THE PROCESSING AND EVALUATION OF AN INVESTMENT CAST MAGNESIUM-BASE ALLOY

by

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Buat abah dan arwah emak yang tidak pernah mengerti erti kepayahan.

Sesungguhnya arwah emak ibarat 'sireh naik, junjung patah'.

Juga buat Ma dan anak-anak yang tabah.

ABSTRACT

Magnesium alloys are well known for their high affinity for oxygen and highly reactive characteristics. This has created difficulties in their processing. During melting, a protective film generated by chloride or fluoride based flux or an inert atmosphere created by a specific gas combination, is required to isolate the melt from the atmosphere in order to suppress any reaction.

At the casting stage, reaction with some mould materials is likely to happen. This has caused a serious problem in the casting of magnesium, particularly with the investment casting process. The mould–metal interface reaction must be eliminated or minimized if the production of castings with close tolerances and good surface finish is to be achieved.

In that respect, an investigation was conducted to establish a suitable investment casting mould material or inhibitor procedure in order to overcome the mould–metal reaction that occurs during the pouring of magnesium alloy ZRE1 into an investment casting alumino –silicate mould. The approaches taken were to: cast the alloy into moulds made up of several identified moulding materials; introduce CO_2/SF_6 gas inside the mould; and to use potassium borofluoride as an inhibitor, mixed with the mould material or coated on the mould face.

The results showed that introducing CO_2/SF_6 gas for several seconds within an enclosure prior to pouring produced a clean metallic surface, i.e no reaction was observed. The other approaches exhibited negative results.

An investigation was also conducted to establish the influence of investment casting process parameters on the mechanical properties of ZRE1 alloy. The relationships were established by casting the alloy at various mould and pouring temperatures. The specimens were then put under tension testing to establish their mechanical properties. The mould temperatures chosen for the investigation were 21 °C, 100 °C, and 200 °C; whilst the pouring temperatures were 700 °C, 730 °C, 750 °C, and 780 °C. Melting was conducted using both the flux method and fluxless method.

The results showed that the ultimate tensile strength was highest at a mould temperature of 200 °C and a pouring temperature of 700 °C and at it lowest when these temperatures were 100°C and 730°C respectively.

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION AND OBJECTIVES

Metal casting denotes a term for any process of melting metals and pouring them into moulds to produce the required solid shapes. Such processes have been known to man for more than 4000 years. Metal casting is considered a unique process among other metal forming processes for a variety of reasons. The most obvious reason is perhaps, the range of moulding processes available that are capable of producing complex components in any metal, ranging from a few grams to single parts weighing several hundred tons. It is estimated that castings are used in 90% or more of all manufactured goods and in all machinery parts used in manufacturing. They are also extensively use in transportation, building construction, oil and gas pipelines and a wide variety of other applications.

In common with other manufacturing processes, casting technology has witnessed huge improvements in terms of quality and productivity during the past thirty years. Many methods and techniques are continuously being developed to cope with the rapid advancement of the material technology, stringent demands on quality and cost, etc. Many precision methods have been developed. They are gravity die casting, otherwise known as permanent mould casting, pressure die casting, plaster mould casting, shell mould casting and investment casting. Out of these many precision casting processes, the investment casting process is the most flexible with respect to attainable intricacy, attainable precision and variety of alloys which may be cast within the size limitations inherent to it.

In the investment casting process, the pattern is built from wax by injecting molten wax into a metal mould, the wax is removed and dipped several times in slurries to build up the required mould thickness. In between dippings, the wax that has been dipped with slurry is stuccoed with refractory materials. It is then dewaxed and fired at high temperature before molten metal is poured into the mould. Currently, the process is being utilised to manufacture a wide variety of ferrous, superalloy and aluminium based components, catering primarily for the aerospace industry.

During recent years, beside considering the precision of the parts produced, automobile, aerospace and engineering industries are now looking into weight reduction. In the United State, requirements for Corporate Average Fuel Economy (CAFE) of 27 MPG set for every vehicle produced from 1989, has forced car manufacturers to develop and implement strategies to reduce vehicle weight, which in turn reduces fuel consumption. This concern applies in the aerospace industry. Manufacturers are looking to replace traditional materials with lighter material so as to produce lighter aircraft which again reduces fuel consumption.

With this trend, magnesium has been seen to be a potential alternative, since it is the lightest metal and its ore is abundant which should ensure a continuous supply and, indirectly, stabilise the price. It possesses many advantages which other structural metals do not.

Although magnesium has many advantages which suits the above demand, attention must be paid to its processing. Magnesium is well known for its high affinity for oxygen and high reactivity characteristics. In its molten state, a protective film generated by chloride or fluoride based flux or an inert atmosphere created by a specific gas combination, is required to isolate the melt from the atmosphere in order to suppress any reaction. This must be addressed promptly when melting, failing which, within seconds many blooms will be formed on the surface, glow red and burst into white bright flames.

With its high reactivity characteristic, reaction with some mould materials is likely to happen. This has caused a serious problem in the casting of magnesium. The mould–

metal interface reaction must be eliminated or minimized if the production of castings with close tolerances and good surface finish is to be achieved. This is particularly true for investment casting, for which a good surface finish and close casting tolerance are characteristic reasons for its choice.

Many recommendations have been made to overcome such problems but their emphasis was on sand casting (1-3), and few have been made for other processes (1)(4). An inhibiting substance used to suppress mould–metal reaction in solid mould investment casting, has been mentioned in some articles (1)(5)(6). The mechanism of the mould–metal reaction has been studied and reported by many investigators, but the studies were based on steel casting (7–11).

Since, in shell investment casting, the mould is fired at a very high temperature, normally up to 1065°C, the recommendations for inhibitors to be used in sand casting or other processes might not apply to investment casting. It is therefore important for a study to be carried out to establish a method or procedure in order to overcome such reaction when casting a magnesium alloy by shell investment casting.

This study was directed to develop a suitable mould material or inhibiting procedure to overcome the reaction between the alloy and the oxide of the refractory material. The study was conducted using magnesium alloy ZRE1 (0.8–3% Zn, 2.5–4% RE, 0.4–1% Zr) cast into shell investment casting moulds of specific shape. In the second phase of the project, assessment of the mechanical properties of the alloy at ambient temperature was conducted as a basis of evaluating the influence of casting parameters on the mechanical properties.

The report is presented in 8 chapters. Chapter 1 highlights the general aspects of magnesium and the problems associated with it, particularly in melting and casting. Chapter 2 describes various aspects of magnesium base alloys with emphasis given to the melting technology of magnesium, inhibitors used in melting technology, casting, and grain refinement. The metallography and testing procedures for magnesium alloy are also briefly presented. The advantages and disadvantages and the details of the investment casting process are presented and discussed in Chapter 3.

Chapter 4 explains the experimental approach and procedures adopted. Chapter 5 tables the findings of these experiments. In Chapter 6 the experimental findings are discussed. Chapter 7 and Chapter 8 present conclusions from the experiments and suggestions for further work, respectively.

CHAPTER 2

MAGNESIUM–BASE ALLOYS

2.1 INTRODUCTION

Magnesium and its alloys are materials with low density. They show good mechanical properties at room temperature, and remarkable properties at elevated temperatures (12). ? Besides, they provide excellent dent resistance and high damping capacity, permit easy machining and fabrication, and a long list of excellent properties ready to be exploited. They have proved to be particularly suitable for certain special applications.

Being the world's most plentiful and useful light metal, magnesium was discovered in 1808. Serious industrial production was only started in the late 1880's, using a relatively simple, inexpensive process and a plentiful supply of cheap power. Magnesium remained an unfashionable material until World War II, when in a short period of time it came into great demand for use in various aircraft components–a very rapid transformation from next to oddity material to a very vital war–time commodity.

The use of magnesium alloys, however, declined slightly starting from the early 1960's due to various drawbacks (12). Magnesium alloys were known to be highly susceptible to corrosion and were higher in price than aluminium alloys, a factor which also applies to the cost of casting, machining and surface treatment. The casting of magnesium generates a higher cost because of the need for safety and environmental protection. Another factor which held back the use of magnesium alloys was the lack of technological knowledge in producing them, which led to various quality problems and restricted their use for many applications.

In recent years, advances in alloying and casting techniques, the use of more exact design criteria, a better understanding of the true economics of production, and the trend towards weight–reduction in aeronautics, automobile and engineering industries, have offered new openings for the light material magnesium.

In this chapter, the various aspects of magnesium base alloys are discussed. This includes the classification of magnesium base alloys and the justification for the use of magnesium with respect to other traditional materials, particularly aluminium and zinc.

Discussion is also focused on the aspects of manufacturing magnesium, and casting processes which might be used in fabricating the alloys.

2.2 DESIGNATION OF MAGNESIUM ALLOYS

Magnesium alloys are designated using a system established by the American Society of Testing and Materials (ASTM) which covers both chemical compositions and temper. Designation would differ from country to country, depending on the convenience of use. For example, the material EZ33, designated by the ASTM, is coded by the British Standard (BS 2970) as MAG6.

Based on ASTM, designations begin with two letters representing the two alloying elements specified in the greatest amount. The letters are arranged in order of decreasing percentages or alphabetically if the elements are present in equal amounts. The letters are followed by respective percentages rounded off to whole numbers followed by a final serial letter. The serial letter indicates some variation in composition of minor alloying constituents or impurities. The letters that designate the more common magnesium alloying elements are:

A – Aluminium

E – Rare Earth

H – Thorium

K – Zirconium

L – Lithium

M – Manganese

Q – Silver

S – Silicon

Z – Zinc

For example, magnesium alloy AZ31 contains 3% aluminium (code letter A) and 1% Zinc (code letter Z).

2.3 WHY MAGNESIUM ALLOYS?

The current trend shows that there is an increase in the usage of magnesium alloys especially in the field of die casting and it is expected to expand more rapidly in the coming years (13). The current increase is due to magnesium's excellent properties, which are detailed in the following sections.

1. Strength–to–Weight/Stiffness–to–Weight Ratio.

Magnesium is by far the lightest of the commonly available metals. With a density of just 1.8 g/cm^3 magnesium alloy AZ91 is one third lighter than aluminium, well under half the weight of titanium and four times lighter than steel. It is similar in weight to many engineering plastics.

Despite being one of the lightest materials, magnesium displays outstanding strength to weight and stiffness to weight ratios. Magnesium alloys have a specific tensile modulus comparable to 380 aluminium and some alloys have excellent elongation properties.

Mechanical properties are in the same range as those of aluminium alloys and this, coupled with the low density, provides magnesium with the best strength–to–weight ratio of any commonly available structural material.

2. Low Modulus of Elasticity

Because of their low modulus of elasticity (45 x 10^3 N/mm^3 compared with aluminium at 67x 10^3 N/mm^3), magnesium alloys can absorb energy elastically and, in combination with moderate strength, this provides excellent dent resistance and high damping capacity which allows the material to absorb noise and vibration. This is an essential consideration when selecting a material for an application which must meet certain noise and vibration criteria.

Furthermore for highly stressed parts where rigidity is a requirement, and necessitates the use of ribs or a slight wall thickness increase, a weight reduction of at least 20% over aluminium can be realised.

3. Corrosion Resistance

This point has actually limited magnesium use on many occasions, particularly in the automotive industry. New metal technology, advances in design and better understanding of the properties of magnesium have reduced the twin threats of galvanic and atmospheric corrosion. Galvanic corrosion occurs when two dissimilar metals come into contact with one another in the presence of electrolytes. This can easily be prevented by introducing a thin insulative membrane separation medium.

Likewise, advances in metallurgy have minimized corrosion due to atmospheric exposure of magnesium. Keys to this advance have been the control of critical contaminants such as copper, nickel and iron. In fact, magnesium typically outperforms steel in severe operating environments. High–purity magnesium alloys will even outperform die cast aluminium. Further corrosion protection can be provided through surface treatment, for example plating and painting.

More recently, high purity alloys have largely eliminated corrosion problems. For example, high purity magnesium die casting alloys AZ91 and AM60B have corrosion resistance equal to, or better than, aluminium alloy 380 (LM24) and cold rolled steel when subjected to the ASTM B117 salt spray test.

4. Machinability

Magnesium has long been recognised as the easiest of all structural metals to machine. Less power is required to machine magnesium than other conventional materials. Chip removal is easier, machining is faster and fine surface finishes can be obtained. Because magnesium alloy castings can typically be machined in one pass, production efficiency is enhanced and tool life can be dramatically improved.

The superior castability and non–galling characteristics of magnesium alloys permit parts to be designed with thinner walls and lower draft requirements. Because magnesium does not adhere or solder to the die, draft and taper clearance angles can often be trimmed by 25% when magnesium is substituted for aluminium. This will actually minimize secondary machining operations.

5. Joining

Many of the sand casting magnesium alloys and the wrought alloys can be welded by conventional arc welding processes. Joining, using adhesive bonding, is possible since the adhesive can act as an insulator and a sealant, so removing the danger of galvanic action. Magnesium alloys will accept phenolic, ethoxyline, rubber and vinyl adhesives as well as epoxies.

6. Parts Per Unit Weight

Though magnesium costs more by weight than some other metals, its total cost is very competitive. With magnesium, there is more volume of metal per unit mass with which to work. For instance, a die caster can produce one and a half times as many parts from magnesium as from an equal mass of aluminium. Every 1 lb (0.45kg) of magnesium used in an automobile instead of cast iron would result in a weight saving of 5.2 lbs (2.31 kg).

7. Price Stability

For commercial die casting alloys magnesium has seen a price stability over recent years, unmatched by other structural materials. Over time, the price of magnesium has also remained more stable than that of aluminium. Because magnesium is the sixth most abundant metal on earth, risks of shortages and price swings are slim. This has also resulted in competitive raw material prices when compared with zinc, aluminium or plastics.

8. Recycling Potential

Another important feature of magnesium alloys is that they have high recycling potential and can be used many times.

Other justifications for the use of magnesium were also mentioned in various papers (14–19).

2.4 CLASSIFICATION OF MAGNESIUM ALLOYS

Duffy (20) has categorised magnesium base alloys into the 6 major categories described in the following sections.

2.4.1 Aluminium–containing Alloys

The first commercially available alloys were based on the Mg–Al–Zn system with a small addition of manganese to improve corrosion resistance. The most widely used alloy is designated AZ91 and has a nominal composition of 9% Al, 1% Zn, 0.2% Mn, balance Mg.

These alloys are the cheapest of all magnesium casting alloys but are also the most difficult to cast successfully. Castings are prone to microporosity and, in the case of sand or gravity castings, normally require extensive sealing treatments to produce pressure tight castings. These alloys are therefore used most widely in high pressure die cast form where microporosity and other problems can be minimised.

They have moderate strength in the fully heat treated (T6) condition although the yield strength is less than half of the tensile strength. Their use is further limited by significant loss of strength with increasing temperature and the alloys are not suitable for use above 120° C. Despite these drawbacks, AZ91 has been used for aerospace casting applications where high temperature mechanical properties are less critical.

The corrosion performance of these alloys has been improved dramatically by the production of high purity variants. Specifically, reducing the levels of residual elements, including Fe, Cu and Ni, has led to a hundred–fold improvement in corrosion resistance in salt fog tests.

2.4.2 Zirconium–containing Alloys

A major step forward in the development of magnesium alloys was the discovery of the intense grain refining effect of zirconium. Typically, grain sizes of 30–40μm can be achieved in sand castings compared to the relatively coarse dendritic structure of Mg–Al–Zn alloys. However, Mg–Zr alloys were not sufficiently strong for commercial applications and Zr could not be used in existing alloys as it is removed from solution to form stable compounds with Al and Mn. This led to a requirement for a totally new series of Zr refined alloys.

Some zirconium refined Mg–Zn alloys, including ZK51 (4.5% Zn, 0.7% Zr) have been developed. Alloying elements that have been used in zirconium refined alloys include rare earth elements, silver, thorium and yttrium

2.4.3 Rare Earth–containing Alloys

Magnesium–Rare Earth alloys have good casting characteristics due to the formation of relatively low melting point eutectics which form as grain boundary networks, suppressing microporosity. However, even with zirconium refinement, Mg–RE alloys do not have sufficiently good properties for commercial applications and further alloying additions are required. The addition of Zn to Mg–RE–Zr produces alloys with commercially acceptable strength levels. Mg–Zn–RE–Zr alloys containing Ce/La rare earth elements are currently the most widely used alloys for sand and gravity castings. These alloys have gained application in aircraft gearbox casings and canopies; and, in motor racing, for gearbox casings, engine covers, sumps and wheels.

2.4.4 Silver–containing Alloys

These alloys were developed for improved ambient and high temperature properties compared to Mg–Zn–RE alloys. In these alloys the Ce/La rare earth elements are replaced by Nd/Pr elements which have higher solubility in Mg. The alloys can be given a full T6 heat treatment after which good tensile properties are maintained up to a temperature in excess of 200° C.

The most common alloy is QE22 which contains 2.5% silver. In a subsequent development, 1% of the silver was replaced by 0.06% copper (EQ21 alloy) giving a lower cost alloy without sacrificing mechanical properties.

These alloys have been used widely for more demanding aerospace applications where improved high temperature performance is required. Silver–containing alloys are being used increasingly for gearboxes for motor racing, primarily due to their superior ambient temperature properties.

2.4.5 Thorium–containing Alloys

Thorium additions confer excellent creep resistance to magnesium alloys enabling them to be used up to 350° C. This alloy has been used widely in aero engines where good creep properties are required. However, thorium is a radioactive element and concerns over manufacture and foundry handling of these alloys has led to more complex and costly production operations while environmental factors have caused significantly increased disposal costs. These alloys will be phased out in the future and no new applications are being considered.

For applications where thorium alloys are currently used, the only alternative materials currently available are high temperature aluminium casting alloys or titanium alloys but these would lead to significant weight or cost penalties.

2.4.6 Yttrium–containing Alloys

A major recent development was the Mg–Y–RE (Nd) WE54 and WE43 alloys. These were developed for improved mechanical properties over existing magnesium alloys, both at ambient and elevated temperatures. These alloys have dramatically improved corrosion resistance compared to other magnesium alloys and are comparable to many aluminium alloys in salt fog tests.

Pure yttrium is an expensive element with high affinity for oxygen and is therefore substituted by a lower grade yttrium concentrate containing around 60% Y_2O_3 to form a 75–80% Y material.

An argon + SF_6 inert gas mixture must be used when melting yttrium–containing alloys due to their reactivity in the molten state. The WE54 alloy has better proof tensile strength but is inferior to WE43.

2.5 MELTING PRACTICE

Added cost has always been involved with the extra attention required by molten magnesium as compared with aluminium and zinc. When molten aluminium is exposed to the atmosphere there is instant reaction and the formation of a thin film of aluminium oxide, Al_2O_3, on the surface of the melt. The film is protective against further oxidation and even though it thickens gradually (at a parabolic rate with time) the melt still appears metallic after hours of exposure.

