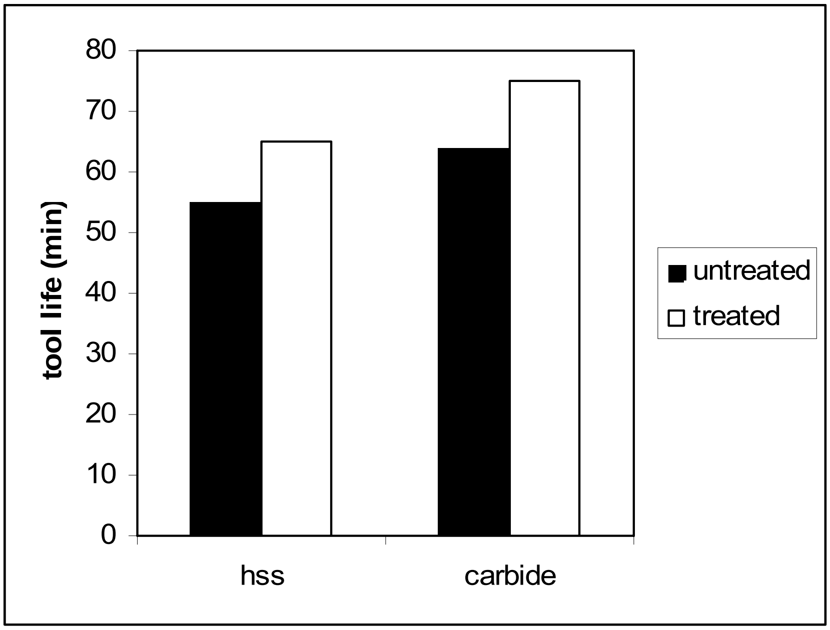


Fig. 6.3. Tool life comparisons between treated and untreated samples

There is an increase of tool life by 19.2% and 17.18% for cryogenically treated HSS and carbide inserts respectively in comparison to the untreated tools. Hence it is evident that there is increase in tool life both fore cryogenically treated HSS tool and carbide insert.

The superior performance of cryogenically treated HSS can be attributed to the transformation of almost all retained austenite into martensite, a harder structure and precipitation of fine and hard carbides[36],[1]. The results for cryogenically treated carbide inserts seem to be in accordance with the results obtained by Seah et. al[36]. According to him increase in number of η phase particles are most likely responsible for these positive results. However the performance of cryogenically treated tools depends on cutting conditions and cutting environment.

The experiments were also conducted to determine tool life for machining mild steel using cryogenically treated HSS tools and carbide inserts using design of experiments for two levels. The details results have been discussed later. The experiments were also conducted to determine tool wear in cryogenically carbide inserts for machining aluminium. It is observed that no flank wear developed in carbide inserts for a time period of machining.

6.1.2. Sliding wear test

S400 and M2 grade HSS samples were subjected to sliding wear test to evaluate the effect of cryogenic treatment on the wear resistance.

First the tests were conducted on S400 samples by varying the load from 0 to 1.2 kg, other parameters being constant. The main aim of the test was to study the effect of load on the wear resistance of the HSS samples as well as to find a suitable value of load based on which the further comparative tests could be conducted more quantitatively. Table 6.6 shows the results of this.

Sl. No.	Load (N)	Wt. Loss for untreated HSS (g)	Wt. Loss for treated HSS (g)
1	0	0	0
2	2.11	0	0
3	7.526	0.0006	0.002
4	11.76	0.0024	0.00046

Table 6.6.Effect of load on wear rate in terms of weight loss

The results indicate that with the increase of force wear rate becomes more prominent. Hence the load weighing 1.2 kg (11.76 N) was decided to be set for all further tests. Moreover it was observed that the cryogenically treated samples are showing higher value of weight loss. So in order to confirm this, the test was repeated for minimum of 5 times for both cryogenically treated and untreated S400 and M2steels by keeping all the three parameters (speed, time, load) constant through out the experiment. The average values of wear rate and wear resistance are shown in table 6.7 and table 6.8 (the calculation being explained in the previous chapter).

Sl. No.	Specimen type	Wt. loss (g)	Wear rate (cm^2)	Wear resistance (cm^{-2})
1	Untreated	0.0012	1.448×10^{-10}	0.6904×10^{-10}
2	Treated	0.0039	4.707×10^{-10}	0.2124×10^{-10}

Table 6.7. Results of sliding wear tests for S400 HSS steel

Sl. No.	Specimen type	Wt. loss (g)	Wear rate (cm^2)	Wear resistance (cm^{-2})
1	Untreated	0.0023	2.776×10^{-10}	0.3602×10^{-10}
2	Treated	0.0033	3.983×10^{-10}	0.2510×10^{-10}

Table 6.8. Results of sliding wear tests for M2 steel

Results showed untreated samples for both S400 and M2 grade superior performance over the treated ones. In case of S400 steel wear resistance decreased almost 3 times after cryogenic treatment while in case of M2 steel wear resistance decreases by 1.4 times. Fig. 6.4 illustrates the comparison wear resistances between cryogenically treated and untreated samples.

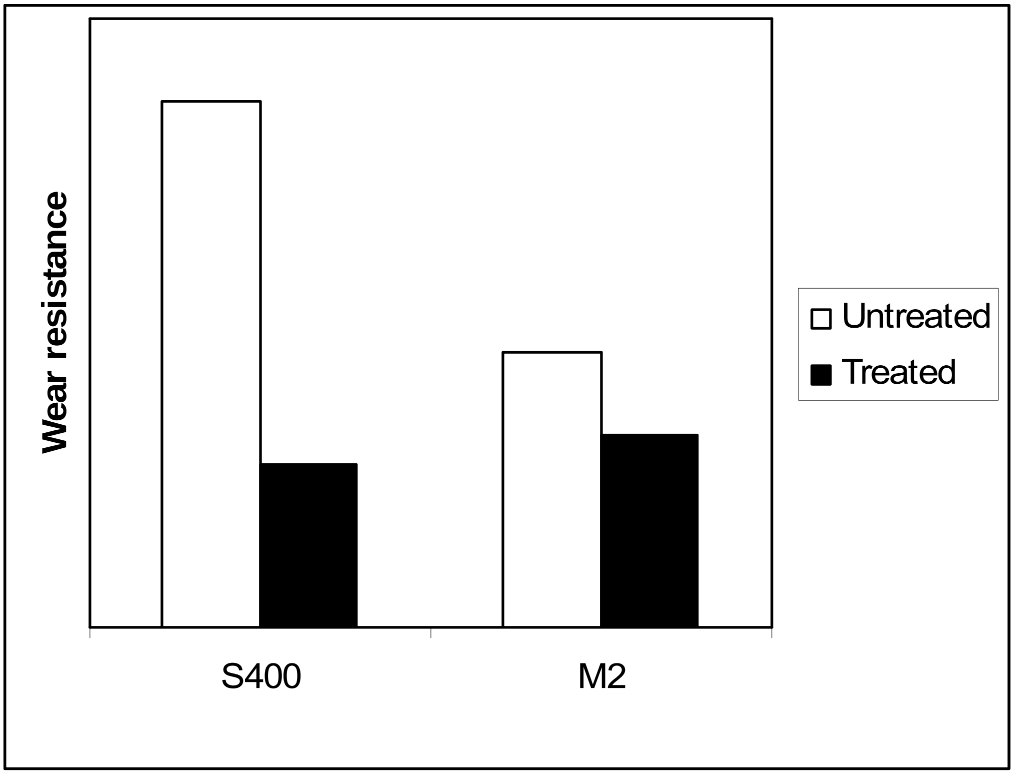


Fig. 6.4. Comparison of wear resistance between treated and untreated HSS

The transformation of retained austenite into martensite after cryogenic treatment did not lead to a significant alteration of the abrasive wear rate at the conditions used in the disc and pinion tests. Literature results[36] showed that depending on the parameters such a s normal load, average grain size and type of the abrasive, quantity and shape of the carbides among others, the increasing of the amount of retained austenite can lead to an increase or decrease in the wear rate of ferro alloys.

In the present work increase in wear rate for cryogenically treated HSS samples can be attributed to the fact that the tool becomes more brittle after the treatment. The wear resistance can be increased by incorporating tempering or plasma nitriding with the cryogenic treatment.