When molten magnesium is exposed to ambient air there is also instant reaction to form magnesium oxide but, in contrast to the aluminium oxide film, it is neither thin nor protective. In a matter of just seconds many blooms form on the surface which first glow red and then soon burst into white flames. The magnesium vapour oxidizing in the gas phase creates a white MgO fume. The MgO on the surface is neither smooth nor coherent with the surface. Because of this, melting of magnesium demands the provision of a protective film in order to avoid further oxidation.

Thus it appears that the solution to the oxidation problem is either to modify the composition of the atmosphere, so that reaction is halted, or to isolate the melt from the air with a non–reactive barrier. The latter is normally achieved through sprinkling of flux prior to charging and during the melting operation. It is normally known as the flux process. The former is a fluxless process which requires that melting be performed in an inert atmosphere.

2.5.1 The Flux Process

Generally, flux is used for two reasons (21):

1. to protect the melt from oxidation during the initial melting operation,

2. to effect melt refining by removal of certain impurities prior to pouring.

A magnesium melt can be covered with a liquid film of flux consisting of $MgCl_2$ and other chlorides (1) and mixtures of salts (22).

In order to protect the metal during melting, a flux addition of about $1^1/_2$% of the charge weight is placed at the bottom of the crucible (23). Emley, in his practice of melting, suggested that the flux should be sprinkled around the sides and on the bottom of the crucible, with the amount varying from 0.5% to 2.0% of the total charge weight (1) (24).

When melting is about to begin, flux is sprinkled over the charge and subsequently added as required to prevent or check burning. Once melting is complete, the metal surface is covered with a different type of flux, and from this stage onwards only an 'inspissated' flux is used.

For melts that are prepared from ingots and a large portion of scrap, as in billet casting, it is possible to use a single all–purpose flux instead of using two different fluxes as was mentioned above (1).

However there are some objections to the use of salt flux. First, the film itself oxidizes in the atmosphere and hardens into a thicker oxychloride type of compound, which is likely to crack and expose the molten metal to the ambient air. It is therefore, necessary to supply fresh flux at those places where the flux cover is cracked or disturbed.

A second problem is the possibility of flux contamination in a finished casting. The flux is hygroscopic and a flux inclusion may become a centre of accelerated corrosion if the article is exposed to the atmosphere. Also, flux or oxide inclusions could cause weak points in a casting. However, if care and a good technique of melting are employed, flux contamination problems can be kept under control.

The third problem associated with the use of flux is that flux fumes and flux dust can cause serious corrosion problems in the foundry. If ventilation is inadequate, the flux fumes will corrode building materials and equipment parts. The hygroscopic nature of the flux dust will result in a wet salt film on a humid day which can be particularly damaging to ferrous materials. The problems of using flux were summarised in two articles (25)(26).

2.5.2 The Fluxless Process

This technique provides solutions to some of the problems created by the flux process, for instance, the defects caused by flux inclusions particularly in die castings. It has also proved to be acceptable because melting losses can be considerably reduced.

Few gases are found to be effective in protecting a magnesium melt from an oxidising atmosphere. The selection will depend on the available melting equipment, type of alloy and, above all, economic considerations. Carbon dioxide, nitrogen, argon, and sulphur dioxide are some of the well known gases that could protect magnesium during melting (27). Emley listed other gases and vapours which have demonstrated an inhibiting action. The list includes HCl, H_3BO_3, NH_4BF_4, NH_4SO_4, and BF_3.

It has been shown that a small percentage of SO_2, BF_3 or SF_6 gas sufficiently protects molten magnesium from oxidizing in air. It was also shown from the experiments conducted by Fruehling and Hanawalt (28) that, in the presence of SO_2, BF_3 or SF_6 in the atmosphere, water vapour had very little effect on the magnesium melt. Since SO_2 and BF_3 are highly toxic and corrosive, SF_6 is preferred. SF_6 is known to be much better than the salt fluxes, because the protective film continually renews itself, provided that the melt is not excessively agitated and sufficient presence of gas is maintained. The SF_6 protection layer is actually invisible being only a few molecules thick (25). Sulphur hexafluoride is also preferred over the other sulphur and fluoride compound families because SF_6 gas is stable, non–corrosive and, most importantly, non–toxic.

A pure carbon dioxide atmosphere proved to be very effective in the temperature range of 621–676°C but when it is contaminated with a substantial quantity of air, blooms tend to form and burning starts (28). It was also concluded that 100% CO_2 provides the most effective protection for the molten AZ91 alloy. However, if contamination cannot be avoided, the combination of CO_2 plus traces of inhibitors, such as SF_6 or SO_2, provides practical and superior protection. It was found that (29) (30) air/sulphur–hexafluoride gas or air/carbon–dioxide/sulphur–hexafluoride gas mixtures of low (<2%) SF_6 concentration provide an effective protection to the melt. For maximum protection some air must always be present since the fine protective film formed is basically magnesium oxide (MgO) (30).

Inert gases like helium and argon (30), were found completely effective in preventing oxidation, but the system requires an air–tight enclosure above the molten metal to ensure effectiveness. This renders the process impractical, i.e it makes routine handling, such as addition of more magnesium or extraction of the melt, difficult. These gases do not produce any protective film which therefore permits free evaporation of magnesium to the air (26). The vapour can condense on a cold surface and crystallise into fine crystals. This can be hazardous if the crystals are exposed to air. Furthermore, both gases are very expensive to use. Despite the impracticality of using helium and argon, Unsworth and King in their attempt to develop a new high performance yttrium–containing alloy, used argon with $^1/_2$% SF_6 as a protective gas (31). However, their experience showed that it did not give as good protection to the melting surface and pouring stream as did a CO_2–2% SF_6 mixture.

With this technique, the protection afforded to the magnesium enables it to be melted effectively with a non–toxic and odourless gas mixture. Furthermore, melt loss can be considerably reduced (30), by about half in some cases,since the amount of sludge at the bottom of the crucibles is greatly reduced.

2.5.3 Grain Refinement

There are various methods of grain refining. One of the methods used in the earliest days was superheating. The typical procedure of superheating is to melt the metal to a temperature of 870 to 925° C (1600 to 1700° F) and hold for about 15 minutes. It is then cooled rapidly to the pouring temperature, by removing the crucible from the furnace, followed by pouring it into the mould. Best results are obtained if the metal is poured immediately. Brace and Allen (21), in reviewing the work by Fox and Lardner, concluded that grain refinement of magnesium alloys by superheating was confined to those containing aluminium. It was also shown that long holding times, at the temperature of 850–900°C, caused grain coarsening and even a short period of holding (about 5 minutes) at 950° C was equally harmful. Baker et al (32) in their study on the effect of aluminium content found that, in general, in order to achieve maximum refinement, alloys with low aluminium content required a greater degree of superheating than alloys of high aluminium content.

The technique has other disadvantages, it shortens crucible life and increases the iron content of the alloy melted.

An alternative technique was developed in Germany in which a small quantity of anhydrous $FeCl_3$ was added to the melt (Elfinal process) and grain refinement was attributed to the nucleation by iron–containing compounds. This method also had its disadvantages since the deliquescent nature of $FeCl_3$ made it hazardous and the presence of as little as 0.005% iron could decrease the corrosion resistance of the alloys. To counteract the corrosion problem, manganese could be added to the melt but it effectively prevented grain refinement by $FeCl_3$.

Inoculation is one of the methods that can be employed for the purpose of grain refining. Inoculation of the melt with fine particles of a suitable compound can promote heterogeneous nucleation. The principal requirements are that the inoculant must be stable in the melt, be finely dispersed, have similar density to the melt and grow epitaxially with the metal to be solidified. This technique has been used successfully to control grain size in a wide range of alloys either by making deliberate additions to the melt or by coating the interior surface of the investment mould (33).

The method in current use, for alloys containing aluminium as a major alloying element, is to add volatile carbon–containing compounds to the melt. Hexachlorethane (0.025 to 0.1% by weight) in the form of briquettes is commonly used and is held at the bottom of the melt (34) where it disassociates into carbon and chlorine. Release of chlorine causes some degassing of the melt which is a further advantage of the method. Hexachloroethane can also be applied by dusting the substance in the mould (35). For effective treatment, the operation should be conducted when the melt is between 760°C and 815°C (1400°F to 1500° F). After the reaction is complete, holding temperatures must be maintained above 760°C (1400° F) (23). A study carried out by Kumar & Mahanty (36) disclosed that an excess addition of hexachloroethane might cause grain coarsening since the effect of hexachloroethane is sensitive to concentration. Hexachlorobenzene (23) and paraffin wax (1) may be used as substitutes for hexachloroethane.

It was also found that aluminium and graphite cause a significant reduction in grain size of commercial magnesium and magnesium–1% aluminium (36).

Calcium carbide is sometimes used for grain refinement, but is only effective if the treatment is conducted at about 760°C (1400°F) and the metal is kept above that temperature during casting (23). After the crucible is desludged, the metal temperature is raised to 760°C to 787°C (1400°F to 1450°F), calcium carbide, amounting to about 1% of the metal weight and in pea size pellets, is sprinkled on the melt surface which is then thoroughly stirred. The metal must be re–treated every 3 to 4 hours and the crucible should be desludged after each treatment.

Failure to achieve successful grain refinement by carbon inoculation or superheating normally indicates the presence of an inhibiting element; for example, through the contamination of the melt with zirconium or beryllium–bearing scrap. When this happens, it is best to decant the melt into ingots or a separate hot crucible, knowing that zirconium or beryllium largely remains behind at the bottom of the melt in the form of insoluble particles. Retreatment of the decanted metal in a separate crucible should give a satisfactory result.

With the magnesium–zirconium alloys, very effective grain refinement is achieved by the zirconium addition. To achieve the optimum fineness of grain, it is necessary for the melt to be supersaturated with soluble zirconium. Thus, it is necessary for an excess of zirconium to be added to the melt, over and above that which is theoretically required to provide the required soluble zirconium level. The solubility of zirconium in liquid magnesium is about 0.7% at 800 °C and increases only slightly at higher temperatures (24). The solubility decreases to about 0.5% at the melting point of magnesium. It is believed that the intense grain refinement of magnesium by zirconium is caused by the precipitation of zirconium–rich particles as the melt cools the precipitated particles serving as nuclei for crystalisation. The following factors must therefore be observed if the zirconium is to be effective (24):

1. The zirconium must be in solution in the magnesium when the melt is prepared.

2. The most effective grain refinement will be achieved only by alloying the maximum amount of zirconium which will go into solution.

3. Control of temperature is essential when preparing the melt.

Another method of grain refinement that has successfully been applied to other alloys is the use of vibration (33)(37)(38). Quoting Campbell's extensive review on the principles of grain refinement by vibration during solidification, McLean (33) states that, in alloys which solidify dendritically, grain refinement occurs by fragmentation of the dendrites and the nucleation of new grains from the misoriented fragments. For aluminium alloy it is suggested that slow but large amplitude vibrations of the moulds will lead to adequate grain refinement. In contrast, the work of Hincliff and Jones reviewed by Walker (37), reported that effective grain refinement could be produced in a high temperature alloy using subsonic frequencies, and that increasing frequency rather than increasing amplitude had the most potent effect on grain size.

Grain refinement by gas bubbling was also reported to be used in refining other alloys (39).

2.5.4 Alloying

The only elements commonly present in magnesium alloys not containing zirconium are aluminium, zinc and manganese, but beryllium may be added to reduce oxidation in the molten stage. Beryllium addition however will coarsen the grain size of castings.

For alloying small melts, using aluminium and zinc, additions can be made with a flux spoon which is held beneath the metal surface until melting of the added metal is complete. For larger melts, the preheated alloying metals are suspended in a perforated alloying bucket or long handled basket which is gradually lowered into the molten magnesium. When melting of the alloying metal is complete, the melt is plunged, or stirred with the basket until well mixed.

In the case of alloying with manganese, it is a common practice to add manganese in the form of $MnCl_2$. The melt temperature is raised to 750°C, the metal surface cleaned by skimming, and powdered $MnCl_2$ shaken onto the metal surface. On stirring the melt, some of the liberated manganese dissolves in the magnesium. Manganese recovery efficiencies of 50–80% are normally obtained, depending on the care with which the process is conducted.

The most convenient way of adding beryllium as an alloying element to zirconium–free melts is by a hardener alloy with aluminium, e.g Al–5% Be. Such an alloy can be readily added to magnesium–aluminium alloys and no special precautions against toxicity are required. Recoveries of beryllium depend on the aluminium contents of the melt and beryllium hardener, the iron content of the melt, and the settling condition employed.

For the zirconium–containing alloys, alloying constituents tend to be lost during each remelt operation. The element has to be added each time remelting occurs. Such correction can be made by adding the pure metals themselves (such as zinc etc) or hardener alloys with a fairly high content of the alloying element. For example zirconium may be added as a master alloy of about 30% of zirconium in magnesium; and cerium rare–earths as a master alloy of 20% cerium rare–earths in magnesium.

Composition control must allow for the fact that the addition of a master alloy to correct one element may lead to a dilution of the melt. This will cause the content of other elements to be reduced. Normally these master alloys are added to the melt by placing them into a preheated steel basket from which they are readily dissolved into the melt.

In adding zirconium master alloy as the alloying element, the required degree of supersaturation of the melt with zirconium may be achieved by a puddling technique which is followed by a settling procedure to allow insoluble zirconium complexes to settle. Care must be taken not to hold the melt for too long since loss of zirconium from the melt will result.

2.5.5 Gas Absorption

As with aluminium, hydrogen is the only gas that dissolves in molten magnesium. The sources of hydrogen are mainly from water vapour, in damp fluxes or corroded scrap/ingot, and improper melting procedure. This was demonstrated experimentally by De Haven, Davis and Eastwood, reviewed by Emley in his book (1). Therefore pick–up can be minimised by taking adequate precautions with materials and practices.

A low hydrogen content reduces the tendency to gas porosity which is common to aluminium and zinc containing alloys and these materials should be degassed with chlorine. The optimum temperature for degassing is 725°C to 750° C. If the melt is below 713 °C then solid $MgCl_2$ will form which gives little protection from burning, whilst at temperatures much above 750° C, magnesium losses through reaction with chlorine become excessive.

Gas porosity is normally not a problem with zirconium–containing alloys since zirconium itself will remove hydrogen as ZrH_2 and it is generally unnecessary to degas these alloys. However, such a treatment will improve the tensile properties of certain Mg–Zn–Zr alloys and indirectly minimise the loss of zirconium as the insoluble ZrH_2.

2.5.6 Inhibitors

The mechanism by which inhibitors function, particularly in sand casting, is not completely understood. However, it has been ascribed to three principal reactions (2)

1. formation of a protective film on the surface of the molten magnesium alloys as it enters and takes form in the sand mould;
2. formation of a protective atmosphere in the mould cavity;
3. formation of a protective coating around the sand grains forming the mould.

The formation of a protective film on the surface of the metal is undoubtedly the most important of the three reactions and is the basis for success of the more important inhibitors. Protective atmospheres are beneficial, but they are unable to confer adequate protection when heavy casting sections are involved. The formation of a protective coating around the grains is of lesser importance but such a coating will prevent reaction with thin wall castings under favourable conditions.

The practice for casting (especially in sand casting) of magnesium alloys differs from that applicable to other metals. This is due to the precautionery steps that must be taken to prevent metal–mould reactions. Metal–mould reactions can be prevented by adding inhibitors to the mould materials. Inhibitors that are normally used are sulphur, boric acid, potassium fluoborate, and ammonium fluosilicate. Sulphur and boric acid are normally added to sand moulds as inhibitors with a little addition of diethylene glycol (e.g up to $^{1}/_{4}$%) to reduce drying of the mould (1)(2).

Sulphur is one of the oldest and most universally used inhibitors. It volatilizes readily in the mould at low temperatures, at the first approach of the liquid metal, to form sulphur vapour which in turn reacts with the metal to form a protective surface film, and simultaneously reacts with the water vapour to form sulphur dioxide. This in turn reacts with the metal forming additional surface protection. Sulphur offers excellent initial protection and is adequate for castings of light section when additions of 0.5 to 2% by weight are made. In a situation where the environment is of greater concern, where sulphur is known to generate toxic fumes, potassium borofluoride may be used in partial replacement of sulphur. A study carried out by Sheptak (4), to evaluate various effects of inhibitors in resin shell moulds, showed that ammonium borofluoride was the only satisfactory addition agent. Its use resulted in slight discolouration of the surface but there was no evidence of burning or of metal sand reaction.

If insufficient inhibitors are present in a sand mould in particular, local reaction can occur between magnesium and moisture in the mould. Mould reaction usually (1) discloses itself as white spots on the outside of the casting, but it appears that appreciable hydrogen pick–up can occur without visible signs of superficial oxidation. This may lead to increased microporosity in alloys containing aluminium and in the zirconium alloys.

In general, the amount of inhibitor added depends on 3 major factors (23).

i. Moisture content of the moulding sand

Other factors being equal, the less moisture in the sand, the less inhibitor is required.

ii. Pouring temperature of the metal

The higher the pouring temperature of the metal, the greater the reactivity, and correspondingly greater amounts of inhibitor will be required.

iii. Cooling rate of the metal in the mould

The larger the casting (or the thicker the sections) the slower the casting will cool. Volatile inhibitors are soon lost from the surface of the mould in contact with the metal,requiring replenishment by inhibitors at a distance from the surface of the mould until the casting surface has solidified.

In the case of precision sand casting using water–free binders, an addition of 0.25–0.5% of KBF_4 and 0.75–1.0% sulphur is sufficient to prevent metal mould reaction (1). Baker used 6% sulphur and 0.5% boric acid in sand moulds used in his study to test for dissolved gas in magnesium alloy castings (40). Reviewing the work by Allen, Emley stated that a powerful liquid obtained from ground NH_4HF_2 and B_2O_3 may resolve the problem of plaster attacked by magnesium melt in the Antioch plaster casting process. In resin shell moulding, 1% of ammonium borofluoride will successfully act as an inhibitor (1).

The jointless method i.e precision casting process, has been successfully applied to magnesium when borofluorides have been added to gypsum bonded investment slurries to act as inhibitors. Pellegrini (5) and Herrick (6) successfully used a mould made up of gypsum–bonded refractory investing slurries inhibited with borofluoride compound in their studies on magnesium alloys. However, Emley stated that in many instances no inhibitor is employed since the mould is fired at red heat before use and there is a reasonably fast rate of solidification of the metal (1).

2.5.7 Pouring methods

Before the pouring operation, the lip of the crucible, the rim, lugs etc. must be cleaned to remove any encrusted flux. The flux cover should then be carefully drawn away from the pouring lip and neighbouring walls, using a flux spoon or skimmer. The exposed metal surface should be prevented from oxidizing by applying flowers of sulphur, dusted lightly. The aim in using sulphur should be to prevent rather than extinguish oxidation and consequently sulphur application should begin before the flux cover is first broken by the skimming tool. The skimming should be done smoothly and the flux cover drawn or pushed gently across the surface metal, not moved along in a series of rough jerks with the skimmer breaking the metal surface at each stroke.

Cleaning the metal surface immediately before pouring is important, for it is at this stage that the flux inclusions are most likely to originate through indifferent practice.

If, when the melt is ready for pouring, the flux cover is for any reason abnormally fluid or abnormally 'dry', correspondingly more care will be needed in cleaning the metal surface before pouring.

2.5.8 Safety Precautions

The usual precautions for handling molten metal are to use face shields and fire proof clothing.

Moisture, as for any other molten metal, could be a threat to safety in handling molten magnesium. It can cause explosion and fire. Therefore to keep moisture to a minimum, the following must be observed (23):

i. since fluxes are hygroscopic materials, they should be kept/stored in airtight containers;

ii. all ladles, thermocouples, sludge pans and other related tools should be cleaned and should be thoroughly preheated to drive off any moisture;

iii. metals to be charged i.e scrap and ingots must be clean and dry.

A serious explosion is likely to result if molten magnesium comes in contact with iron oxide. Minimising scaling of the steel crucible will reduce the danger of explosion, because scaling may lead to crucible failure, which in turn may lead to molten magnesium dropping down onto the scale in the furnace which may set off an explosion.

Inspection of the crucible at regular intervals is recommended to ensure the thickness of the crucible walls is sufficient to avoid breakthrough. The frequency of inspection may vary depending on the type of usage, quality of crucible used, fuel and operating temperature. An inspection schedule should be established to ensure that the condition of the crucible is known at all times.

The location of the main valves which control the flow of the fuel and air to the burners should be located at some distance from the furnace. This will permit shutting off the furnace in the event of crucible failure without endangering the operating personnel.

2.6 MAGNESIUM ALLOY CASTING TECHNIQUES

Magnesium alloy castings can be produced by nearly all of the conventional casting methods, namely sand, shell, investment, permanent and die casting. The choice of casting method for a particular part depends upon factors such as configuration of the proposed design, the application, the properties required, the total number of castings required, and the properties of the alloy.

2.6.1 Sand Casting

Sand castings can be produced in a wide range of magnesium alloys. Castings weighing as much as 1400 kg cast weight can be produced. Although sand casting has certain limitations, it is economical because the sand can be reused repeatedly.

The sand mould is bonded by natural clay activated by the addition of water. Diethylene glycol is frequently added to the mix in order to minimize the amount of water necessary to prevent the sand from drying out. A small percentage of inhibitor such as potassium fluoborate, boric acid, or sulphur (usually at an amount of 2% of the sand weight) is added to prevent metal–mould reaction. For large and heavy sectioned castings sulphur addition can be increased to 4 to 8 % (2). To reuse the sand, it is only necessary to restore the moisture, inhibitor and glycol content and then to mull the sand mixture.

In magnesium base alloy casting, a shrinkage allowance of 1 to 2% is added to dimensions to compensate for the shrinkage of the magnesium alloy during solid state contraction (41). The solidification time of the melt in sand moulds is longer in comparison with dies. The metal structure is therefore coarser and the mechanical properties are inferior to those of pressure die castings.

2.6.2 Permanent Mould Casting (Gravity Die Casting)

Casting of magnesium in permanent moulds should be considered when permitted by part design and when the cost of permanent mould equipment is justified by the number of castings required. In general, alloys that are suitable for casting in sand moulds can also be cast in permanent moulds.

With good foundry technique, permanent mould castings are equal in strength and performance to their sand cast counterparts. Compared to sand casting, permanent mould casting provides better surface finish and permits closer control of dimensions from casting to casting. If the part to be made is subject to frequent redesign, however, it would be uneconomical to use permanent mould casting.

2.6.3 Die Casting (Pressure Die Casting)

Die casting of magnesium alloys offers the same advantages as for other metals. The process has sometimes been used for making a few castings because of some special requirement, but die casting of magnesium alloys is usually applied to high–volume production. The size of magnesium casting that can be produced in a die is subject to the same limits as aluminium alloy die castings.

An important advantage in magnesium die casting is that molten magnesium does not weld onto the die in the same way that aluminium does. Therefore, the expensive dies last much longer. Dies may last two to three times longer than for aluminium.

The number of alloys that can successfully be cast by this process is much more restricted than is the case with sand castings and the most frequently used alloys for die castings are AZ91A and AZ91B. These two alloys are identical except that up to 0.35% copper is permitted in AZ91B.

2.6.4 Investment Casting

The investment casting method is used for casting magnesium alloys when the products are complex in nature, demand very thin wall sections, fine surface finish, and require tight dimensional tolerances. The same alloys can be cast by this process as with sand casting. The processes used are mainly 'jointless' in character and use waxes as patterns. This gives a general improvement in dimensional accuracy by the elimination of the parting line.

The disadvantages of the investment process are the cost in terms of both capital equipment and production cost per casting. There is also a much greater restriction on the size of the casting that can be produced. Further elaboration on the process can be found in chapter three.

2.7 INSPECTION AND TESTING

2.7.1 Tensile Testing

The engineering tensile test is widely used to provide basic design information on the strength of materials and as an acceptance test for the specification of materials. Analysis of the test results provides data about elastic and plastic deformation, modulus of elasticity or Young's Modulus, Poisson's ratio, yield and tensile strengths, elongation and reduction in area, and stress–strain behaviour. These data are useful for quality control in production, for ranking performance of structural materials, for evaluation of newly developed alloys and for dealing with the static strength requirements of design. However, before any mechanical test results can be interpreted properly, two basic questions have to be addressed, i.e :

1. how to assess the mechanical properties of complex casting shapes, and

2. what are the factors affecting the mechanical properties.

The results of any mechanical property test are influenced by the thermal/mechanical history of the test specimen and the details of specimen shape and applied load. Other factors were also found to influence the mechanical properties of magnesium alloy castings (42).

Standardized tensile testing methods for magnesium alloys have been developed under the auspices of ASTM and are published in the Annual Book of ASTM Standards. In the test procedure, a specimen is subjected to a continually increasing uniaxial load (force), while simultaneous observations are made of the elongation of the specimen.

2.7.2 Metallography

Magnesium alloy specimens chosen for metallography examination should be representative. Several parts of the cast component should be examined in order to give a true representation of the specimen. The coupon cut from a casting to represent its metallographic structure should be selected taking into consideration the casting wall thickness, mould design and location of chills, gates and risers. For example, for a large casting, observation should be made at the edge and centre of the casting.

2.7.2.1 Specimen Preparation

In metallography, several procedures have to be observed before the actual examination of the metal structure can be conducted. The preparations involve:

1. Specimen sectioning

Specimens are removed from the metal mass by band sawing or hacksawing. Care should be exercised to prevent cold working of the metal, which can alter the microstructure and complicate interpretation of constituents.

2. Mounting

Specimens that are too small to be held conveniently for grinding and polishing or that have an edge that is to be studied can be mounted in one of the common plastic mounting materials. Cold mounting materials are preferred, because the use of mounting materials that require pressure, such as Bakelite, may result in cold working of the specimen.

3. Grinding.

Grinding is designed to remove the disturbed metal from the face of the metallographic sample and to prepare a flat face that can be polished later. The surface of the specimen to be examined is ground, either dry or wet, on rotating abrasive belts or discs, or by rubbing the specimen across abrasive papers by hand. Abrasives commonly used are aluminium oxide, silicon carbide, or emery, that are bonded either to a paper or cloth back. Water–proof papers must be used for wet grinding

For dry grinding, the specimen must not overheat during grinding; overheating will affect the microstructure and can produce a separation between the plastic mount and the metal specimen. The sample is normally ground on nos. 50, 150 and 320 aloxite, and no. 0 emery discs rotating on vertical wheels. An oil bath below the disks catches the grinding swarf. The sample is rotated 90 degrees between steps and is ground at least twice the amount necessary to remove the scratches from the previous wheel. Pressure is applied to the trailing side of the specimen to keep the surface parallel and flat.

Wet grinding is preferable because it prevents overheating of the specimen and maintains exposure of the sharp edges of the abrasive. A conventional 200 mm diameter horizontal polishing wheel and discs of 240–, 320–, 400–, and 600–grit silicon carbide paper are often used for rough through fine grinding. A small stream of water run onto the disc flushes away the abraded fragments. As with dry grinding, the sample is rotated 90° between steps.

4. Polishing

Polishing operations can be mechanical, chemical or electrolytical. Mechanical polishing is performed in two stages: rough and finish. Rough polishing removes the major part of the disturbed metal remaining after the final grinding step. Finish polishing removes the superficial scratches that remain after rough polishing. Chemical polishing is not as effective as mechanical polishing for bringing up details, but it is satisfactory for routine examination. Before chemical polishing, the specimen is ground with 000 emery paper or 600–grit silicon carbide paper. It is then swabbed for 30–60 seconds with approximately 10% nital. Chemical polishing is done with a slow–acting chemical polish, usually glycol etchant with 1% concentrated nitric acid. Following polishing, the specimen is etched as usual.

Electrolytic polishing is used when a scratch–free surface is desired for critical examination without etching. It is useful for polishing large pieces. For polishing magnesium alloys, a conventional setup and a stainless steel cathode are used. The electrolyte consists of three parts 85% phosphoric acid (H_3PO_4) and five parts 95% ethanol; both are cooled to 2°C before mixing. Prior to electrolytic polishing, the specimens should be ground through 000 emery paper using a kerosene and paraffin mixture as a lubricant, and then cleaned.

2.7.2.2 Etchants

Composition, physical condition and temper of the specimen will determine the type of etchant used and the etching time required. For instance, an alloy in the as–cast or aged condition may take 5 to 10 seconds to etch whilst the solution heat–treated alloys may need 60 seconds.

For sand, permanent mould and die cast alloys in the as–cast condition, and virtually all the alloys in the aged condition, the glycol etchant is used. This is useful for the magnesium/rare earth alloys. Acetic–picral etchants are used to stain the grains especially for viewing with sensitive–tint or polarized light.

For cast metal in the solution–heat–treated condition, the glycol, acetic glycol, and acetic–picral etchants are satisfactory. To reveal the grain structure of alloys containing zinc and zirconium, a mixture of 48% HF, HNO_3 and H_2O may be used. A phospho–picral etchant with 6% picral, darkens the magnesium solid and leaves the other phases unchanged. The saturation of the solid solution will determine the amount of staining developed. This echant is particularly useful for quick estimation of the amount of undissolved second phase in solution–heat–treated metal, because of the extreme contrast it produces between the darkened matrix and the unetched second phase. Phospo–picral etchant is also useful for resolving grain boundaries and lamellar precipitate.

2.8 SUMMARY

Magnesium–base alloy is known to be the lightest metal and exhibits the potential to replace traditional materials used in automobile, aerospace and engineering industries. Despite the advantages displayed by magnesium alloys, special attention needs to be paid when handling the alloys. During melting and pouring, magnesium needs a protective layer on its surface formed by applying flux with a fluoride or chloride base or by using a protective inert atmosphere in a fluxless method. When casting into a mould, some protective substance or inhibitor must be included in the mould in order to suppress any mould–metal reaction.

Many of the casting processes that were developed to be used on other metals seem to suit magnesium alloys without many problems. They are sand casting, permanent mould casting, die casting, investment casting, etc.

CHAPTER 3

THE INVESTMENT CASTING PROCESS

3.1 INTRODUCTION

Investment casting, has endured due to the many advantages it offers. Close tolerances, intricate casting capability, minimal machining and superior surface finish explain its survival until the present from its beginning in the Bronze Age.

It has also been called the lost wax process , but the preferred name is investment casting. ' Investment ' implies the forming of an outer layer or envelope around a pattern. The pattern (usually of wax or plastic) is removed from the envelope through melting or burn–out leaving the mould cavity. Metal is then poured into the cavity to form the casting.

There are two alternative processes in use for the production of investment castings and, although the ceramic shell process is used predominantly throughout the industry, the block mould technique is also employed providing versatility in the range of alloys cast and, in many cases, a more precise component.

In both cases the manufacturing cycle commences with the production of an expendable pattern, usually wax, from a pattern die. The patterns are mounted onto a runner system to form an assembly, which in turn is invested with a fine coating of refractory slurry and stuccoed with refractory particles. Successive coats of refractory are applied until a complete shell has been formed around the assembly.

With the block mould technique, the bulk of the mould is produced in one operation by pouring a refractory slurry around the assembly contained by a metal flask, to produce a solid mould. In each case, the mould is dewaxed and then fired to induce strength. Molten metal is then poured into the hot mould and, after cooling, the refractory mould material is removed. The casting is then cut from the runner system and processed in accordance with the customer's requirement. The preparation of a shell mould can also be conducted by an electrophoresis method (46).

In summary, the investment casting process is the production of castings using an expendable pattern and entails six steps from start to finish (43–45), i.e,

- *pattern production*
- *pattern assembly*
- *ceramic investment*
- *dewaxing / firing*
- *casting*
- *cleaning*

Although investment casting is one of the most ancient metal forming crafts, it remains an important technique for producing premium quality castings, particularly complex shaped and thin walled components. The principal advances (33) in modern investment casting can be summarised as follows:

i. the coupling of geometrical precision with control of the microstructure of the end product in order to achieve improved and reproducible mechanical properties;

ii. the increase in complexity, precision and size of casting produced while maintaining improvements in performance;

iii. the extension of investment casting to a wide range of ‘difficult’ alloys which cannot effectively be produced by thermomechanical processing;

iv. the maintenance of a cost advantage over rival production techniques.

The investment casting industry has evolved a wide range of procedures and techniques to achieve the properties demanded by designers. In many applications an investment cast product is superior to its closest forged equivalent, as for gas turbine blades (33). In other cases, economic benefits result from near net shape processing with equivalent service performance. The first priority in all the developments is to minimize the occurrence of casting defects that can limit service performance or introduce unpredictable variability in component behaviour

In addition, positive control of aspects of the microstructure has enabled materials to be tailored to specific casting applications. However, the measures required to achieve these individual objectives are often incompatible and the final process is necessarily a compromise to obtain optimum performance.

3.2 ADVANTAGES AND LIMITATIONS

The investment casting process displays the many advantages (23) (44) listed below:

1. The process permits mass production of complex shapes that are difficult or impossible to produce by the more conventional casting processes or by machining.

2. The mould materials and the technique used permit reproduction of fine details, greater dimensional accuracy, and smoother surfaces than can be obtained by other casting processes. This enables costly finishing operations to be reduced to an absolute minimum and so facilitates savings in materials, labour and expenditure on capital investment.

3. The process is adaptable to almost any metal that can be melted and poured. Castings containing more than one metal, such as an integrally cast steel hub in a copper rotor, are also feasible.

4. With the modification of the basic process, production of castings weighing up to 22 kg (50 lb) is not uncommon. The production of very large castings weighing as much as 450 kg (1000 lb) is sometimes feasible, although such castings are rarely produced by the investment method.

5. Castings can be produced that require little or no finishing for completion, thus minimizing the importance of selecting easy–to–machine metals.

6. The process permits close control of metallurgical properties such as grain size, grain orientation, and directional solidification, which results in close control of mechanical properties.

7. The process is adaptable to the melting and casting of alloys that must be poured in a vacuum or under protection of an inert atmosphere.

8. The castings do not vary in dimensions across parting lines, as do castings made by other processes. In fact, parting lines are normally eliminated because of the monolithic moulds used in investment casting (when a 'parting line' shows, it has been caused by flash on the pattern that was not removed before investment). The absence of a joint line improves freedom of design.

The process has some limitations (23) which are summarised below.

1. The size and weight of castings that can be produced by the process is usually limited by physical and economic considerations, as well as by the capacity of available equipment. In general, the process can be applied most advantageously to castings weighing not more than 5 kg (10 lb).

2. The initial tooling costs for larger (5 kg to 22 kg) castings are generally high.

3.3 CLASSIFICATION OF THE INVESTMENT CASTING PROCESSES

Two distinct processes, differing in method of mould preparation, are used in the production of investment castings – namely, the *shell investment process* and the *solid investment process*.

3.3.1 Ceramic Shell Investment Casting

Ceramic shell moulds are used primarily for investment casting of carbon and alloy steels, stainless steels, heat resistant alloys, and other alloys with melting points above 1087°C (2000° F). The moulds are built up by dipping pattern assemblies into slurries of ceramic powders suspended in a liquid. The excess is drained and the wetted surface is stuccoed with dry refractory grain which is done either by sprinkling in a rainfall sander or by immersing in a fluidized bed, and the resultant coating is then dried. This process is repeated until the required mould thickness is achieved. The number of coatings applied depends on the required thickness of the shell. The thickness of a ceramic shell depends mainly on the casting shape and weight, cluster size and type of binder and ceramic. To some extent, shell thickness also depends on the temperature and humidity of the environment in which the shell is made. Usually the thickness of the shell ranges from 6.4 mm to 12.7 mm (1/4 to 1/2 in) (23).

The initial coat or the precoat, usually employs a slurry that is made up of finely ground particles, to provide a smooth surface. The finished surface of the casting produced largely depends on the smoothness of the precoat layer. Subsequent coatings usually contain increasingly coarser refractory grains.

Ceramic shells are dried or cured after each dipping operation, to allow bonding of the individual layers. In drying, the environmental factors, defined by Twarog (47) as the temperature, humidity and air velocity must be carefully controlled. This is because they will affect the drying characteristic of the shell. To some extent, the shape of the casting also exerts a similar influence to the three former factors mentioned (23). Inconsistency in the temperature of the surroundings will create an internal stress potential between the wax and the shell, which arises from the different thermal expansion of both materials. High air flow can cause undesirable fast drying on the edge of the pattern, which prevents stucco from adhering to the coated pattern edges. In the case of low humidity, uneven drying in the holes and slots compared to exposed sectors may be experienced.

Final drying and bonding of the shell usually occurs during the dewaxing operation and during the firing of the mould before casting. Shells that have been completely fabricated can be dewaxed after 8 hours but generally, a minimum of 24 hours is allocated for drying prior to dewaxing (23).

3.3.2 Solid Investment Casting

Generally, the solid investment process can be divided into 2 basic processes, due to its application, i.e for the non–ferrous alloys and for the ferrous alloys. The two processes differ primarily because of the effects of pouring temperature on the bonding materials and refractories.