6.1.3. Hardness test

Table 6.9 and table 6.10 shows the hardness of both cryogenically treated and untreated S400 and M2 grade HSS samples. They are practically the same thus indicating that the cryogenic treatments had no influence on this property of this tools.

Specimen type (S400)	Hardness (HRc)
Untreated	66
Treated	66

Table 6.9. Results of hardness test for S400 HSS

Specimen type (M2)	Hardness (HRc)
Untreated	68
Treated	68

Table 6.10. Results of hardness tests for M2 HSS

The results obtained in the present study are in accordance with the results obtained by Barron and Flavio [36]. Even the microhardness results also did not show conspicuous difference between the treated and untreated tools [10]. The precipitation of fine carbides during the cryogenic treatment cycle may affect the wear resistance and the tool toughness but only a small, if any in tool hardness [2]. It was observed that initially the hardness falls sharply at the cryogenic cycle and when the tool is heated to the room temperature the hardness is totally recovered.

6.1.3. Metallographic examination

Characterization by optical microscope

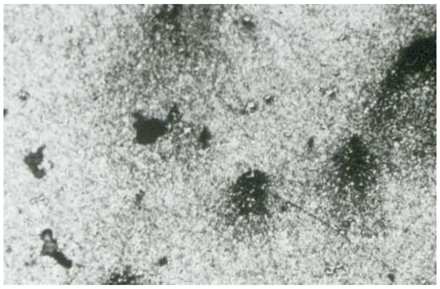
Fig 6.5 shows the microstructures of S400 and M2 grade HSS samples. Not much could be inferred from this as no significant changes in microstructure after the cryogenic treatment observed literature data indicates transformation of retained austenite into martensite as well a carbide refinement [36][3]. But it was very difficult to detect such changes with the help of an optical microscope.

X-Ray diffraction (XRD) analysis

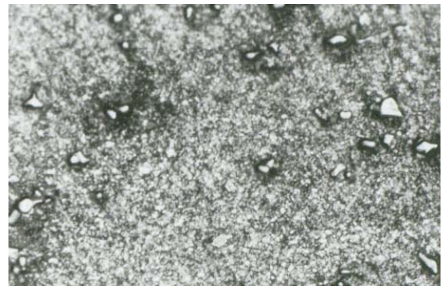
XRD analysis was carried out for both cryogenically treated and untreated HSS tools using X-ray generator (make: Philips). Fig6.6 shows results from XRD investigations on untreated and cryogenically treated HSS samples respectively.

SEM analysis

SEM was carried for both cryogenically treated and untreated HSS samples to study the microstructural changes. Results of the SEM analysis are shown in fig. 6.7 and fig. 6.8 for cryogenically treated and untreated HSS samples respectively. The results showed the presence of the fine precipitated carbide particles in case of cryogenically treated samples which verify that the refinement of carbides takes place after the cryogenic treatment.

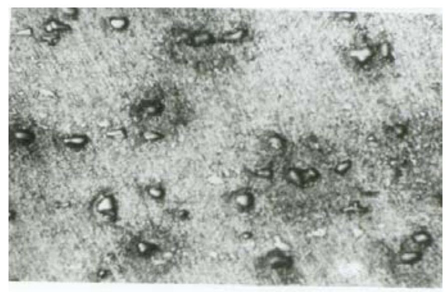


(a) x200 magnification (untreated S400)

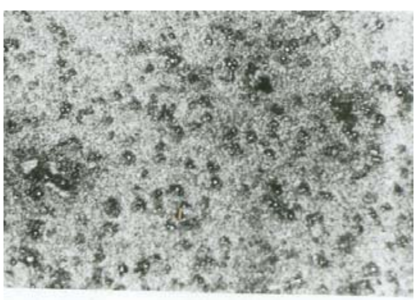


(b)x400 magnification(untreatedS400)

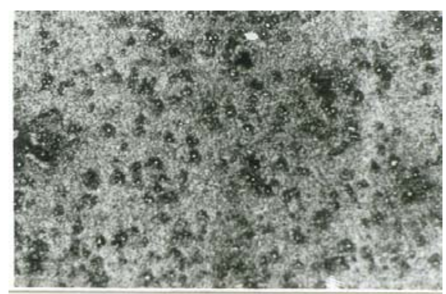
(c) x200 magnification (untreated M2)



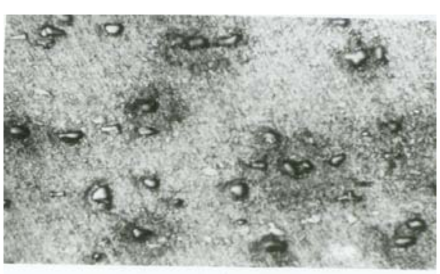
(d) x400 magnification (untreated M2)



(e) x200 magnification (treated S400)



(f) x400 magnification (treated S400)



(g) x200 magnification (treated M2)

(h)x400 magnification (treated M2)

Fig. 6.5. Microstructure of cryogenically treated and untreated S400 M2 HSS samples at x200 and x 400 magnification