3.3.2.1 The Solid Investment Process for Non–ferrous Alloys.

This process is used particularly for non–ferrous alloys which have a pouring temperature below 1087°C (2000° F) (Aluminium–base alloys and many copper–base alloys). The mould material generally consists of alpha gypsum , which serves as both a binder and refractory, and a blend of other refractories, such as various grades of silica, to control mould permeability. The refractories used are fine–grained, and precoating is not required.

The pattern assembly that has been prepared, is placed on a plate and encircled by a metal flask that is open at both ends. The investment slurry is poured directly into the flask, which is vibrated or subjected to vacuum or both, to remove bubbles. The slurry is then permitted to set by chemical reaction.

Alpha gypsum (calcium sulphate hemihydrate) is a high–strength and relatively low cost product that makes use of water as the liquid vehicle. A typical gypsum–bonded material contains about 35.5% cristobalite, 29% silica flour, 33.5% calcium sulphate, and 2% of a highly absorbent diatomaceous earth, to provide permeability (23). Small amounts of commercial accelerators and retarders may be added to control setting and thermal expansion. Commonly used accelerators are sodium chloride, potassium sulphate, and powdered calcium sulphate dihydrate. Citric acid and sodium citrate are used as retarders.

The recommended consistency for the investment slurry is determined by the ratio of water to powder. Small variations in water content will produce large variations in properties of the investment. Therefore, the proportion of water and powder must be accurately measured.

3.3.2.2 The Solid Investment Process for Ferrous Alloys.

Mould materials for investment casting of ferrous alloys, and non–ferrous alloys with high pouring temperature, must be capable of withstanding temperatures above 1087°C (2000° F) without deteriorating. Therefore, gypsum is not a suitable bonding or refractory material. Precoating is a standard practice in the preparation of moulds for ferrous castings. Precoating patterns with fine–grain slurry makes it possible to use a coarser grain backing slurry, which is less expensive, without affecting surface finish.

The slurries used for precoating patterns are based on binders such as sodium silicate, ethyl silicate, or the most widely used binder–colloidal silica. Precoats must be properly dried or cured. High humidity during fabrication, for instance in excess of 80%, will reduce the strength of the precoat. Insufficient drying will weaken the coat, and sloughing off into the casting is possible. This will result in dirty castings.

Control of stuccoing following the precoat is essential. A coarse stucco will give less trouble compared to a finer one (150–mesh and finer), since it will permit more open spaces for better adhesion of subsequent layers (23). A fine grain produces a more uniform surface but prevents optimum bonding of subsequent coats. Matzek in his paper (48) recommended that an intermediate coat with stucco size of either a –30+70 mesh or a –40+80 mesh be used. This is to minimize the potential for: metal breakthrough, on those parts which have slots; edge wash at sharp corners or radii; and shell delamination, which is due to differences in the expansion coefficients of the different refractories.

During casting, buckling may happen which is due to the difference in thermal expansion between the precoat and the back-up investment, whereby the precoat expands more than the latter. To avoid this, i.e reducing the thermal gradient between the coats, milled zircon may be added to the silica flour precoat slurry, in the mixture of one is to two parts by weight (23).

3.4 CERAMIC SHELL VS SOLID INVESTMENT CASTING

The choice between the shell investment process and the solid investment process is based on :

i. casting size

ii required tolerance

iii. composition of metal poured

iv. cost incurred, and

v. availability of equipment.

Many hollow castings or castings with large cored passages can be produced without ceramic cores, and hence at less cost, by the solid mould process. Some castings that require complex ceramic cores cannot be made by the solid mould process, partly because of mould growth and core shifting.

More mould material is required for solid moulds than for shell moulds. For small castings, the cost of the mould material per casting is low compared to other costs, and the the solid mould technique is often less expensive. As the casting size increases, the cost of the mould per casting also increases.

3.5 INVESTMENT CASTING TECHNOLOGY

3.5.1 Patterns

The expendable pattern materials used in the investment casting process are waxes and plastics (usually polystyrene). The latter is a thermoplastic material that vaporises at mould firing temperature. Both of these materials are considered to be expendable or consumable since the pattern is used up, or lost, when forming the mould cavity where the part is shaped. Various materials have been tried with mixed results, but wax and polystyrene continue to be preferred (29)(49).

Of these two main materials, waxes are the most widely used. Pattern waxes are complex mixtures composed of relatively sophisticated, high priced ingredients that have been formulated and blended to make the best wax pattern possible. Pattern wax should be considered a valuable raw material that is the starting point for the production of an investment casting. The higher the quality of wax patterns produced by the wax room, the higher the quality of the casting produced.

Dense polystyrene may be used for producing patterns, or parts of patterns, that require strength higher than that conferred by wax. Foamed polystyrene may be used in the Replicast Ceramic Shell (CS) process and in the evaporative pattern casting process.

3.5.1.1 Waxes

Wax is the oldest thermoplastic material known to man. It can be cast or formed while in a liquid, semi–solid or plastic state. The term wax, especially in the investment casting industry, can refer to any substance having a wax–like property. Hence modern blends of pattern waxes are compounds containing numerous components such as natural hydrocarbon waxes, natural ester waxes, synthetic resins, organic filler materials and water (50). Many variations of such compounds have been formulated to suit various requirements.

As was categorised by Chambers and reviewed by Clegg (51), waxes fall into the 3 main categories described below.

1. Natural Ester waxes

Natural ester waxes are used in small amounts, generally up to 10% of the total formulation, to increase the hardness of the blend and accelerate the setting–off process when the wax cools (52). They are often high in price, possess inconsistent properties and high ash content. Natural waxes used at present are mainly carnauba and candelila both of which are very hard, and are extracted from plants. Beeswax is also being used but in small quantity due to its softness and high price.

2. Natural Hydrocarbon waxes

Natural hydrocarbon waxes are those derived from petroleum crude oils. The most commonly used in investment castings are paraffin waxes and microcrystalline waxes. They are used either singly or in combination, to achieve the required properties. The former, normally are unsuitable for use solely with resins as they can be incompatible with some of them and a very crumbly pattern would be obtained. The incompatibility can be reduced by using microcrystalline waxes which are much less 'cheesy' and crumbly than paraffin waxes and generally have higher melting points.

Thus a blend of paraffin and microcrystalline waxes, together with resin, is the basis of most casting waxes. The resin reduces the contraction of the blend and also gives it hardness. The microcrystalline wax will prevent the wax blend from being crumbly and can also give some flexibility if desired.

3. Synthetic waxes

Synthetic waxes are made synthetically from petroleum type raw materials but possess many of the properties of natural waxes. In addition, they do not have the purity problems associated with natural waxes and their properties are consistent since they can be checked and controlled during manufacture.

The final item that is often present in a basic wax blend is a small amount of polymeric material. An addition of up to 5% of this material is sufficient to increase the viscosity of the wax blend to suit the injection pressure necessary to inject the wax into a mould (52). If an appreciably larger amount of polymer is used, the wax will have a very high viscosity which would make it difficult or impossible to inject at a reasonable temperature.

Base waxes (waxes containing paraffin wax, microcrystalline wax, resin, possibly natural or synthetic wax and possibly polymeric material) are sometimes called unfilled waxes. A filler may be added, at a loading of anywhere between 10% and 50% of the total formulation, to reduce and control contraction in the wax pattern.

3.5.1.2 Plastics

Next to wax, plastic is the most widely used pattern material. Usually a general–purpose grade of polystyrene is preferred. Polystyrene and other plastics are able to be moulded at high production rates on automatic equipment and are resistant to handling damage, even in extremely thin sections. Polystyrene is very economical and can be stored indefinitely without deterioration.

A disadvantage that limits the use of polystyrene is its tendency to cause mould cracking during pattern removal. In addition, tooling and equipment cost is more expensive compared to wax.

3.5.2 Pattern Assembly

Wax components can readily be assembled by wax welding using a hot spatula or small gas flame. The wax at the interface between two components is quickly melted, and components are pressed together until the wax resolidifies. The joint is then smoothed over. A hot melt adhesive can be used instead of, or in addition, to wax welding. In manual wax welding, fixtures are important tools to ensure accurate alignment in assembling patterns. Patterns must be equally spaced and aligned. Joints must be completely sealed with no undercuts. Above all, care must be taken to avoid damaging the patterns or splattering drops of molten wax on them.

Welding of wax patterns is also possible using a CO_2 laser (53). It was reported that with this technique strong, uniform welds with good surface quality can be obtained.

3.5.3 Shell Preparation

Investment casting moulds are made by applying a series of ceramic coatings to the pattern clusters. Each consists of a fine ceramic layer with coarse ceramic particles embedded in its outer surface.

A cluster is dipped into a bath of slurry of fine ceramic material, withdrawn and manipulated to drain off excess slurry to produce a uniform layer. A coating of stucco is applied to the wet slurry, usually in a fluidized bed or rainfall device. The process is repeated until a self-supporting structure or shell is obtained. Usually when the coating is 5 mm to 16 mm ($^3/_{16}$ to $^5/_8$ ins.) thick (54). The final coat, sometimes referred to as a seal coat, is usually unstuccoed in order to avoid the occurrence of loose particles on the mould surface.

Each coating is allowed to set before the next one is applied. This can be done by drying, chemical gelling or a combination of both. Careful control of temperature, humidity and air flow is a vital task to ensure the desired result is achieved (47)(55). Humidity is critical in stabilizing the wax and shell and indirectly preventing cracks. Likewise, constant temperature is crucial in maintaining relative humidity and preventing contraction and expansion of the pattern and shell. To promote a constant rate of drying, air flow over the shell is usually controlled with some type of ventilating system.

3.5.4 Binders

The refractory materials must be bound together in a matrix that includes chemical binders such as ethyl silicate, sodium silicate, or colloidal silica (29). Hybrid binders have also been developed as well as alumina or zirconia binders which are used for some processes.

3.5.4.1 Colloidal Silica

Colloidal silica is the most widely used binder. It is a suspension of discrete particles of amorphous silicon dioxide (SiO_2) in water. Colloidal silica particles range in size from 6 to 100 nm. For each gram of colloidal silica the surface area is between 100–400 square metres. Colloidal silica is manufactured by removing sodium ions from sodium silicate.

The product consists of a colloidal dispersion of virtually spherical silica particles in water. The dispersion is stabilized by an ionic charge, which causes the particles to repel one another, thus preventing agglomeration. The stabilizing ion is either sodium (up to 0.6%) or ammonium, both of which are alkaline.

The outer layer of each particle has a layer of hydroxyl (OH) ions. As water evaporates from the system, the particles are forced together. The negative charge of each particle is overcome and a stronger attractive force between the hydrogen (H) of one colloidal particle and the oxygen (O) of another particle form a hydrated silanol bond which links each colloidal particle to the other.

The hydrated silanol layer provides the green strength needed while the ceramic mould is drying. These silanol groups convert to the more stable Si–O–Si siloxane when heated above 350°C (400°F). The last trace of water is believed to be eliminated at a temperature above 1100°C (1400°F). This explains why the fired shell will be strongest when fired above 1100°C (56).

The most popular grades of colloidal silica used are sodium stabilized with 30% silica content and with an average particle size of between 7 and 12 nm. They are used either at the 30% level or diluted with water, which reduces the silica content to between 18 and 30%. Coherent gels having excellent bonding properties are formed by adding ionic salts that will neutralize the ionic charge. This can also be done by concentrating the sol (as by drying a coating).

In order to optimize the use of colloidal silica, various factors that will affect its stability must be understood and taken care of. This includes the pH modifiers, soluble salts, alcohols and freezing (56). Through experiments it was found that the maximum unfired strength of a shell is a function of the alkali content of the silica sol and not its particle size (57).

With colloidal silica an excellent surface can be obtained and, if kept covered when not in use, to prevent evaporation of the liquid, slurries can be kept usable for several days or even weeks. The disadvantage of using colloidal silica is that its water base makes it slow drying, especially in inaccessible pockets or cores.

3.5.4.2 Ethyl Silicate

Another type of binder which has wide application in investment casting is ethyl silicate. Ethyl silicate is produced by the reaction of silicon tetrachloride with ethyl alcohol (29).The basic compound formed is tetraethylorthosilicate ($Si(OC_2H_5)_4$). The grade used for the investment application is designated ethyl silicate 40. It consists of a mixture of ethyl polysilicates and contains 40% silica.

By itself ethyl silicate has no bonding properties but by reaction with water (hydrolysis), it is converted to ethyl silicate binder.

Ethyl silicate with its alcohol base dries much faster than colloidal silica. It is, however, more expensive and poses a fire and environmental hazard. Ethyl silicate slurries are readily gelled by exposure to an ammonia atmosphere. This will permit dips to be applied quickly, however, proper drying can also be achieved without the presence of ammonia since the alcohol content in ethyl silicate is highly volatile.

Some technological improvement has also been observed in the binding media for lost wax moulds. Lost wax moulds can now be developed using water in substitution for organic binder (58). The binder with water is explosion and combustion safe and this technology has been adopted by automobile engine plants.

3.5.5 Refractory Materials

The most common refractory materials for ceramic shell moulds are siliceous, for example fused silica, zircon, alumino–silicates and alumina. Other refractories such as graphite, zirconia (ZrO_2) and ytrria (Y_2O_3), have been suggested for use with reactive alloys.

Silica is generally used in the form of silica glass (fused silica), which is made by melting natural quartz sand and then solidifying it to form glass. It is crushed and screened to form stucco particles or ground to powder for use in slurries. It has a low coefficient of thermal expansion which enables the mould to resist thermal shock. It is readily soluble in molten caustic and this characteristic provides a means of removing shell material chemically from areas of the casting that are difficult to clean by other methods. It is reasonably good for casting most iron–base alloys and some cobalt–base alloys that are air melted. Under vacuum silica breaks down in direct contact with metal, and may cause serious metal–mould reactions (23).

Silica is also used as naturally occurring quartz. However its utility is limited due to its high coefficient of expansion and by the high abrupt expansion at 573° C (1063° F) (29). As a result, shells containing quartz must be fired slowly.

Zircon (zirconium orthosilicate) is probably the refractory most widely used for ceramic shell moulds. It occurs naturally as sand and is used in this form as stucco. It provides good surface quality, high temperature stability, good refractory properties and good resistance to metal attack. Due to its particles' spherical shape, zircon has the lowest surface area and the least number of contact points. Thus, pattern coverage is good but strength is fairly low because of poor point–to–point contact. It is generally limited to use with prime coats because it does not occur in sizes coarse enough for stuccoing back–up coats. It is ground to powder (sometimes calcined) for use in slurries and is often used in conjunction with fused silica or alumino–silicates. It was found that a fused silica system of coating is able to withstand the greatest stress to breakage which means that it produces the stiffest shell with the best stability when compared with alumino–silicate systems (59).

Zircon shell castings are more difficult to clean than those produced in silica shells. Due to its relatively high thermal conductivity, misruns may occur in thin sections (23).

Alumino–silicates for investment casting are made by calcining fireclays or other suitable materials to produce a series of products ranging in alumina content from about 42 to 72%, with the remainder being silica plus impurities (29).

Refractoriness and cost increase with alumina content. The only stable compound between alumina and silica at elevated temperature is mullite ($3Al_2O_3.2SiO_2$), which contains 72% alumina. Mixtures containing less than 72% alumina produce mullite plus free silica which is usually in the form of silica glass. As the alumina content increases, the amount of mullite increases and free silica decreases until at about 72% alumina, the material contains only mullite. Fired pellets of these materials are crushed or ground and carefully sized to produce a range of powder sizes for use in slurries, and granular materials for use as stuccos.

Alumina is little used due to its poor resistance to thermal shock. However, it is sometimes desirable because it can be used up to 1739°C (3200° F). It is more refractory than silica or mullite and is less reactive toward many alloys than the siliceous refractories (29).

3.5.6 Slurry Formulation

The actual percentage composition of ceramic shell slurries depends on the particular refractory powder, type and concentration of binder, liquid vehicle and desired slurry viscosity (23).

The slurries used for the primary and secondary dip coats contain a fine refractory powder and liquid silicate binder as the principal ingredients. Other ingredients, such as wetting agents , antifoam compounds and strength enhancers may be present. The wetting agent is usually confined to colloidal silica primary coats and is added in an amount between

0.03 and 0.3% by weight of liquid. The antifoam compounds commonly used are aqueous silicone emulsions or liquid fatty alcohols, added in very low concentrations of 0.002 to 0.1% by weight. The use of antifoam compounds, normally in association with wetting agents, permits suppression of foam formation and promotes air bubble escape, especially from the pattern surface.

3.5.7 Slurry Preparation and Control

Slurries are prepared by adding refractory powder to the binder liquid. Sufficient agitation is necessary to break up agglomerates and thoroughly wet and disperse the powder. A prolonged period of mixing is required to promote deaeration and particle wetting. Even though homogenisation of the mixture has been achieved, continuous stirring is advisable, but at a reduced level, to keep the powder from settling out of suspension.

The most prevalent controls are the measurement of the initial ingredients and the viscosity of the slurry. The viscosity can be measured by using the no.4 and no.5 Zahn cups.

Beside viscosity, other parameters that are often controlled include, slurry temperature, density, pH value and percentage of SiO_2 (29)(60-62). The actual composition of the slurry in terms of water content, silica binder and refractory powder may also be determined but because it is time consuming it is reserved for trouble–shooting rather than control.

To control and maintain the life of ceramic shell slurries, the procedures described below have to be observed.

1. Use an on and off or a timer controlled, variable speed, propeller type mixer for slurry makeup tanks and working tanks that are non–rotating.

2. Use only deionized or distilled water in shell slurries. Tap water impurities could destabilise the slurry or cause premature gelling.

3. Control the pH of the soluble wax rinse tank. When an acid leach is used to eliminate soluble wax, the pH of the slurry and rinse tank should be controlled with reagent grade ammonium hydroxide (NH_4OH). To control the latter, NH_4OH is added directly to the tank whilst 0.5–1% by weight of reagent–grade NH_4OH solution is used to adjust the slurry's pH.

4. An addition of deionised or distilled water, to control viscosity and/or binder solid, should be made in a daily small amount rather than in one large addition every 3 days or so.

5. Avoid carry over of excess stucco material into the slurry tank which can cause inconsistent slurry control and lead to weak shells.

6. Keep the tank equipments, for example propeller shaft or tank sides, free from dried slurry build-up. Build-up can cause weak spots in a shell if they are broken off and create small turbulence zones which can introduce excess air into the slurry.

7. Control the binder solids in the pre–wetting tank at a level below that of the tank into which the pre–wet parts will be dipped. Excessive pre–wet carry–over will raise binder solids by replacing water lost by evaporation. This increase in binder solids can affect the slurry defoaming characteristics, slurry coating properties, and lead to premature gelling.

8. Avoid adding materials such as wetting agents and defoamers not recommended by the binder manufacture.

9. Never mix different binders, especially high–performance binders.

10. Document all slurry control results and additions.

Some properties of the finished ceramic shells can also be monitored to indicate the quality of the shells produced. This includes weight, modulus of rupture (green and/or fired) and permeability (60).

3.5.8 Coating and Drying

The patterns, which have been readily assembled in tree form, are dipped into the prime slurry and rotated to ensure uniform contact, withdrawn and drained over the slurry tank. The stucco particles are then applied by placing the cluster under a rainfall sander or a fluidized bed.

Generally, prime slurries contain finer refractory powder, are used at a higher viscosity and are stuccoed with finer particles than back-up coats. This will provide a smooth surfaced mould which is capable of resisting metal penetration.

Back-up coats are formulated to provide high strength and to build up the required thickness with a minimum number of coats. The number of coats applied is related to the size of clusters and the weight of the metal to be cast. For most applications the number of coats ranges from 6–9. With the new development in shell building, i.e through a combination of particle sizes stronger shell strength can be achieved thus permitting a reduction in shell thickness, which in turn reduces production time (63).

Two methods of hardening the slurries can be employed: air drying or chemical gelling. In the air drying process, which is the most common method employed, temperature, humidity and circulating air are the vital parameters to be controlled in order to achieve the required result. Drying is complicated by the high thermal expansion and contraction characteristic of waxes. Too rapid drying will cause a chilling effect which forces the pattern to contract while the coating is still wet and unbonded. As the coating develops strength and shrinks to a certain extent, the wax begins to regain temperature and expand. This can crack the coating. Relative humidity is normally kept above 40% or 50% for normal conditions (29). Dalgetty and Mills' work, reviewed by Atanmo and Piwonka (64), showed that both green and fired strength increase with the extent of drying. They also showed that the green strength of the mould is highest immediately after the shell build-up and decreases with drying time. Atanmo and Piwonka also reviewed the work of Hasley which showed that the green strength increases with the percentage of solvent removed and the possibility of cracking during dewaxing is reduced as a large amount of solvent is removed from the mould.

Atanmo and Piwonka, in their study on drying rate measurement, using an electrical resistivity method, showed that as the number of dips increases the complete drying time is increased. They also showed that flat surfaces dry faster than corners or crevices (64). Electrostatic drying has proved to be a successful method of improving the uniformity of drying. It has been found that electrostatic drying rates are equivalent to a forced air stream at 2.54 m/s (500 f/m) in the absence of a field (65).

In the chemical gelling technique, which is much more successful with the ethyl silicate binders, surface gelling is accomplished by exposure to an ammonia atmosphere. Even though the time taken for gelling is shorter, when compared to the air drying process, the shells must also be dried in order to gain strength. Failing to do so will result in a weaker shell being produced even when subsequently dried.

3.5.9 Dewaxing Operations

Dewaxing is a process of removing wax patterns from the ceramic shell moulds. However, the removal of the wax patterns presents a problem due to the different rates of expansion between the wax and the mould before the wax melts. The amount of expansion varies but generally the volume would increase by up to 10% (29). The pressure generated due to the increase in volume of the wax is sufficient to break the shell during the dewaxing operation. Increasing the strength of the shell to accommodate the pressure imposed by the increasing volume of wax would not be a good remedy since collapsibility of the mould would be poor and poor heat dissipation would result. This could lead to hot tears in castings.

Two methods can be employed to prevent mould breakage (29):

1. supplying external pressure to counterbalance the pressure generated internally due to wax expansion;

2. rapidly dissolving or melting the surface layer of the wax at the interface of the ceramic mould and the wax pattern. This would create a cavity into which the remaining wax could expand.

In the former technique, one of the methods is to place the pattern inside a container with the pouring cup being placed accurately over a ready made hole. Sand or other granular or powder refractory, heated to 326°C (600° F) is poured around the shell and the container is vibrated to pack down the refractory. The wax is melted by heat supplied by the heated refractory. The magnitude of the pressure applied to the shell depends on the depth to which it is buried and the packed density of the refractory surrounding it.

Wax patterns can also be dewaxed by the solvent method whereby the wax is removed by the action of the hot vapour of a solvent such as trichlorethylene. The vapour permeates the porous shell and immediately dissolves the wax faces adjoining the investment before the heat of the solvent vapour expands the wax. The shells are normally supported on a wire tray with the pouring cup down. During the latter stages of dewaxing, the wax patterns melt and run into the reservoir of the solvent. It should be noted that, if the penetration of the ceramic mould by the solvent is too slow, premature wax expansion can occur and will crack the shell.

Even though a number of methods have been developed for applying the surface melting method concept, only autoclave dewaxing and high temperature flash dewaxing have found widespread use (29)(66).

In autoclave dewaxing, saturated steam is used in a jacketed vessel which is generally equipped with a steam accumulator to ensure rapid pressurization because rapid heating is the key to success. Dewaxing depends on the latent heat of vaporisation of steam for its energy The autoclave must reach a pressure of at least 414 kPa (60 psi) within 10 seconds to be successful (49). A large instantaneous amount of energy, estimated about 454.8 kJ/kg of water at 100°C (970 Btu/lb at 212° F), is liberated as the superheated steam condenses on the cold shell surface, this dewaxes the shell without cracking. The operating temperature at that instance is about 174°C (320° F) and the average processing time is 10 minutes.

Flash dewaxing is carried out by inserting the shell into a hot furnace at 870°C to 1095°C (1600° F to 2000° F). The furnace is usually equipped with an open bottom so that wax can fall out of the furnace as soon as it melts.

By this method, a high thermal gradient is established across the ceramic shell–pattern section. This causes melting at the wax–mould interface before the main portion of the pattern is heated and begins to expand. The time interval for loading the shells to the furnace must be regulated so as to avoid heating the shells too slowly, because this will allow the wax to expand and thus crack the shells. Two major disadvantages of this technique are that it can easily cause fire and it generates smoke.

3.5.10 Mould Preheating and Firing

Preheat time for shell moulds ordinarily ranges from 30 mins. to 2 hours. The moulds, at room temperature, placed in a furnace maintained at 1033°C (1900°F), will require approximately 40 mins. soaking time before the moulds attain the furnace temperature (23).

Preheat temperatures vary over a large range above and below the firing temperature, depending on part configuration and the alloy to be cast. It ranges from 150°C to 540° C (300°F to 1000° F) for aluminium alloys to 870°C to 1095° C (1600°F to 2000° F) for steels and superalloys. Many moulds can be placed directly into the hot furnace, others may be loaded at a low temperature and heated gradually.

The length of the preheating cycle depends on the type of furnace used, the type of mould (solid or shell), the condition of the mould (dewaxed or not dewaxed), the amount of insulation of the mould and the preheat temperature (29).

Ceramic shell moulds are fired with the objects of (29):

1. removing moisture (free and chemically combined);
2. burning off residual pattern material and any organic material used in the shell slurry;
3. sintering the ceramic;
4. preheating the mould to the temperature required for casting.

In addition, moulds are also fired to (23):

1. permit filling of mould sections too thin to be filled in a cold mould;
2. minimize the size of risers;
3. minimize hot tearing.

3.6 SUMMARY

Investment casting or the lost wax process is a precision method of forming liquid metal and exhibits a unique and broad spectrum of advantages which other metal forming techniques do not offer . It is not a recent idea as it has been known since the Bronze Age. It is based upon the use of an expendable pattern and a ceramic mould to produce an accurate casting. Consisting of several operations, this process is suitable for ferrous and non–ferrous metals.

Investment castings are widely used for aircraft parts (both military and civil) and for general commercial applications.

CHAPTER 4

EXPERIMENTAL PROGRAMME

4.1 INTRODUCTION

This chapter describes the experiments carried out to meet the objectives outlined earlier. It consists of six sections. Section 4.2 describes the preliminary experiments conducted to identify the possible causes of shell investment mould cracking and the construction of an alternative mould used in some of the experiments. Section 4.3 outlines the experiments conducted to investigate the influence of atmosphere in the casting of magnesium alloy in Shaw process and shell investment moulds. The section also covers the tests to determine the minimum level of inhibitor gas required to suppress mould–metal reaction. Section 4.4 describes the investigation of the influence of pouring temperature on metal–mould reaction. It also includes the assessment of the effect of various pouring temperatures and inhibitor substances on the reaction. Section 4.5 describes the casting of tensile test specimens using various mould and pouring temperature combinations. Tensile test procedures adopted for assessing the mechanical properties of the specimens are also described. In a subsection, experiments to select control measures to reduce the variation of the results obtained and the recasting of tensile test specimens with improved melting control are presented. Finally, in section 4.6 the procedures for preparing the specimens for microstructural observation are outlined.

Preliminary experiments were conducted to obtain some idea of the effect of the temperature of the mould and the melt. The experiments were also meant to judge the strength of the shell built using the shell investment procedure and to assess the extent of the mould–metal reaction anticipated from preliminary reading. Due to breakages of the ceramic shell moulds during dewaxing and pouring in the preliminary experiments, Shaw

dewaxing – how?

process moulds were used as an alternative. This was to allow saving of materials and time in building shell moulds.

An experiment to investigate the cause/s of shell cracking was also conducted. The specific gravity of the slurry for building the moulds for these experiments was controlled to between 67–69 on the Baume scale. The first experiment was conducted by pouring magnesium alloy into two moulds at pouring temperatures of 800°C and 780°C respectively. The objective was to investigate whether the temperature of 780°C was the cause of the mould breakage since all the moulds in the preliminary experiment cracked at that particular temperature. A mould produced from a pattern with rounded corners was also used to investigate the effect of sharp corners on the 'strength' of the mould, pouring was conducted at a temperature of 800°C.

In addition, an experiment was conducted to establish the effect of varying the drying time after the final coat was applied. The experiment compared two drying times: the first involved leaving the shell to dry for 1 week and the other for 24 hours. Taking into consideration the quality of the wax used for the pouring cup, which might cause the cracking of the moulds, an experiment was conducted by building two moulds, each with a different grade of wax. The first was built with a degraded wax and the second was produced with wax of the same quality as the component pattern's wax. The degraded wax used in this experiment was that from dewaxed moulds.

To meet the objective of the project, i.e to overcome the mould–metal reaction, two approaches were taken: the first was the use of inhibitors; and the second was the casting of the magnesium alloy in different mould materials.

Since the pouring temperature was considered an important factor in the casting of magnesium, experiments to establish a 'suitable' pouring temperature were conducted. This was carried out by casting magnesium alloy at five different pouring temperatures i.e 780°C, 750°C, 730°C, 700°C, and 680°C.

4.1.1 The Use of Inhibitors

The investigation of inhibitor use to overcome the metal–mould reaction in magnesium alloy casting was carried out in two ways:

1. Using various percentages of potassium borofluoride (KBF_4);
2. Using a CO_2/SF_6 gas mixture.

In the investigation using KBF_4, two methods were employed: first, by mixing various percentages of KBF_4 with the mould material; and second, mixing KBF_4 with a coating medium subsequently applied to the interior face of the mould.

The CO_2/SF_6 gas mixture was used in two ways: by flushing the gas directly inside the mould cavity; and by flushing the gas within an enclosure surrounding the mould. These experiments were conducted using Shaw process moulds.

The use of the CO_2/SF_6 gas mixture was repeated on shell investment casting moulds to confirm its effectiveness and to determine the optimum amount of inhibiting gas required.

4.1.2 The Use of Alternative Mould Materials

In the second approach, the materials used were graphite, silicon carbide, calcium oxide, calcium carbonate, magnesium oxide and anhydrous calcium sulphate (gypsum). These materials were used to produce moulds, using Shaw process procedures where appropriate, which were cast at a temperature of 730°C.

4.1.3 The Effect of Process Parameters on Mechanical Properties

In the later stage of the investigation, an experiment to establish the influence of investment casting process parameters on the mechanical properties of cast magnesium alloy was conducted. The relationships were established by casting the alloy at various mould and pouring temperatures. The mould temperatures chosen for the investigation were 21 °C, 100 °C, and 200 °C; whilst the pouring temperatures were 700 °C, 730 °C, 750 °C, and 780 °C. Melting was conducted using the flux method.

To evaluate the mechanical properties of the cast magnesium alloy, the cast specimens were subjected to tensile testing. The main objective of the tensile testing was to establish the influence of mould temperature and pouring/casting temperature on the ultimate tensile strength, modulus of elasticity (Young's Modulus), and elongation. The test specimens were prepared by machining the cast specimens to the required dimensions (Hounsfield test bar no. 16 as shown in figure 4.7). An x–ray examination was also conducted to determine the quality of the castings.

Inclusions were found to be a major problem that affected the mechanical properties of the cast samples. In order to acquire better results, a series of experiments was conducted using mild steel strainers, melting with the fluxless method, and casting at a high mould temperature.

Finally, investment casting with alloy processed by fluxless melting was conducted. Tensile testing was repeated to establish the mechanical properties versus process parameters relationship.

The overall plan is summarised in the experimental plan flow chart shown in figure 4.1.

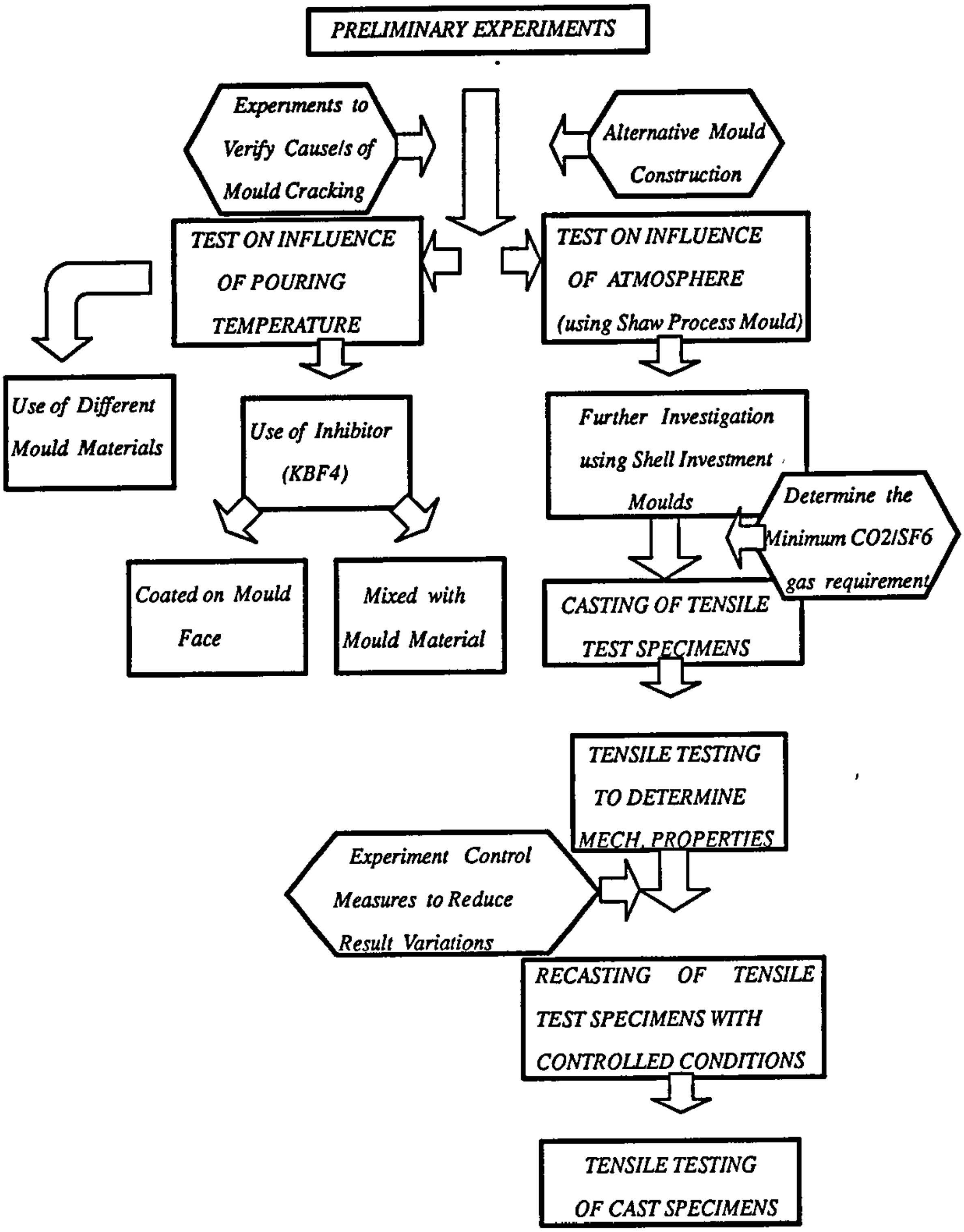

Figure 4.1 : Experimental plan flow diagram

4.2 PRELIMINARY EXPERIMENTS

Two batches of shell investment casting moulds (lost wax process) were prepared , each of which consisted of two control moulds (alumino–silicate alone), one mould mixed with 5% by weight KBF_4, one mould with 7% KBF_4, and one mould with 10% KBF_4.

The first batch of moulds was cast at a mould temperature of 500°C, with a temperature variation of ± 10°C, and the second batch at normal ambient temperature, ranging from 24.3°C to 25.7°C. Both batches were cast with a pouring temperature of 780°C ± 10°C.

The configuration of the pattern and the mould is shown in figures 4.2 and 4.3 respectively.

4.2.1 Verification of the Cause/s of Mould Cracking

A series of experiments was conducted to determine the cause/s of mould cracking. Factors considered included the quality of wax used, mould drying time and mould pouring temperature.

4.2.1.1 Pouring at Higher Temperature

In the investigation, three shell investment process moulds were prepared, two of which were of the original design (refer to figure 4.2) whilst the other was produced using a pattern with rounded corners. One of the former moulds and that with the rounded corners were poured at a pouring temperature of 800°C whilst the third was poured at 780°C. The experiment was repeated twice to confirm the result obtained. All of the moulds were cast in the presence of a CO_2/SF_6 gas atmosphere.

4.2.1.2 Extended Drying Time and Use of the same Quality of Wax for Pouring Cup

Two moulds were constructed using a pouring cup and component that were made from wax of the same quality . The moulds consisted of 6 layers formed by dipping the wax assembly into slurry of a viscosity ranging from 67–69 on the Baume scale. After applying the seal coat, the moulds were left to dry for 1 week. The moulds were then dewaxed at a temperature of 175°C.

4.2.1.2.1 Extended Drying Time After the Final Coat

In this experiment , the time of drying after the final coat (seal coat) was applied was varied. Two moulds were constructed with 6 layers of coating and a single seal coat. One of the moulds was left to dry for a period of 1 week while the other was left for 24 hours. Both moulds were dewaxed at a temperature of 175°C.

4.2.1.2.2 Use of Different Quality of Wax for Pouring Cup

The experiment used two moulds made up of 6 layers of coating and a single seal coat. For the first mould, the pouring cup was made from a degraded wax while the second was produced from wax of the same quality as that used for the components. The degraded wax used was that from pattern dewaxing. The moulds were dewaxed at a temperature of 175°C.