Fig. 6.6. (a) XRD analysis for untreated HSS samples

Fig. 6.6. (b) XRD analysis for treated HSS samples

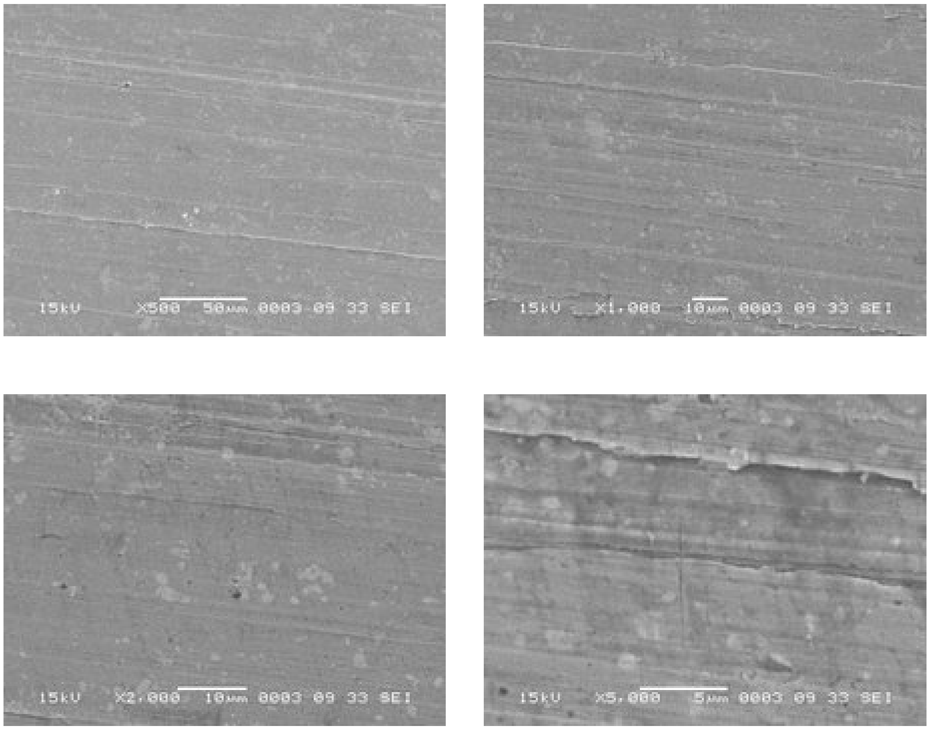


Fig. 6.7. Results of SEM for cryogenically treated HSS samples

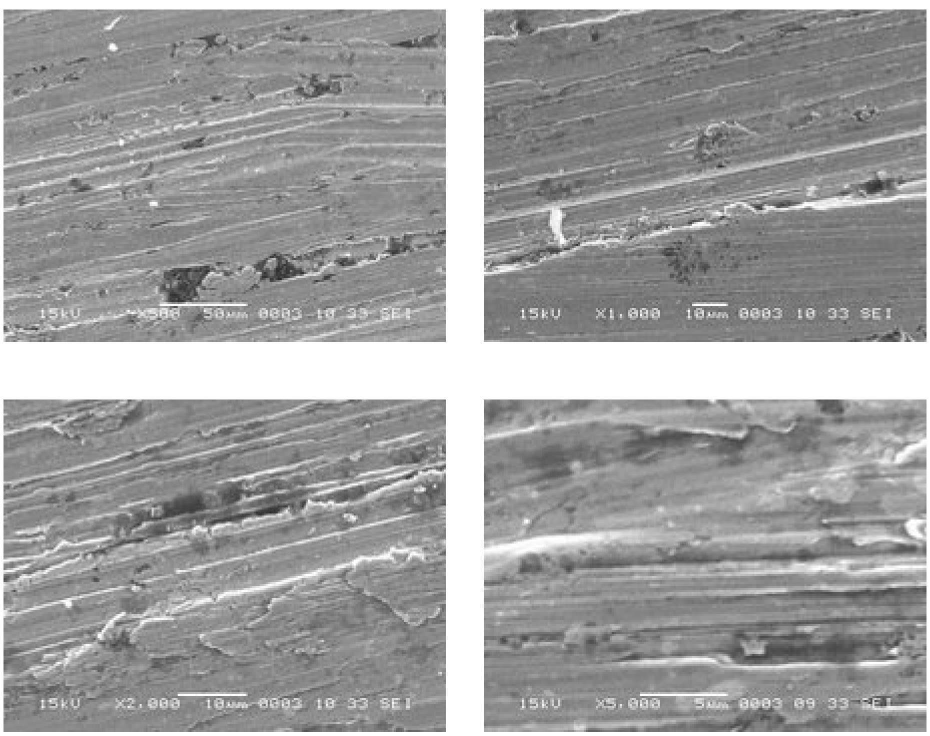


Fig. 6.8. Results of SEM analysis for untreated HSS samples

6.2. IMPLEMENTATION OF DOE FOR WEAR BEHAVIOR AND TOOL LIFE PREDICTION

6.2.1. Tool life equation for cryogenically treated single point HSS tools

Table 6.11 shows the process parameters (factors) that were chosen for machining mild steel using cryogenically treated HSS single point cutting tools. Two levels were specified for each parameters. Turning tests were conducted on lathe and flank wear was measured with the help of a tool makers microscope. Tool life was evaluated by calculating the time to reach a flank wear of 0.3mm.

Symbol	Factors	Factor Levels	
		-1	+1
A	Cutting Velocity (m/min)	30	60
B	Depth of cut (mm)	0.5	1
C	Feed (mm/rev)	0.05	0.1

Table 6.11. Cutting parameters for 2^3 factorial design

Table 6.12 illustrates the experimental results for tool life.

Run	Level of Factors							Tool Life (min)
	A	B	C	AB	BC	AC	ABC	
1	-1	-1	-1	+1	+1	+1	-1	17.8
2	+1	-1	-1	-1	+1	-1	+1	18.3
3	-1	+1	-1	-1	-1	+1	+1	20
4	-1	+1	-1	+1	-1	-1	-1	16.8
5	+1	-1	+1	+1	-1	-1	+1	17.0
6	-1	-1	+1	-1	-1	+1	-1	15.4
7	+1	+1	+1	-1	+1	-1	-1	16.0
8	-1	+1	+1	+1	+1	+1	+1	12.0

Table 6.12. Results for tool life for cryogenically treated HSS tools

Table 6.13 depicts the factor effect summary. It was observed that the only significant factor for the tool life is feed which explains 50% of total variation. The second largest contribution comes from cutting velocity with 22% of total variation. The depth of cut alone has almost no statistical signification. However it has some contribution in combination with speed and feed with 11.92% and 8.33% of total variation respectively.

Factor	Effect Estimate	Sums of Squares(SS)	%Contribution
A	-2.075	8.61125	22.0695
B	-0.925	1.71125	4.385
C	-3.125	19.53125	**50.056**
AB	-1.525	4.65125	11.92
BC	-1.275	3.25125	8.332
AC	-0.725	1.05125	2.694
ABC	0.325	0.21125	0.541

Table 6.13. Factor effect summary for cryogenically treated HSS

The experimental results were then analyzed with ANOVA, as shown in table 6.14.

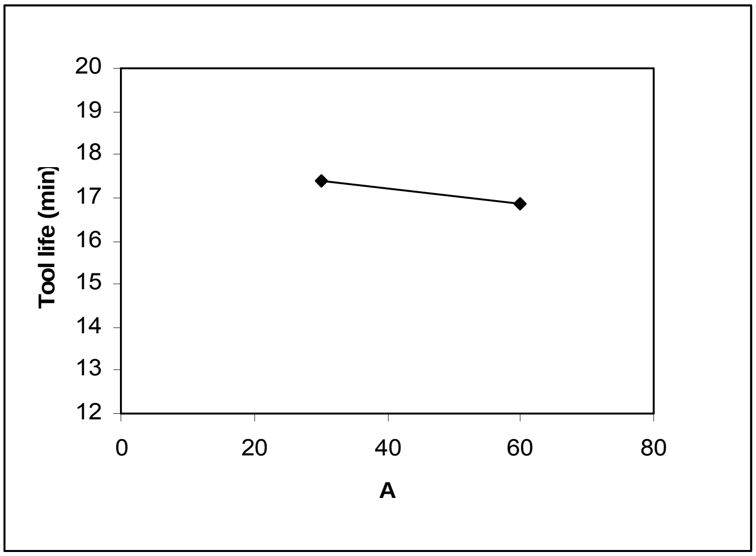
Source of Variation	Sums of Square (SS)	DOF	Mean Square (MS=SS/DOF)	Fo
A	8.61125	1	8.61125	19,68,285.714
B	1.71125	1	1.71125	3,91,142.8571
C	19.53125	1	19.53125	44,64,285.714
AB	4.65125	1	4.65125	10,63,142.857
BC	3.25125	1	3.25125	7,43,285.8571
AC	1.05125	1	1.05125	2,40,285.7143
ABC	0.21125	1	0.21125	48,285.714
Error	0.000035	8	0.00000437	
Total	39.01875	15		

Table 6.14. ANOVA results for cryogenically treated HSS tools

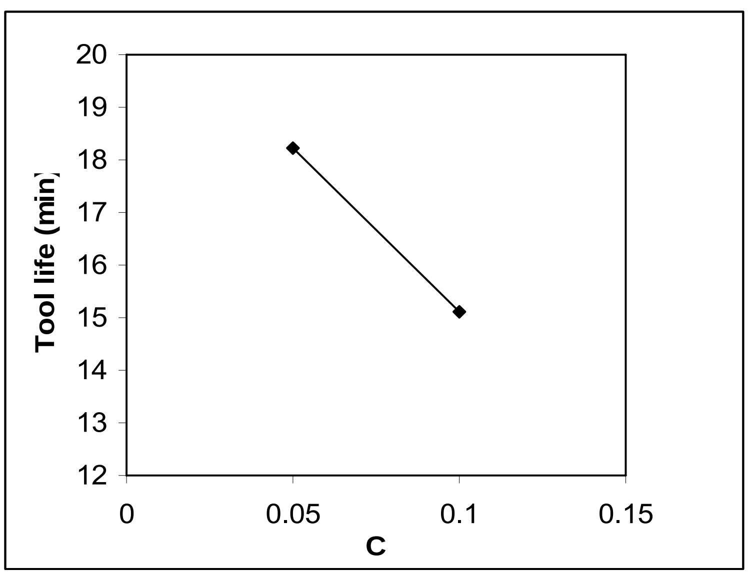
F-statistic is calculated which came out to be 12, 74, 080.49. Since its value is large, it is stated that at least one variable has non zero effect. Each of the factor is tested for significance using the F-statistic. From this it was confirmed that feed (C) and cutting velocity (A) are the most significant factors, while the depth of cut does not have any impact on tool life. Most significant interaction effects were found between cutting velocity and depth of cut as well as feed and depth of cut.

The main effects of A and C are plotted in fig 6.9 (a) and fig.6.9 (b). Both the effects are negative and if only these effects are considered, the two factors i.e. cutting velocity and feed would be run at the low level to obtain higher tool life. However, it is always necessary to examine any interaction effects that are important because the main effects do not have much meaning when they are involved in significant interactions.