4.2.2 Alternative Mould Construction

Since the preliminary experiments did not provide any useful results primarily because of mould failure (Refer to Chapter 5), an alternative mould construction was devised to undertake further experiments. The moulds were block moulds produced using a permanent pattern rather than an expendable pattern.

The alternative moulds were built using the Shaw process, with alumino–silicate used as the main refractory material. Alumino– silicate, in molochite form, with various sizes i.e –200, –30+80 and –8+16 for back up was used. Ethyl silicate was used as the binder with ammonia as the gelling agent. The composition of the mould build–up was as follows:

50% of molochite size –200

25% of molochite size –30+80

25% of molochite size –8+16

22% weight of ethyl silicate

1% of ammonia

a drop of anti–foam agent

The construction of the mould is shown in figures 4.4 and 4.5.

4.3 THE INFLUENCE OF ATMOSPHERE

4.3.1 The Influence of Atmosphere Using Shaw Process Moulds

Three moulds were prepared using the Shaw process with the constitution of materials described in experiment 4.2.2. The moulds were fired for 2 hours at a temperature of 1000°C and were allowed to cool slowly in the furnace to normal room temperature at 25°C ± 0.7°C. The moulds were arranged as follows: the first mould was cast in air; the second was flushed with CO_2/SF_6; and the third was flushed with CO_2/SF_6 using an enclosure around the mould.

The melt was prepared according to Magnesium Electron Ltd (MEL) melting practice. Hardener and Zirmax (grain refiner) were added at temperatures of 725°C and 780°C respectively. The crucible was removed from the furnace when the melt temperature was about 790°C to allow the temperature to drop to the intended pouring temperature of 730°C ±5°C. To expedite the temperature reduction, the melt was stirred gently, avoiding breakage of the protective film on the melt surface. The pouring temperature of 730°C ±5°C was chosen based on the result of the previous experiment (refer to chapter 5).

4.3.2 Further Investigation of the Influence of Atmosphere Using Shell Investment Moulds.

Since the results of the experiments using Shaw process moulds appeared to be positive, a further investigation was necessary to determine the relevance of the finding for ceramic shell investment moulds. For this, two shell moulds were produced. The moulds consisted of 7 dipped layers with a thickness of approximately 6.5 mm, fired at a temperature of 1000°C for 2 hours and allowed to cool in the furnace to normal ambient temperature. One mould was flushed directly inside the moulds with CO_2/SF_6 gas while the other was flushed with the enclosure around the mould. The construction of the mould and the enclosure are schematically presented in figure 4.6. The flow rate of the CO_2/SF_6 gas used was 40 litres/min and flushing was for approximately 30 seconds for each mould. Melting was carried out in a steel crucible and fluxes were used to suppress oxidation and burning throughout melting. Pouring was first conducted on the enclosed mould followed by the open mould at the temperature of 730°C±5°C.

4.3.3 Test to Determine the Minimum Inhibitive Gas Requirement

In this experiment, three ceramic shell investment moulds were used. The mould construction was as described in 4.3.2. The CO_2/SF_6 gas mixture was regulated to three different flow rates. The first mould was flushed at a rate of 25 litres/min, the second at 15 litres/min and the third at 5 litres/min. The gas was flushed for approximately 30 seconds. The melts were poured at a temperature of 730°C±5°C into moulds at ambient temperature (25°C ± 0.7°C).

4.4 INFLUENCE OF POURING TEMPERATURE

4.4.1 Casting in Shaw Process Moulds

Five moulds were prepared from alumino–silicate refractory material bonded with ethyl silicate using the Shaw process. The moulds were fired in a furnace for 2 hours at a temperature of 1000°C and were then allowed to cool slowly to ambient temperature, 25°C ± 0.7°C.

The melt was prepared from the following ingredients: 92.6% magnesium ingot and returns; 4.6% Zirmax grain refiner; and 2.8% misch metal hardener (24). Of the 92.6% magnesium used, 20% was returns and the rest virgin metal. The hardener and grain refiner used were supplied by MEL.

Melting was carried out in a steel crucible using an electric furnace. During melting, flux was used to exclude the atmosphere from the melt, thus preventing any reaction from occurring. For safety and cleanliness of the melt, the crucible and all the related tools which contacted the molten metal, and the magnesium returns were sand blasted prior to use. After melting and treatment, the melt temperature was 810°C and it was allowed to cool to a temperature of 780°C by removing the crucible from the furnace. Pouring was conducted in a descendant manner at five different temperatures i.e 780°C, 750°C, 730°C, 700°C and 680°C. Variation of the temperature was controlled within ±5°C.

The castings were removed from their moulds after 3 hours to ensure complete cooling had occurred.

4.5 THE USE OF POTASSIUM BOROFLUORIDE (KBF_4) AS AN INHIBITOR

4.5.1 Mould Material Mixtures

Using the Shaw process, nine moulds were prepared. The mould refractories were mixed with potassium borofluoride (KBF_4) at the following percentages: 2%, 4%, 6%, 8%, 10%, 20%, 30%, 40% and 50% by weight. The moulds were fired at a temperature of 1000°C for a period of 2 hours. It was found that the use of more than 20% by weight of KBF_4 was not advisable since moulds mixed with more than 30% KBF_4 were found to disintegrate during firing. The remaining moulds were left in the furnace to cool to room temperature.

4.5.2 Mould Face Coatings

As an alternative approach, potassium borofluoride was applied to two shell investment moulds through mixing the substance with SEMCO (a product of SMC Foundry Products Limited) mould coat. An addition of 2% KBF4 was mixed with the coating and applied on the inner face of the moulds by pouring the coat into the moulds. The excess coating was poured out of the mould, leaving behind a thin coating layer on the inner surface. The coating was applied twice to ensure every part of the mould was covered. The moulds were allowed to dry for 2 days. Pouring for all of the moulds was conducted at a temperature of 730°C±5°C.

4.6 THE USE OF DIFFERENT MOULD REFRACTORY MATERIALS

Six moulds were built, each from a different material. The materials were: magnesium oxide; calcium oxide; silicon carbide; calcium carbonate; graphite; and alumino–silicate as a control mould. All were constructed using the Shaw process and bonded with ethyl silicate. All of the moulds were fired at a temperature of 1000°C for a period of 2 hours and allowed to cool to ambient temperature. Melting was conducted in a steel crucible and the melt was prepared according to the recommended MEL melting practice. Pouring was conducted at a temperature of 730°C±5°C.

A further four moulds were made from anhydrous calcium sulphate (gypsum). The moulds were fired in stages, as per the supplier's instructions. Two stages of firing were recommended. In the first stage the moulds were fired at a rate of 175°C per hour to 720°C from the dewax temperature of 175°C. The second stage required that the mould be held at a firing temperature of 720°C for 2 hours. The moulds were then allowed to cool to normal ambient temperature. Casting was conducted at four different pouring temperatures in a descendant order i.e 780°C, 750°C, 730°C and 700°C.

4.7 CASTING OF TENSILE TEST SPECIMENS

4.7.1 Casting with Flux Method

Tensile test patterns were prepared and welded to the pouring cups as shown in figure 4.7. Twelve ceramic shell moulds were prepared and after dewaxing, the moulds were fired at a temperature of 1000°C with a soaking period of 2 hours. The moulds were then allowed to cool to room temperature. Pouring was conducted at four different temperatures i.e 780°C,750°C, 730°C, and 700°C. At each pouring temperature, moulds at three different temperatures were poured; one mould was at ambient temperature i.e 21°C, the second was heated to 100°C and the third was at 200°C. The equipment set–ups used for mould heating are shown in figures 4.8 and 4.9.

Pouring of the melt was conducted in a descendant pattern i.e started with 780°C followed by 750°C, 730°C, and finally 700°C. For each pouring, the melt was poured into moulds at temperatures of 21°C, 100°C and 200°C. Immediately after each pouring the power supply for mould heating was switched off to ensure a simultaneous cooling of the mould and melt. An example of the mould used is shown in figure 4.10.

4.7.2 Tensile Test

The test bar specimens were prepared by machining the cast specimens to the required dimensions (Hounsfield test bar no. 16) as shown in figure 4.7. Before each test commenced, the test bar diameter was measured. The test bar was then placed on an elongation gauge and reduction in area gauge to determine the initial length and area.

After the tensile test was completed, the percentage area reduction was measured. The measurement was taken as close as possible to the fractured edge. With the same procedure, the percentage elongation was taken by placing the best assembled fractured test bar onto the elongation gauge.

4.7.3 Experiment Control Measures to Reduce Result Variations

Four ceramic shell investment moulds, each comprising of 4 specimens, were prepared. The construction of the moulds was as described in subsection 4.3.2. The first mould was cast with a mild steel strainer placed at the neck of the pouring cup. The second was cast at a mould temperature of 630°C±10°C i.e immediately taken out from the firing furnace, while the third mould was a control mould. The fourth mould was poured using metal prepared by the fluxless method. The third and the fourth moulds were poured at an ambient temperature of 21°C. Pouring of this melt was conducted at a temperature of 750°C ±10°C and within an inert atmosphere. An x–ray was taken to determine the quality of the castings.

4.7.4 Casting with Fluxless Method

This experiment was a repetition of that described in subsection 4.7.1. The procedures adopted were the same, except that the melt was prepared by a fluxless method. The holding time interval between each pour was approximately 1 hour. Before each pouring was conducted, the melt was skimmed within an inert atmosphere of CO_2/SF_6. Supply of the CO_2/SF_6 gas was continued until immediately before pouring.

Tensile testing was conducted on the 'machined to dimension' specimens as described in the subsection 4.7.2.

4.8 MICROSTRUCTURE OBSERVATION

4.8.1 Sample Preparation

A sample was taken from a cast specimen produced using each combination of pouring and mould temperature. Each sample was prepared on 240, 320, 400 and 600 grit sand paper with running water and polishing was performed using 6 micron, 1 micron and 0.25 micron diamond paste impregnated polishing wheels. It was then etched with a solution of 2% nitric acid in alcohol. The specimen was washed with running water and methanol and dried with an electric drier. The microstructure was observed using a x100 magnification.

4.8.2 Grain Size Measurement

The measurement of the number of grains was performed by magnifying the grain structure to a magnification of 200x. The image was then projected onto a television screen via a video camera which further magnifies the image to 960x magnification. A square frame of 100mm by 100 mm was drawn on the television screen. The number of grains counted were those contained within the frame. The results presented are the averages of four counts conducted on four different areas and represent the number of grains counted within the $0.01m^2$ screen area.

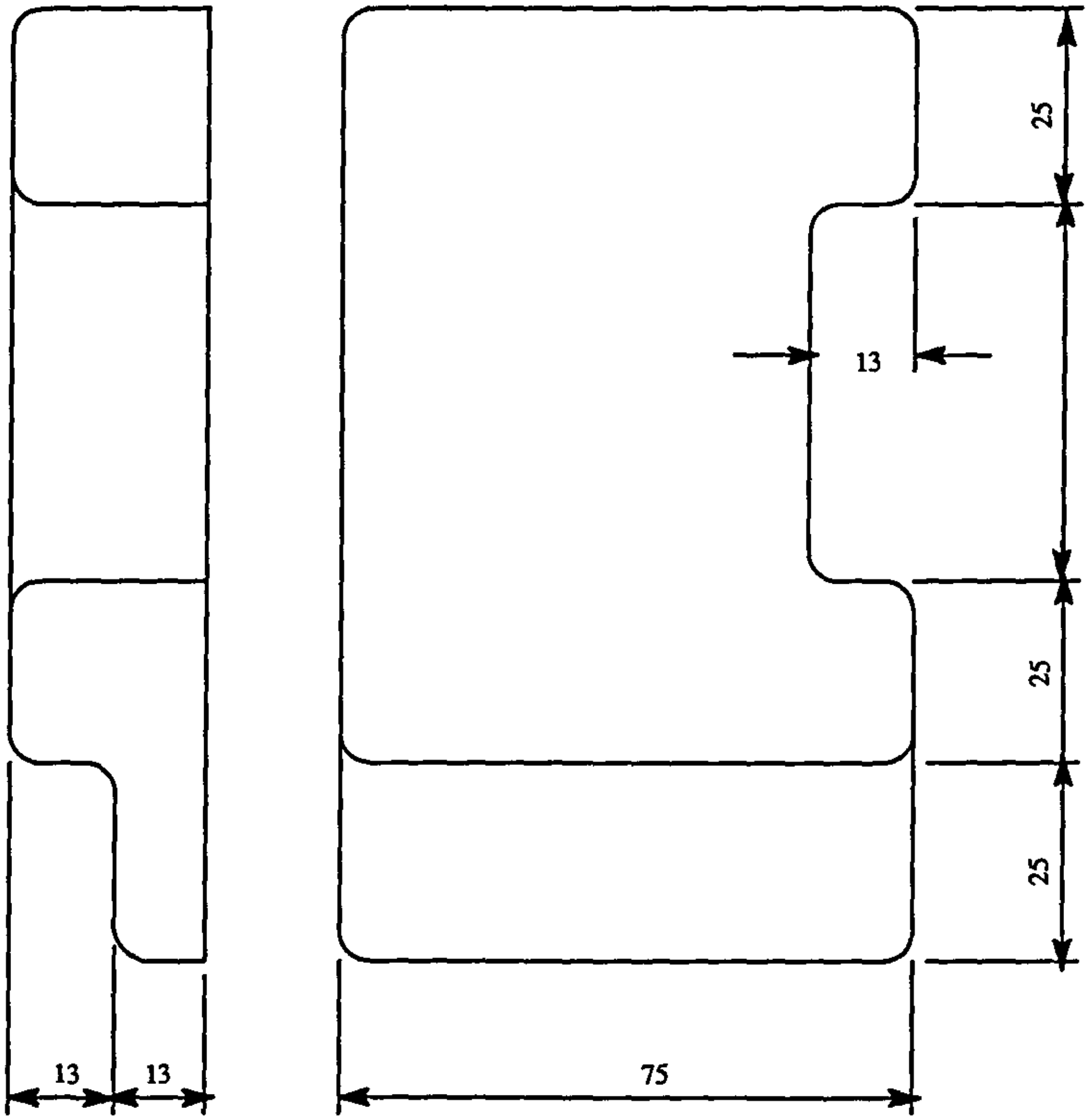

Figure 4.2 : Test casting configuration and dimensions

does drawing describe part?

Figure 4.3 : The shell investment mould

Figure 4.4 : The Shaw process mould

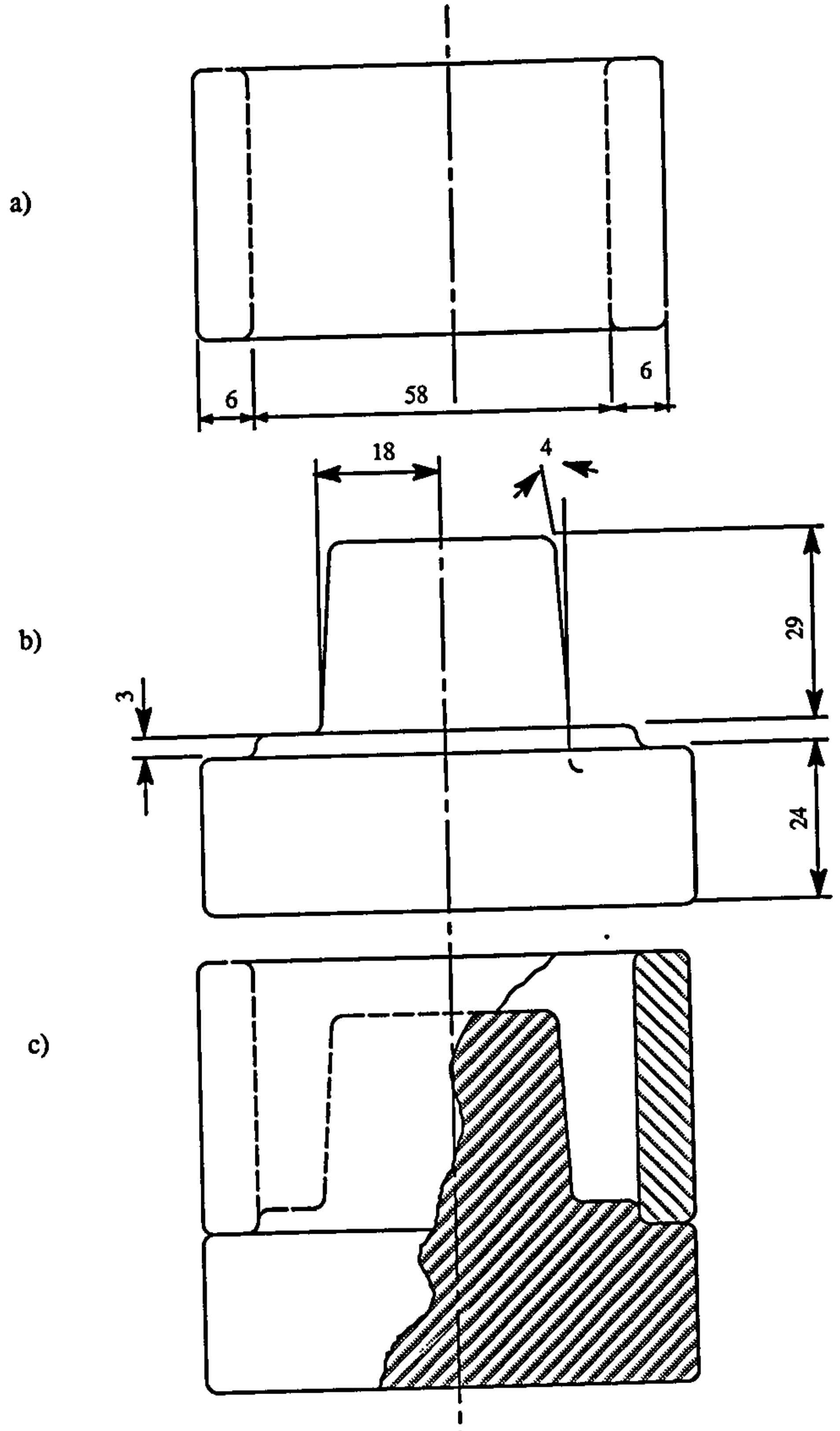

Figure 4.5 : Schematic drawings of the Shaw process mould a) moulding box b) male mould c) a sectioned assembly drawing.

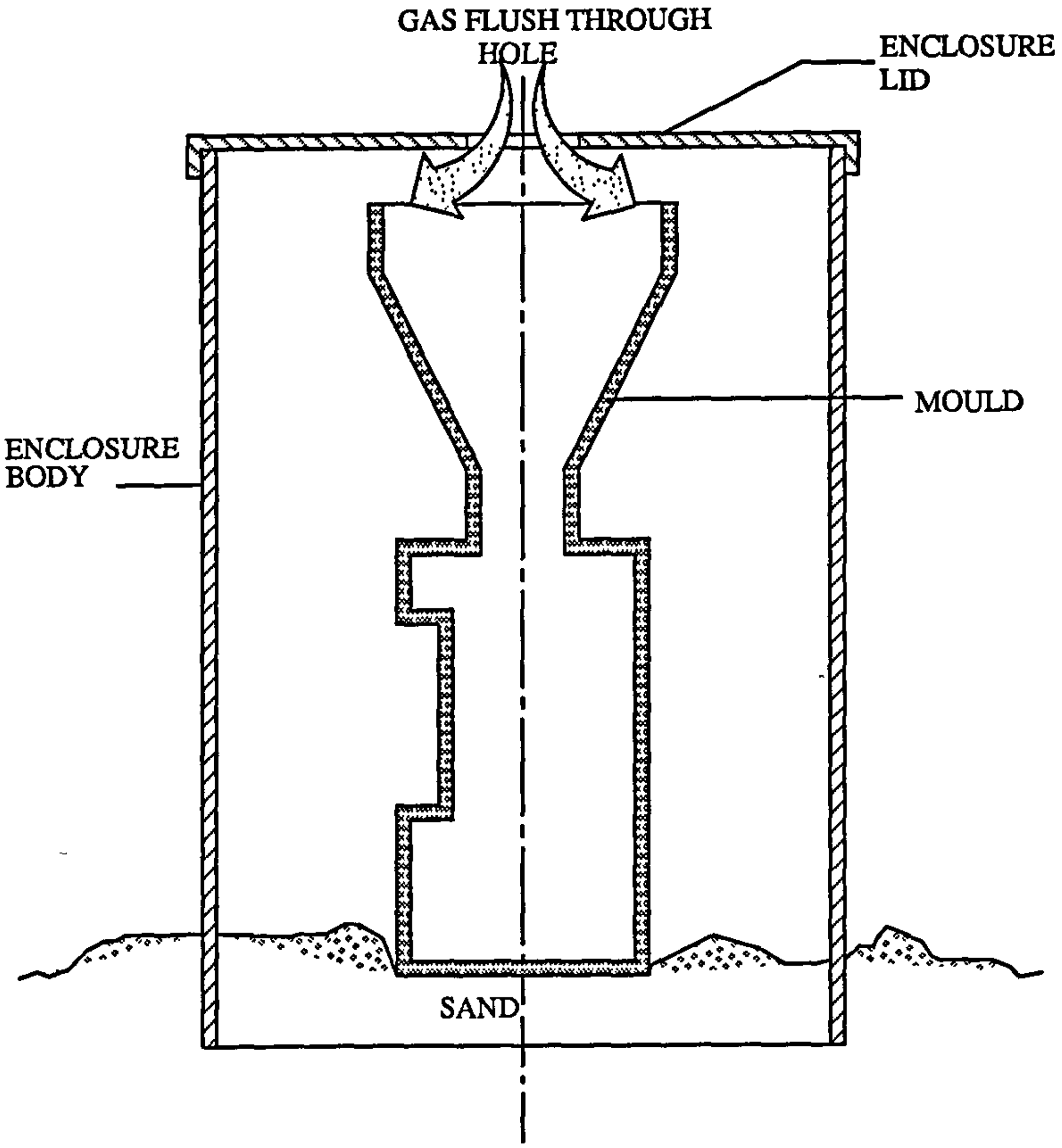

Figure 4.6 : Schematic representation of the enclosure for CO_2/SF_6 flushing prior to pouring

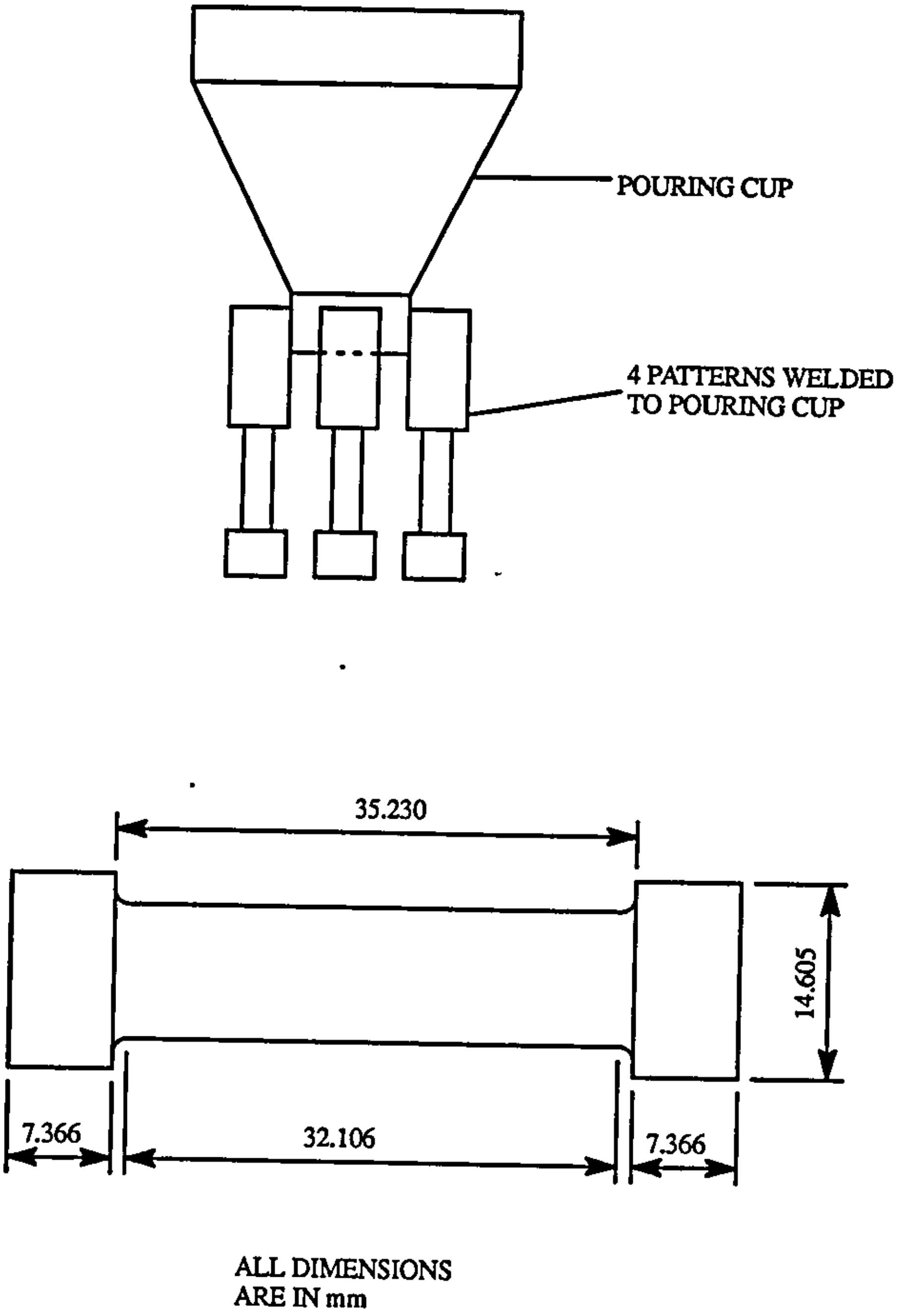

Figure 4.7 : Schematic drawings of welded patterns and the standard tensile test specimen used.

What test m/c ?
how was E measured?

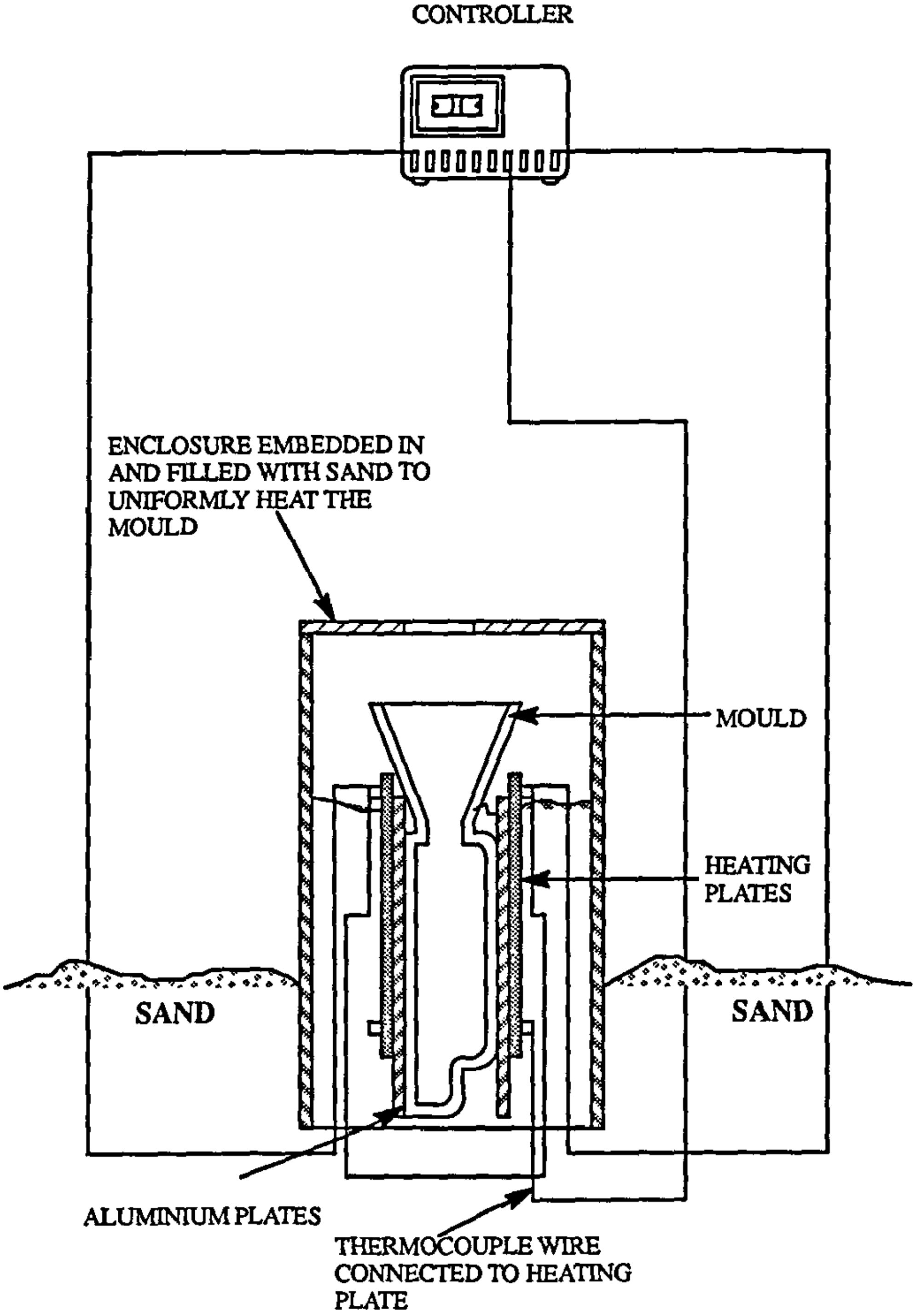

Figure 4.8 : Schematic apparatus set up for mould heating for the temperature of 200°C

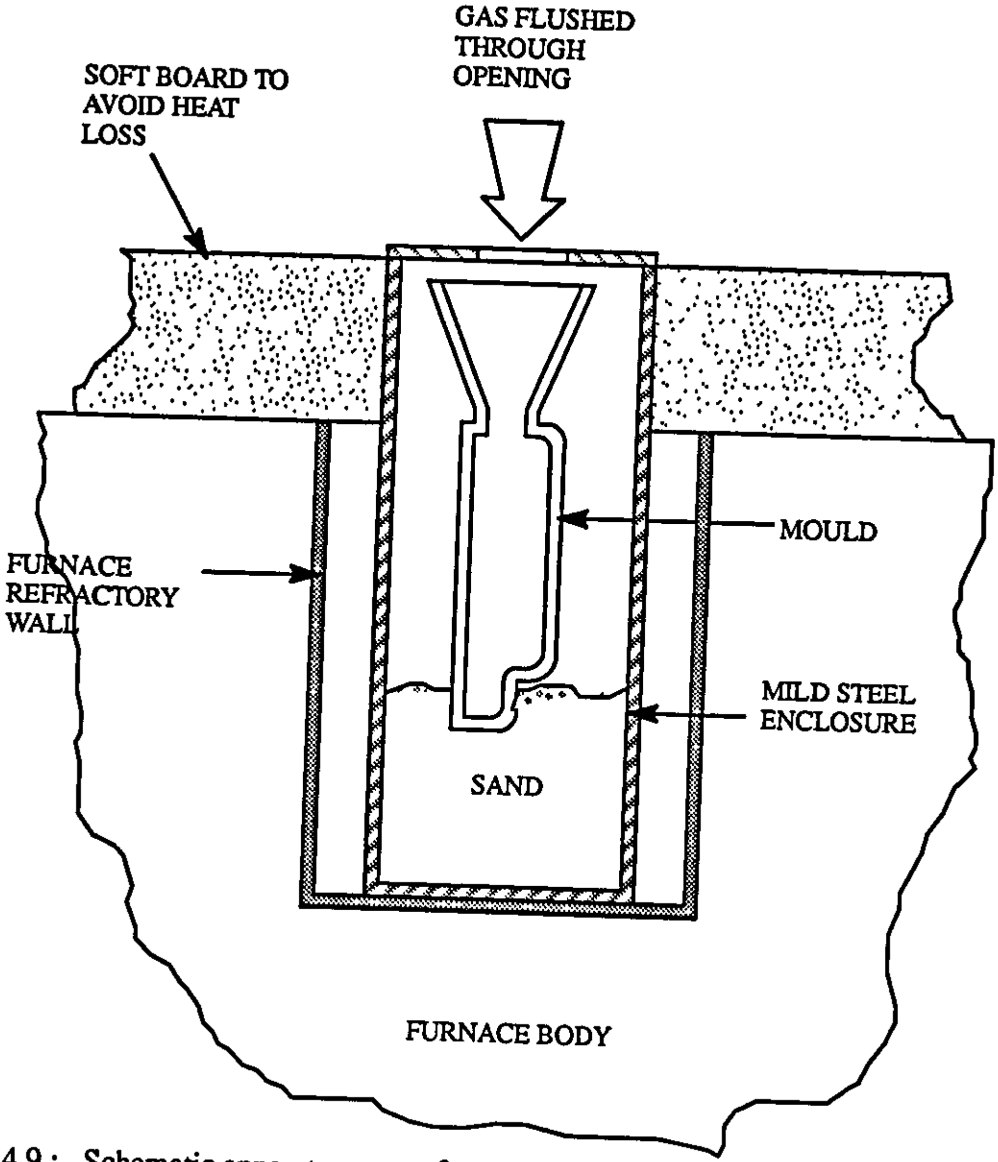

Figure 4.9 : Schematic apparatus set up for mould heating for the temperature of 100°C

Figure 4.10 : A shell investment mould for casting of tensile test specimens.

CHAPTER 5

RESULTS AND OBSERVATIONS

5.1 PRELIMINARY EXPERIMENTS

For the experiments conducted using ceramic shell moulds with mould temperatures of ambient and 500°C, and from the Table 5.1, it was observed that as soon as each mould was poured it cracked and leaked. The leaking alloy started to glow and slowly burn. None of the samples could be salvaged for examination.

TEMP. (°C)	MOULD	MOULD TEMP. °C	OBSERVATIONS
780	1st	500	cracked
	2nd	500	cracked
	3rd	500	cracked
	4th	500	cracked
780	1st	ambient	cracked
	2nd	ambient	cracked
	3rd	ambient	cracked
	4th	ambient	cracked

Table 5.1 : Preliminary experiment casting results

5.2 VERIFICATION OF THE CAUSE/S OF MOULD CRACKING

5.2.1 Pouring at Higher Temperature

Neither of the moulds, i.e the mould of the original design or that with rounded corners, which were poured with metal at a temperature of 800°C exhibited cracks Similarly, the mould of the original design poured at 780°C did not crack.

Is this set of results consistent with those of Table 5.1

5.2.2 Extended Drying Time and Use of the same Quality Wax for Pouring Cup

Neither of the moulds, which had been dried for one week prior to firing exhibited cracking.

5.2.3 Extended Drying Time

Neither of the moulds, which were left to dry for 24 hours and 1 week respectively, prior to firing showed signs of cracking.

5.2.4 Use of Different Quality of Wax for Pouring Cup

Neither the mould produced with a quality wax pouring cup or that produced using a degraded wax pouring cup showed signs of cracking.

5.3 THE INFLUENCE OF ATMOSPHERE

It was observed that the casting produced from the mould which was flushed directly with CO_2/SF_6 gas, showed signs of reaction at its surface. This was manifested as a black rough surface, particularly at the sides of the casting.

The mould that was flushed within the enclosure produced a clean–metallic casting surface. This indicated that no reaction had taken place. Figure 5.1 shows the castings produced.

5.3.1 Further Investigation on the Influence of Atmosphere Using Shell Investment Moulds.

The casting produced in the shell mould flushed directly with CO_2/SF_6 exhibited a similar surface to that produced in the Shaw process mould (figure 5.2). The only difference was that black spots were observed all over the casting.

In the case of the casting produced from the shell investment mould flushed within the enclosure, a clean–metallic surface was observed. There was no sign of black spots.

5.3.2 Test to Determine the Minimum Inhibitive Gas Requirement

The casting formed in the mould flushed with the flow rate of 25 litres/min exhibited a clean metallic finish. There was no sign of a reaction having taken place. The castings produced at flow rates of 15 and 5 litres/min demonstrated dull metallic surfaces (figure 5.3).

5.4 INFLUENCE OF POURING TEMPERATURE

The castings produced in Shaw process moulds and poured at temperatures of 780°C and 750°C had a poor surface finish with burn marks and signs of heavy reaction at the casting sides. However, the castings poured at temperatures of 730°C, 700°C and 680°C had a better finish with only a slight indication of reactions at their sides. In general, it was observed that as the pouring temperature was reduced the surface finish at the casting sides seemed to improve. Surprisingly, it was found that the bottom surfaces of all the castings were free from any sign of reactions. For further investigation, the temperature of 730°C was selected as the reference temperature at which further castings would be made. Although temperatures of 700°C and 680°C produced a better finish than did 730°C, it was considered that at these temperatures the fluidity of the melt was too low. This might create a problem should thin section samples be cast. The castings produced are shown in figure 5.4.