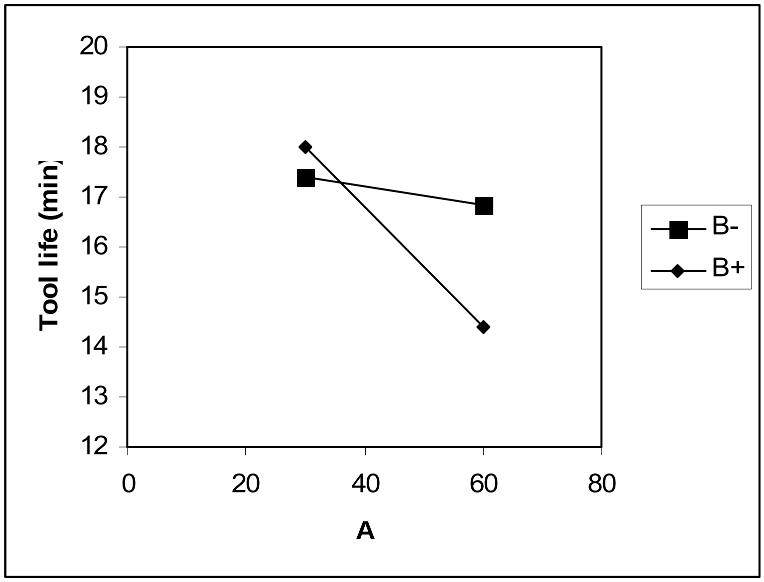
The AB and BC interactions are plotted in fig.6.9(c) and fig. 6.9(d). From the AB interaction it was noted that velocity effect is very small when depth of cut is low and very large when depth of cut is high with the best results obtained with low velocity and high depth of cut. From the BC interaction it was observed that depth of effect is comparatively more when feed is high showing higher tool life in case of low feed and high depth of cut. In both the cases of interaction, higher depth of cut was found to be favourable.



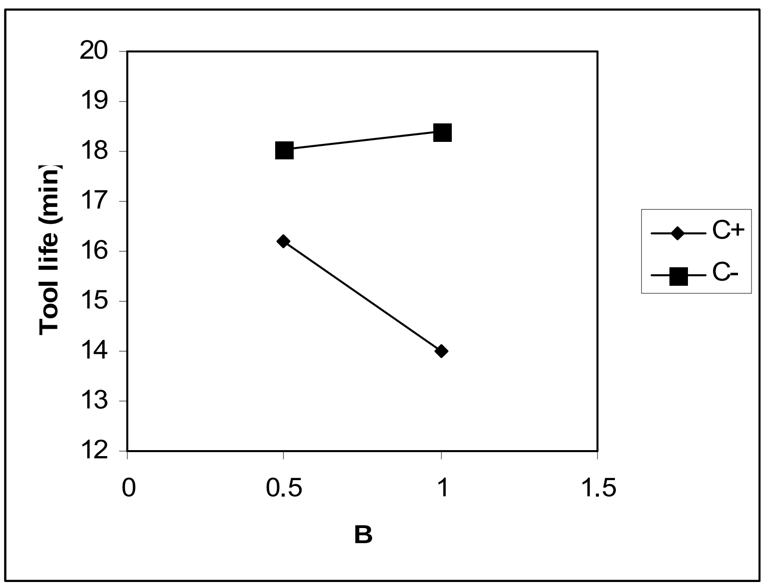
(a)Main effect plot for cutting velocity (A)



(b) Main effect plot for feed rate (C)



(c)Interaction effect plot for cutting velocity (A) and depth of cut (B)



(d) Interaction effect plot for depth of cut (B) and feed rate (C)

Fig. 6.9. Main effect and interaction effect plots for HSS samples

The tool life equation was developed for cryogenically treated HSS tools comprising of the significant factors with effect estimate values in the brackets which is as shown:

$$y = 16.6625 + \left(\frac{-2.075}{2}\right)X_1 + \left(\frac{-3.125}{2}\right)X_3 + \left(\frac{-1.525}{2}\right)X_1X_2 + \left(\frac{-1.275}{2}\right)X_2X_3$$

R^2=0.923

R^2 values for the equation are high enough to obtain the reliable estimates.

For further check, the equation so developed is applied to all the 8 tests to calculate the average percentage error. Residuals being the difference between the observed and predicted value were also calculated as shown in table 6.15

	Predicted value(y)	Measured value(y')	Residual (e=y-y')
(1)	17.8	17.82625	-0.02625
a	18.3	17.27625	1.02375
b	20	20.62625	-0.62625
ab	16.8	17.02625	-0.22625
c	17.0	15.98755	1.014125
ac	15.4	15.49825	-0.09825
bc	16.0	16.29875	-0.29875
abc	12.0	12.71125	0.71125

Table 6.15. Differences between predicted values and measured values for tool life

It was observed that of residuals in most of the cases differences between predicted values and observed values were found to be less than 1 thus contributing fairly low average percentage of error.

2.2 Tool life equation for cryogenically treated carbide inserts

The cutting parameters (factors) along with their levels are listed in table 6.16. The cutting tool used was cryogenically treated carbide insert with ISO designation of SNMS120408 (Kennametal).The flank wear test was carried out and tool life was calculated for each of the test runs.

Symbol	Factors	Factor Levels	
		-1	+1
A	Cutting Velocity (m/min)	85	135
B	Depth of cut (mm)	0.5	1
C	Feed (mm/rev)	0.05	0.1

Table 6.16. Cutting parameters for 2^3 factorial design

The experimental results for the tool life given in table6.17.

Run	Level of Factors							Tool Life (min)
	A	B	C	AB	BC	AC	ABC	
1	-1	-1	-1	+1	+1	+1	-1	12
2	+1	-1	-1	-1	+1	-1	+1	4
3	-1	+1	-1	-1	-1	+1	+1	10
4	-1	+1	-1	+1	-1	-1	-1	12
5	+1	-1	+1	+1	-1	-1	+1	16
6	-1	-1	+1	-1	-1	+1	-1	4
7	+1	+1	+1	-1	+1	-1	-1	4.5
8	-1	+1	+1	+1	+1	+1	+1	11

Table 6.17. Results for tool life for carbide insert

Table 6.18 and table 6.19 illustrate the effect estimate summary and results of ANOVA respectively.

Factor	Effect Estimate	Sums of Squares	%Contribution
A	-2.875	16.53125	11.644
B	0.375	0.28125	0.198
C	-0.625	0.78125	0.55
AB	7.125	101.53125	**71.51**
BC	-2.625	13.78125	9.707
AC	0.125	0.03125	0.02
ABC	2.125	9.03125	6.361

Table 6.18. Effect estimate summary for tool life of carbides

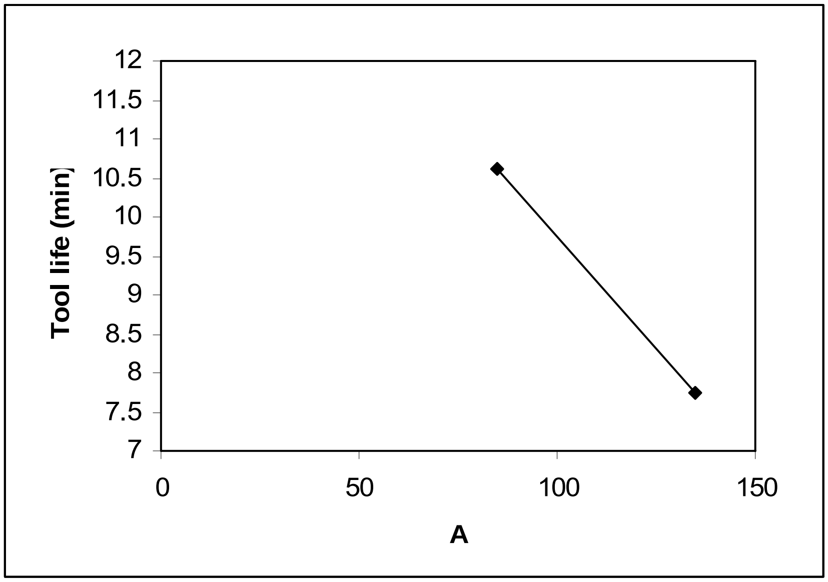
From the table it is inferred that the cutting velocity has some significant effect on the tool life, with 11.647% of total variation in comparison to that of depth of cut and feed rate which are considered to be statistically insignificant. The interactions among cutting velocity and depth pf cutting seem to dominate all the main effects greatly with 71.51% of total variation. The interaction between depth of cut and feed rate also affects the tool life though with a lower level of contribution (9.75% of total variation) as compared to the main effects of A and interacting effects of AB.

Source of Variation	Sums of Square (SS)	DOF	Mean Square (MS)	Fo
A	16.53125	1	16.53125	26,45,000
B	0.28125	1	0.28125	45,000
C	0.78125	1	0.78125	1,25,000
AB	101.53125	1	101.53125	1,62,45,000
BC	13.78125	1	13.78125	22,05,000
AC	0.03125	1	0.03125	5,000
ABC	9.03125	1	9.03125	14,45,000
Error	0.00005	8	0.00000625	
Total	141.96875	15		

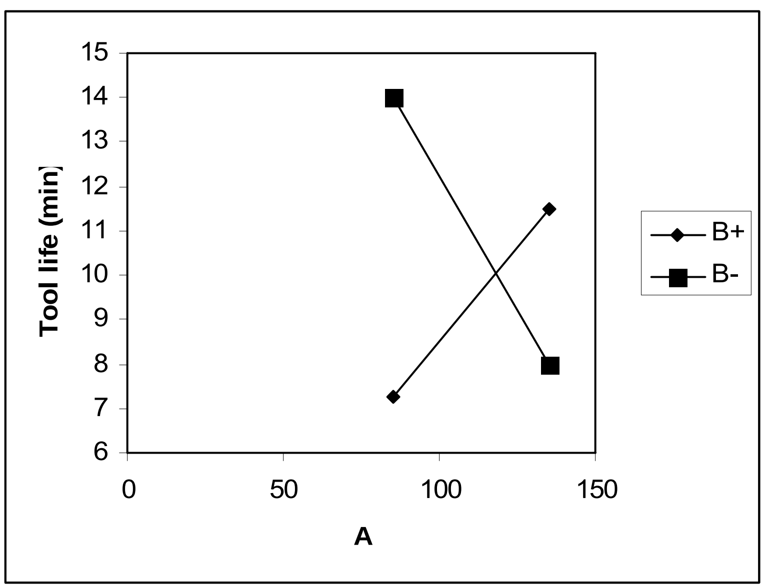
Table 6.19. Results of ANOVA for tool life of cryogenically treated carbides

F statistic calculated came out to 32, 44,999 which is quite a large value thus indicating the fact that atleast one variable has a non- zero effect. Each of the factorial effects were tested individually using the F statistic to identify the factors significantly affecting performance measures. The factors that have Fo values greater or nearly closer to the value of F statistic are considered to be statistically significant.

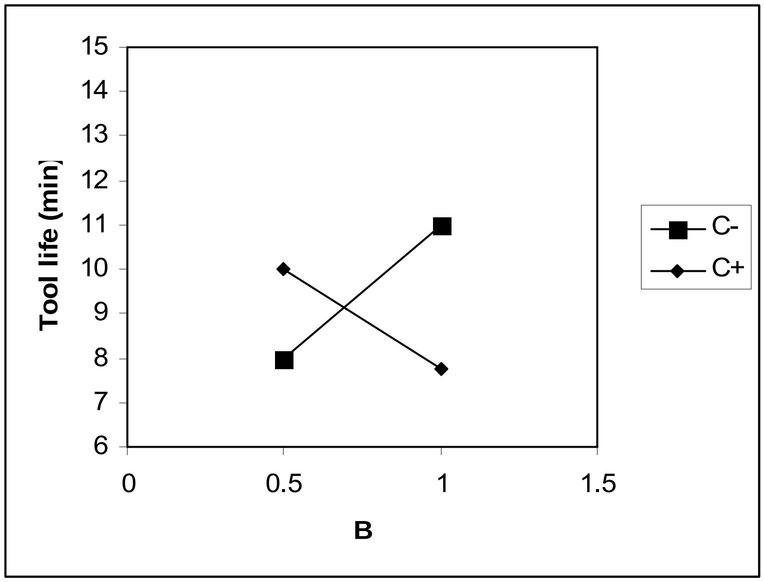
Effect plots can be used to help understand the nature of main effects and interaction effects as shown in fig.6.10.The main effects of A are plotted in fig. 6.10(a).Tool life appears to be almost linear decreasing function of cutting velocity. The decrease in tool life may be attributed to significant increased heat involved in the cutting process leading to tool wear. Hence if only the main effects are considered, cutting velocity when decreased from 135m/min to 85 m/min would give a higher tool life.AB interaction is plotted in fig 6.8(b). It is noticed that velocity effect is significantly high at low depth of cut and depth of cut effect is very high at low cutting velocity with the best results obtained at low velocity and low depth of cut. From BC interaction as shown in fig. 6.10 (c)it is inferred that interaction effect is more prominent for higher value of depth of cut. When the feed is decreased from 0.1mm/ rev to 0.5 mm / rev at high depth of cut, favourable results for tool life are obtained.



(a) Main effect plot for cutting velocity (A)



(b) Interaction effect plot for cutting velocity (A) and depth of cut (B)



(c) Interaction effect plot for depth of cut (B) and feed rate (C)

Fig.6.10. Main effect and interaction plots for tool life of carbide inserts

The tool life equation showing the significant factors along with the effect estimates is given as follows:

$$y = 9.1875 + \left(\frac{-2.875}{2}\right)X_1 + \left(\frac{7.125}{2}\right)X_1X_2 + \left(\frac{-2.625}{2}\right)X_2X_3$$

The R^2 values for the developed model were calculated which are given as follows:

R^2=0.9286

R^2adj=0.9988

Residuals were calculated by applying the regression equation developed to the 8 test points as listed in table 6.20

	Predicted value (y)	Measured value (y)'	Residuals (e=y-y')
(1)	12	12.875	-0.875
a	4	2.875	1.125
b	10	8.3375	1.625
ab	12	12.625	-0.625
c	16	15.5	0.5
ac	4	5.5	-1.5
bc	4.5	5.75	-1.25
abc	11	10	1

Table 6.20. Difference between predicted value and measured value of tool life

It is observed that there is much difference between the predicted values and the measured values which indicates that the tool life equation developed for carbide inserts for machining mild steel can give rough estimates.

2.3.Flank wear prediction model for carbide inserts

The flank wear behaviour of cryogenically treated carbide with ISO designation SNMG120412MP (Kennametal) was studied while machining mild steel considering the cutting conditions as shown in table 6.21

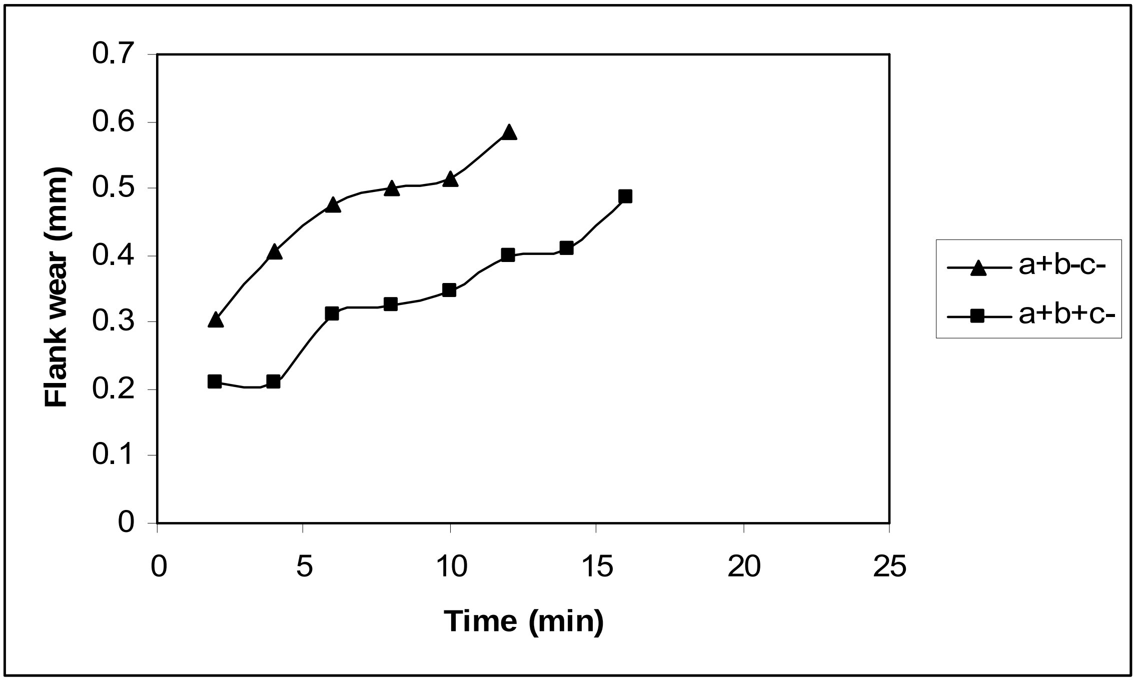
Symbol	Factors	Factor Levels	
		-1	+1
A	Cutting Velocity (m/min)	45	85
B	Depth of cut (mm)	0.5	1
C	Feed (mm/rev)	0.05	0.1

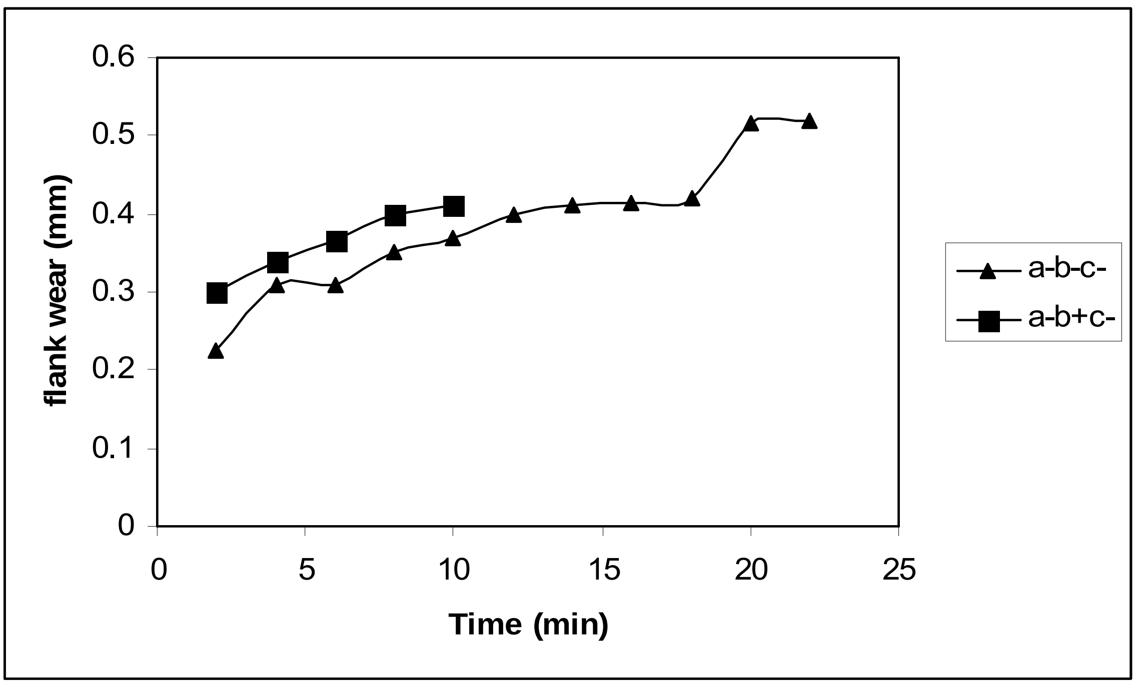
Table 6.21. Cutting parameters for 2^3 factorial design for carbide inserts

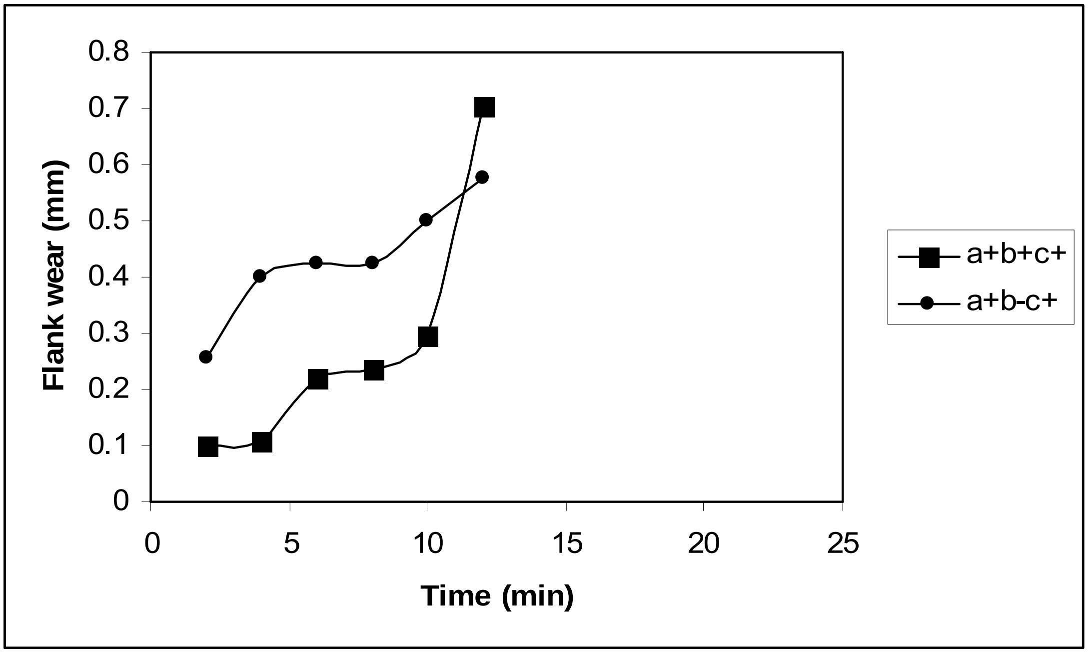
Turning test were carried and flank wear measurements were done at regular intervals of 2 minutes for a fixed time period of 16 minutes in each of the test runs. Table 6.22 illustrates the experimental results for flank wear.

Run	Level of Factors							Flank Wear (mm)
	A	B	C	AB	BC	AC	ABC	
1	-1	-1	-1	+1	+1	+1	-1	0.155
2	+1	-1	-1	-1	+1	-1	+1	0.085
3	-1	+1	-1	-1	-1	+1	+1	0.138
4	-1	+1	-1	+1	-1	-1	-1	0.130
5	+1	-1	+1	+1	-1	-1	+1	0.105
6	-1	-1	+1	-1	-1	+1	-1	0.149
7	+1	+1	+1	-1	+1	-1	-1	0.135
8	-1	+1	+1	+1	+1	+1	+1	0.140

Table 6.22. Results of flank wear for carbide inserts


0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Flank wear (mm)
0
5
10
15
20
25
Time (min)
a+b-c-
a+b+c-


0.6
0.5
0.4
0.3
0.2
0.1
0
flank wear (mm)
0
5
10
15
20
25
Time (min)
a-b-c-
a-b+c-



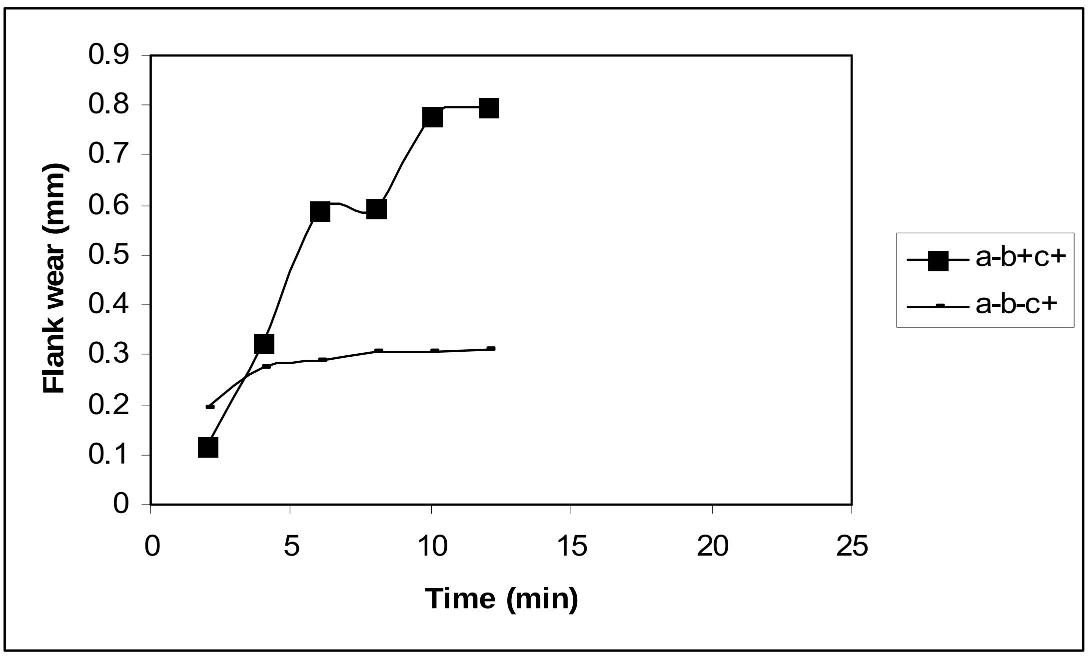


Fig. 6.11. Experimental results of flank wear

The experimental results were analysed with ANOVA which is used for identifying the factors significantly affecting the performance measures. The effect estimate summary and results of ANOVA are shown in table 6.23 and table 6.24

Factor	Effect Estimate	Sums of Squares(SS)	%Contribution
A	-0.007125	0.000101531	2.65
B	0.01225	0.000300125	7.87
C	0.00525	0.000055125	1.44
AB	0.00575	0.000066125	1.729
BC	0.0005	0.000005	0.013
AC	0.0285	0.0016245	**42.48**
ABC	-0.0265	0.0014045	**36.72**

Table 6.23 Factor effect summary for flank wear of carbides

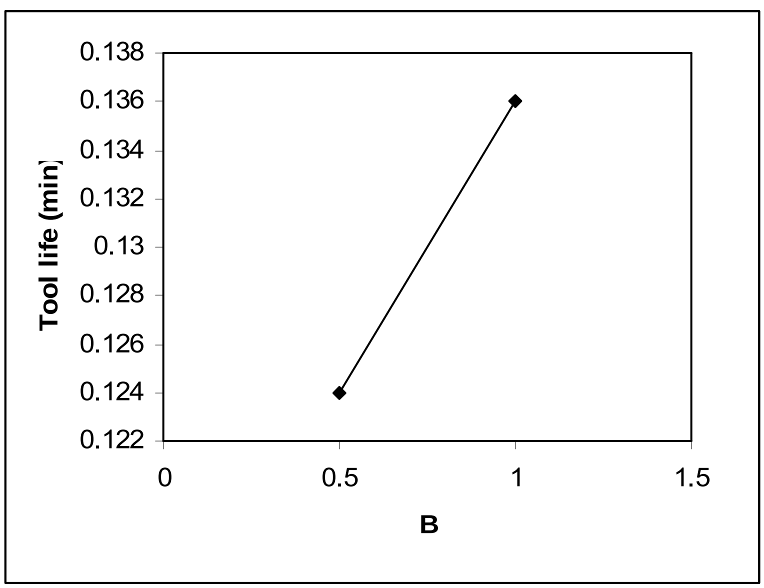
Source of Variation	Sums of Square (SS)	DOF	Mean Square (MS)	Fo
A	0.000101531	1	0.000101531	2.992102084
B	0.000300125	1	0.000300125	8.844635016
C	0.000055125	1	0.000055125	1.624524799
AB	0.000066125	1	0.000066125	1.948693013
BC	0.000005	1	0.000005	0.014736221
AC	0.0016245	1	0.0016245	47.87375122
ABC	0.0014045	1	0.0014045	41.39038694
Error	0.000271469	8	0.000033933	
Total	0.003823875	15		

Table 6.24. Results of ANOVA for flank wear of carbides

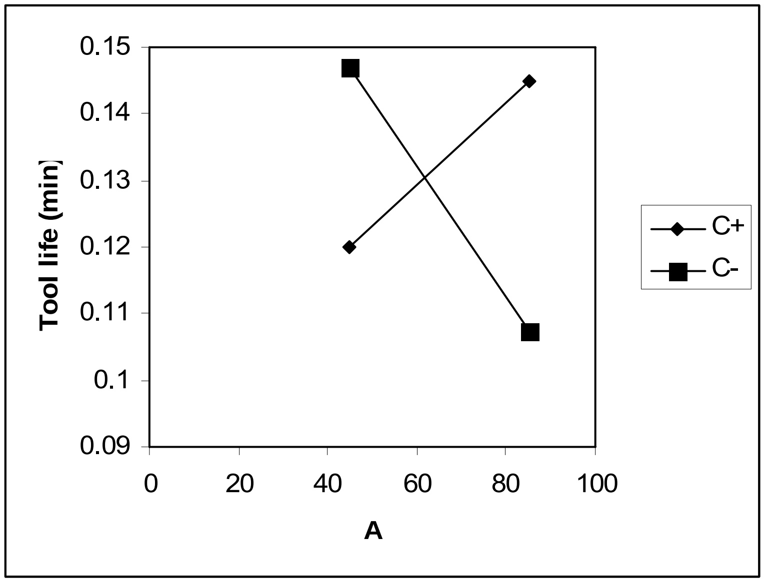
The tables showed that the interaction effect of cutting velocity and the feed rate has the most dominant effect on flank wear which explains 42.48% of total variation. The next significant contribution comes from the interaction effect of cutting velocity, depth of cut and feed rate with 36.72% of total variation followed by depth of cut having much lower level of contribution.

To check the adequacy of the model, Fstatistic was calculated which came out to be 14.9555468.Since F statistic is large, it can be concluded that atleast one variable has non-zero effect on tool wear. Each of the factorial effect was tested using Fstatistic and it was confirmed that AC interaction and ABC interaction are the only significant factors that affect flank wear in the present case.

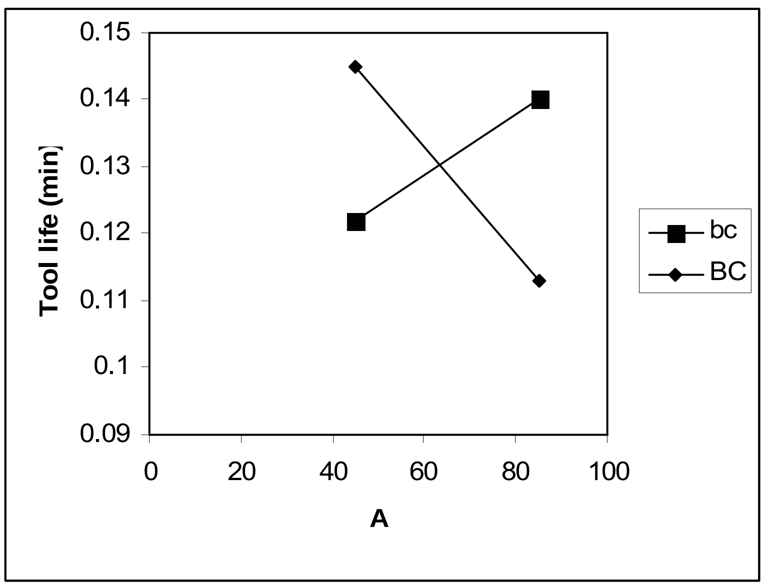
Main effect and interaction effect plots are drawn to study the behaviour of flank wear properly. Fig. 6.12 (a) explains the cutting velocity effect on flank wear. Tool wear appears to be increasing as the depth of cut increases from 0.5 mm to 1 mm. In fig. 6.12 (b) it is observed that at low level of feed rate tool wear is greatly affected by the cutting velocity. With low cutting velocity and high feed rate, tool wear increases drastically. This can be explained in terms of built up edge (BUE) formation which are supposed to disappear at higher values of cutting velocity. Hence minimum tool wear can be obtained with high cutting velocity and low feed rate. Fig 6.12 (c) illustrates the interaction effect between cutting velocity, depth of cut and feed rate.



(a) Main effect plot for depth of cut (B)



(b) Interaction effect plot for cutting velocity (A) and feed rate (C)



(d) Interaction plot for cutting velocity (A), depth of cut (B) and feed rate (C)

Fig. 6.12. Main effect and interaction effect plots for the flank wear in case of carbides

Following flank wear equation for machining carbide by using cryogenically treated carbide inserts showing the significant factors with the respective effect summary is shown as follows:

$$y = 0.129625 + \left(\frac{0.01225}{2}\right)X_2 + \left(\frac{0.0285}{2}\right)X_1X_3 + \left(\frac{-0.0265}{2}\right)X_1X_2X_3$$

The R^2 values for the developed model were calculated which are given as follows:
R^2=0.870

The flank wear equation so developed was applied to each of the test points to evaluate the residuals. Table 6.25 shows the predicted values and the residuals.

	Predicted value (y)	Observed value(y)'	Residuals (e=y-y')
(1)	0.155	0.150875	0.004125
a	0.085	0.095875	-0.010875
b	0.138	0.136515	0.001485
ab	0.130	0.134875	-0.000875
c	0.105	0.124375	-0.019375
ac	0.149	0.150825	0.001875
bc	0.135	0.134875	0.000125
abc	0.140	0.130625	0.009375

Table 6.25. Difference between predicted values and measured values of flank wear

The predicted values and the observed values are found to be fairly close indicating the fact that the flank wear equation developed for carbide (SNMG120412MP) insert while machining mild steel can be used for reliable estimates.

CHAPTER 7

CONCLUSION

1. The tool life is increased by 19% for M2 grade HSS single point cutting tools and 17% for carbide inserts for machining mild steel after the cryogenic treatment.
2. In the sliding wear test, the weight loss of cryogenically treated tools is more as compared to that of untreated tools. This can be attributed to the fact that tool becomes brittle after cryogenic treatment.
3. From SEM analysis, it is evident that refinement of carbides is more in case of cryogenically treated HSS tools in comparison to that of untreated tools.
4. There is not much difference in hardness between cryogenically treated and untreated M2 as well as S400 HSS tools.
5. Tool life equations have been developed using design of experiment (DOE) for machining mild steel by cryogenically treated HSS tools and carbide inserts.
6. For cryogenically treated HSS tools feed rate was found to affect the tool life most significantly. The second most significant factor came out to be the interaction effect of cutting velocity and depth of cut followed by the interaction effect of depth of cut and feed rate while machining mild steel.
7. For cryogenically treated carbide inserts (SNMG120412MP) the significant factor was found to be the interaction effect of cutting velocity and feed followed by the cutting velocity, depth of cut and feed rate followed by the depth of cut with much lower level of contribution while machining mild steel.

CHAPTER 8

REFERENCES

[1] Machado,M.B. da Silva, Metal Machining. Brazil : MG, (2003)

[2] Paulin, Frozen gears, Gear Technol. (1993), pp. 26–28.

[3] Smolnikov, Kossovich, "Cold Treatment of Cutting Tools" Volume No. 10, (1980): pp. 5–7.

[4] Tseitlin,. Kolensnichenko, Karnaushenko, Umanets, Zhmud, "Tool Life of High Speed Steel Cutters After Cold Treatment". Volume No. 10, (1980):pp. 7–9.

[5] Zhmud E.S., "Improved Tool Life after Shock Cooling". Volume No.10, (1980): pp. 3–5.

[6] Gulyaev A.P., "Improved methods of heat treating high speed steels to improve the cutting properties", Metallurgy .Volume No.12 1937 :p. 65.

[7] Reasbeck, "Improved tool life by the cryotough treatment". Metallurgia .(1989): pp. 178–179.

[8] Zamborsky D.S., "Control of distortion in tools steels", *The Heat Treating Source Book*, (1986) : pp. 73–79.

[9] Heberling J.M., Tool steel tutorial, Heat Treating 1992

[10] Barron F.R., "Yes—Cryogenic Treatments Can Save You Money! Here's Why",.Tapi Volume No. 57 ,(1974) : pp. 35–40.

[11] Popandopulo, Zhukova, "Transformations in High Speed Steels During Cold Treatment". Volume No.10, (1980): pp. 9–11.

[12] Huang, Zhu, Liao, Beyerlein, M.A. Bourke and T.E. Mitchell, Microstructure of cryogenic treated M2 tool steel, Material Sci. Eng.(2003): pp. 241–244.

[14] Vaccari J.A., "Deep freeze improves products". Amer. Machinist Automated Manuf. March (1986)

[15] Barron R.F., "Cryogenic treatment of metals to improve wear resistance" Cryogenics (1982): pp. 409–413.

[16] Moore M.A., A review of two-boby abrasive wear, Wear Volume No. 27, (1974): pp. 1–17.

[17] Yun, Xiaoping ,Hongshen, "Deep cryogenic treatment of high-speed steel and its mechanism". Heat Treat. Met. (1998): pp. 55–59.

[18] Molinari, Pellizzari, Gialanella, Straffelini,. Stiasny, "Effect of deep cryogenic treatment on the mechanical properties of tool steels", J. Mater. Process. Technol. Volume No. 118 (2001): pp. 350–355.

[19] D. Mohan Lal, Renganarayanan. Kalanidhi, "Cryogenic treatment to augment wear resistance of tool and die steels", Cryogenics . Volume No. 41,(2001): pp. 149–155.

[20] Huang,. Gao ,Huang, "Study on cryogenic phase change and wear characteristic of high speed steel", Acta Metall. Sin. Volume No.16 ,(2003):pp. 524–530.

[21] Leskovsek ,Ule, "Influence of deep cryogenic treatment on microstructure, mechanical properties and dimensional changes of vacuum heat-treated high-speed steel", Heat Treat. Met. Volume No. 29,(2002) (3): pp. 72–76.

[22] Mahmudi, Ghasemi ,Faradji, "Effect of cryogenic treatments on the mechanical properties and wear behaviour of high-speed steel M2", Heat Treat. Met.Volume No.27 ,(2000): pp. 69–72.

[23] Lin, Dong Wang, "Study on cryogenic treatment technology and mechanism of high speed steels", Trans. Met. Heat Treat. Volume No.19, (1998) : pp. 21–25.

[24] Collins D.N., “Deep cryogenic treatment of tool steels: a review”, Heat Treat. Met. (1996) (2): pp. 40–42.

[25] Zum Gahr, Microstructure and Wear of Materials, Amsterdam:Elsevier Science Publishers Inc., 1987

[26] J.D. Kamody, “Using deep cryogenics to advantage”, Adv. Mater. Process. Volume No. 10 (1998): pp. 215–218.

[27] Cohen ,Kamody, “Cryogenics goes deeper”, Cutting Tool Eng. Volume No.l50 , (1998) (7): pp. 46–50.

[28] Meng ,Tagashira, “Wear resistance and microstructure of cryogenic treated Fe–1.4Cr–1C bearing steel”. Scri Metall Mater. Volume No. 31 (1994): pp. 865–868.

[29] Dong, Lin ,Xiao, “Deep cryogenic treatment of high-speed steel and its mechanism”. Heat Treatment of Metals. Volume .No. 3**,** (1998): pp. 55–59.

[30] Collins ,Dormer, “Deep cryogenic treatment of a D2 cold-work tool steel”. Heat Treatment of Metals. Volume No. 3, (1997): pp. 71–74.

[31] Seah, Rahman ,Yong, “Performance evaluation of cryogenically treated tungsten carbide cutting tool inserts”. Journal of Engineering Manufacture **.**Volume No. 1, (2003): pp. 29–43.

[32] W.E. Bryson. Cryogenics, Cincinnati :Hanser Gardner Publications, 1999.

[33] Dong, Lin, Xiao. Heat Treatment of Metals .1998, p. 55.

[34]S.H. Avner. Introduction to physical metallurgy. New York: McGraw-Hill, 1982.

[35] Flávio J. da Silva**, “**Performance of cryogenically treated HSS tools”**.** ”. Journal of Engineering Manufacture . Volume No. 1**,** (2006): pp. 50–59

[36] K. Seah,Rahman,Yong, “Performance evaluation of cryogenically treated tungsten carbide tools in turning”. Journal of Engineering Manufacture . Volume No. 2**,** (2006): pp. 40–46