5.5 THE USE OF POTASSIUM BOROFLUORIDE (KBF_4) AS AN INHIBITOR

5.5.1 Mould Material Mixtures

Apart from the casting produced in the mould containing 20% KBF_4, all of the castings exhibited heavy reactions on their surfaces, i.e a dark 'burn–like' appearance. The bottom part of the castings showed a better finish (metallic surface). Generally, it was observed that as the KBF_4 addition was increased, the extent of reactions seemed to reduce. The castings produced are shown in figure 5.5.

The casting produced from the mould containing 20% KBF_4 exhibited a metallic appearance with black spots all over its surface.

5.5.2 Mould Face Coatings

Both castings exhibited a 'burn–like' appearance on their surfaces which indicated that a heavy reaction had taken place (see figure 5.6).

5.6 THE USE OF DIFFERENT MOULD REFRACTORY MATERIALS

The Shaw moulds which were built using magnesium oxide, calcium oxide and calcium carbonate burned away and disintegrated slowly after the magnesium melt was poured into the moulds. The castings produced using graphite and silicon carbide moulds exhibited burns all around their upper surface.

It was observed that all the castings produced in the anhydrous calcium sulphate moulds manifested burns around their upper surfaces (see figure 5.7). Their surfaces were very rough, indicating that reaction had taken place. In general, it was observed that, comparatively, as the pouring temperature was reduced, the extent of reaction seemed to be less severe.

5.7 CASTING OF TENSILE TEST SPECIMENS

5.7.1 Tensile Test Results for the Castings Produced by the Flux Method

5.7.1.1 Observations

Average mechanical property results are presented in Table 5.2 (see Appendix A for detail results) for variation of mould and casting temperatures for the castings produced by the flux method. The results presented are the averages of three to eleven tests. Examination of Table 5.2 reveals a pronounced effect of the pouring temperature on the tensile test results i.e as the pouring temperature was decreased from 780°C to 700°C, the UTS value increased. The results also show that there is no significant effect from mould temperature, except for the pouring temperatures of 700°C and 730°C which show a drop in UTS at the mould temperature of 100°C.

The highest UTS value recorded was 136 MPa at a combination of pouring and mould temperature of 700°C and 200°C respectively. At the pouring and mould temperature combination of 730°C and 100°C, the UTS value was at its lowest.

Casting Temperature (°C)	Mould Temperature (°C)	Number of Specimens Tested	Average UTS (MPa)	Average Young's Mod. (GPa)	Average Elong. (%)
700	21	3	130	34	2.5
	100	4	113	29	1.1
	200	3	136	30	3.3
730	21	11	128	33	3.6
	100	6	105	31	2.4
	200	8	125	35	2.8
750	21	8	120	26	3.1
	100	6	120	27	3.3
	200	8	128	30	3.9
780	21	3	108	21	3.2
	100	4	118	20	3.5
	200	4	119	17	3.0

Table 5.2 : Summary of the average tensile test results for castings produced by the flux method

5.7.2 Experiment Control Measures to Reduce Result Variations

The presence of a mild steel strainer resulted in two of the four specimens being defective due to cold shuts. The casting poured at a mould temperature of 630°C ± 10°C exhibited heavy reactions on its surface and a hard shell was observed on the casting (figure 5.8). The control casting and the casting produced from the fluxless melt showed no reaction on their surfaces. Radiographic examination revealed that some of the castings produced from a fluxless melt contained minor defects. The defects were believed to be inclusions of thin oxide films which were included close to the casting surface. The depth of the inclusion can be observed from the exposure intensity of the defect on the negative compared to the aluminium wire placed on top of the x–ray film cartridge before exposure was conducted. The defects were removed during machining to final dimensions.

5.7.3 Tensile Test Results for the Castings Produced by the Fluxless Method

5.7.3.1 Observations

The average results for the castings produced by the fluxless method are presented in Table 5.3 (see Appendix A for detail results). The results are mainly averages of three to four tests except for the specimens prepared at the pouring temperature of 700°C for mould preheat temperatures of 21°C and 200°C which were taken from ten and seven tests respectively. Examination of Table 5.3 reveals that there is a slight tendency for the extreme temperatures of 700°C and 780°C to produce higher UTS values than the intermediate temperatures.

The highest UTS value recorded resulted from the combination of pouring and mould preheat temperature of 700°C and 200°C respectively while the lowest resulted from the pouring temperature of 730°C and mould preheat temperature of 200°C. Generally, the UTS values recorded are well above the minima given in the specifications for sand casting of the same alloy.

Casting Temperature (°C)	Mould Temperature (°C)	Number of Specimens Tested	Average UTS (MPa)	Average Young's Mod. (GPa)	Average Elong. (%)
700	21	10	154	40	4.2
	100	4	158	37	5.0
	200	7	163	42	5.0
730	21	4	144	35	4.0
	100	3	129	37	3.0
	200	4	128	40	2.8
750	21	3	131	43	3.2
	100	4	138	42	3.7
	200	4	133	38	3.3
780	21	4	150	36	3.3
	100	3	145	33	3.6
	200	3	149	31	4.6

Table 5.3 : Summary of the average tensile test results for castings produced by the fluxless method

5.8 MICROSTRUCTURE OBSERVATION

The results of the observation can be found in Table 5.4. It can be observed that the number of grains counted for some of the temperature combinations does not correspond with the value of the UTS recorded. For example, at the pouring temperature of 780°C and mould preheat temperature of 21°C, the difference in the number of grains counted for castings in batch 1 and 2 varied by a factor of 2 even though the variation in UTS was small.

Pouring Temp. (°C)	Mould Temp. (°C)	Casting Batch	Average number of Grains/0.01m^2	UTS (MPa)
700	21	1	22.7	159
		1	17.8	151
		1	14.3	151
	100	1	17.0	158
	200	1	19.7	162
		1	9.7	164
730	21	1	15.6	144
	100	1	11.7	129
	200	1	9.3	128
750	21	1	10.4	122
		2	16.0	149
		3	11.0	122
	100	1	8.8	133
		2	11.3	147
		3	8.3	133
	200	1	8.7	129
		2	12.7	141
		3	9.6	130
780	21	1	26.0	151
		2	13.5	148
	100	1	7.0	154
		2	10.7	136
	200	1	13.7	150
		2	8.7	156

Figure 5.4 : Summary of the grain measurement results for castings produced by the fluxless method

Figure 5.1 : Castings produced using Shaw process moulds and CO_2/SF_6 gas mixture:

1) flushed within an enclosure 2) flushed directly inside the mould

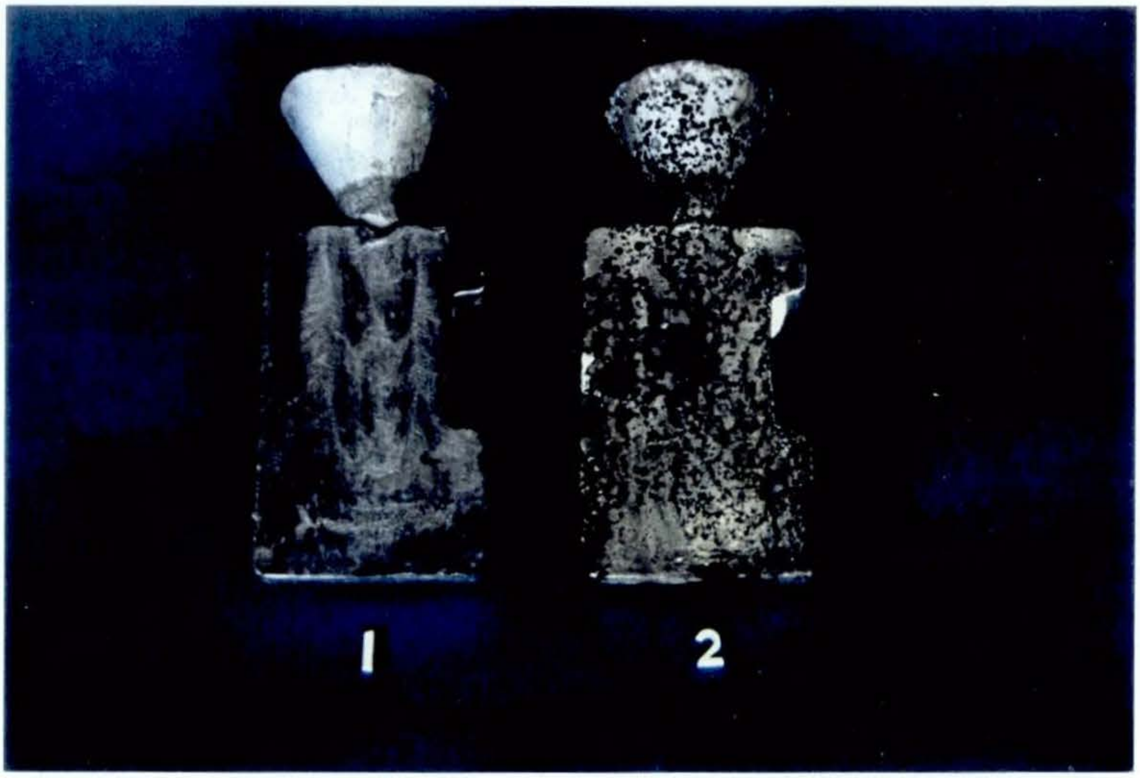

Figure 5.2 : Castings produced using Shell Investment moulds and CO_2/SF_6 gas mixture 1) flushed within an enclosure 2) flushed directly inside the mould

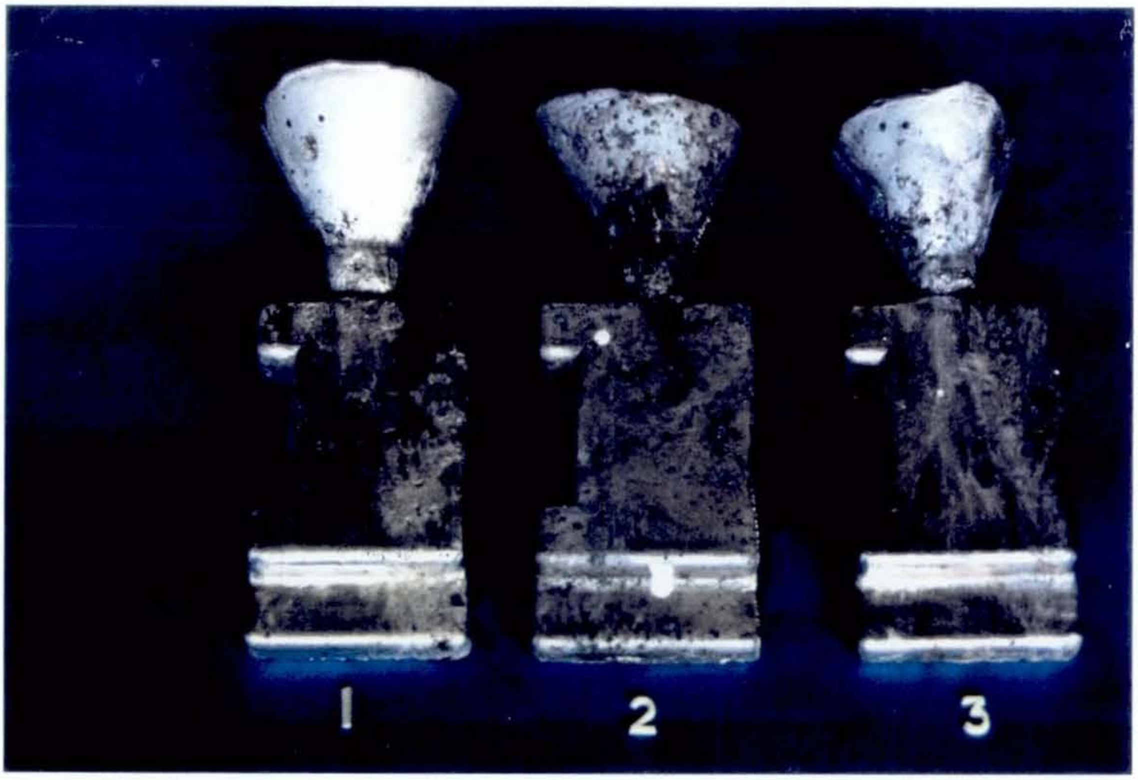

Figure 5.3 : Castings produced using different CO_2/SF_6 gas mixture flow rates:

1) 25 litres/min 2) 15 litres/min 3) 5 litres/min

Figure 5.4 : Castings produced to evaluate the influence of pouring temperature on metal–mould reaction : 1) 680°C 2) 700°C 3) 730°C 4) 750°C 5) 780°C

Figure 5.5 : Castings produced using moulds containing various percentages of KBF_4 :

1) 2% 2) 4% 3) 6% 4) 10% 5) 20%

Figure 5.6 : Casting produced in a mould coated with SEMCO mixed with 2% KBF_4.

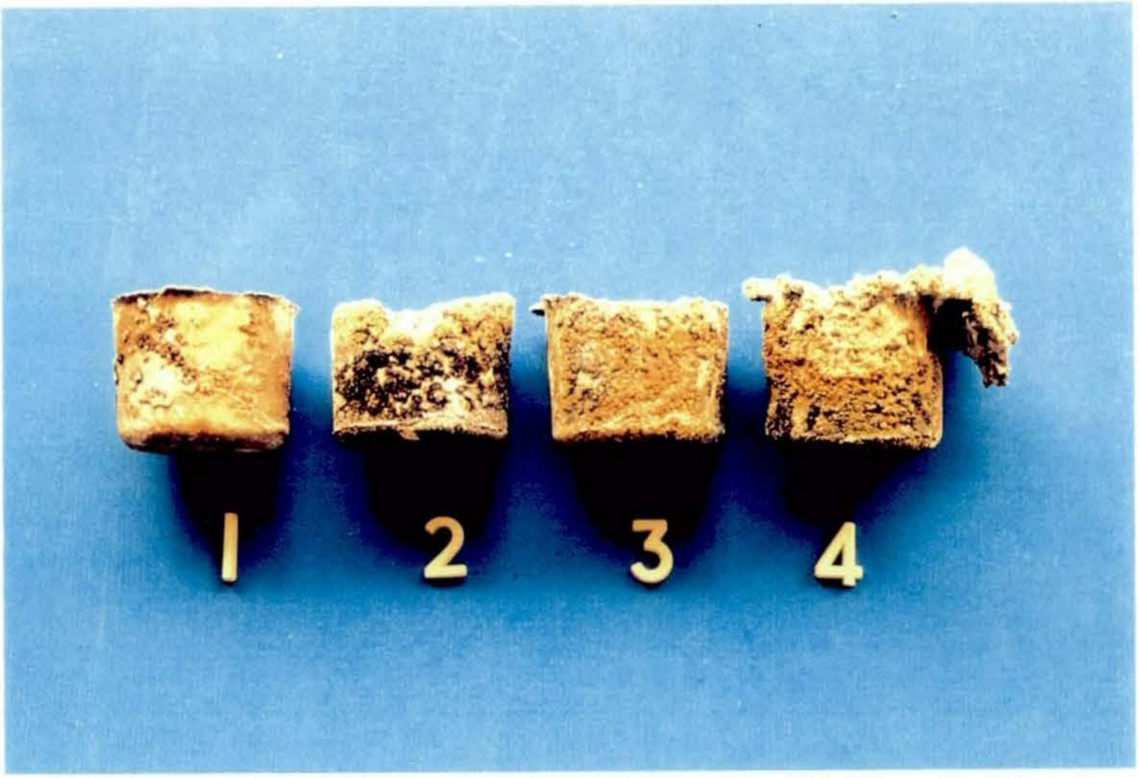

Figure 5.7 : Castings produced in anhydrous calcium sulphate moulds poured at different pouring temperatures : 1) 700°C 2) 730°C 3) 750°C 4) 780°C

Figure 5.8 : Casting produced in a mould heated to a temperature of 630 °C ± 10 °C

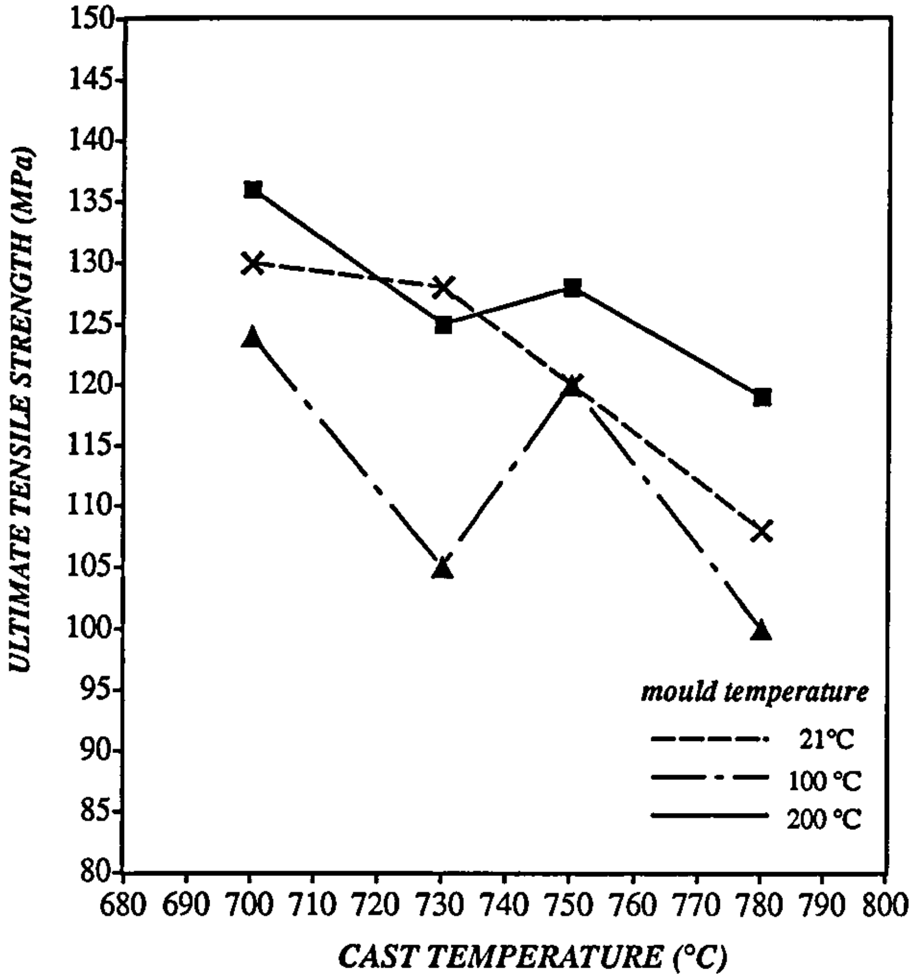

Figure 5.9 : Effect of pouring temperature and mould preheat temperature on the ultimate tensile strength of castings produced using the flux method.

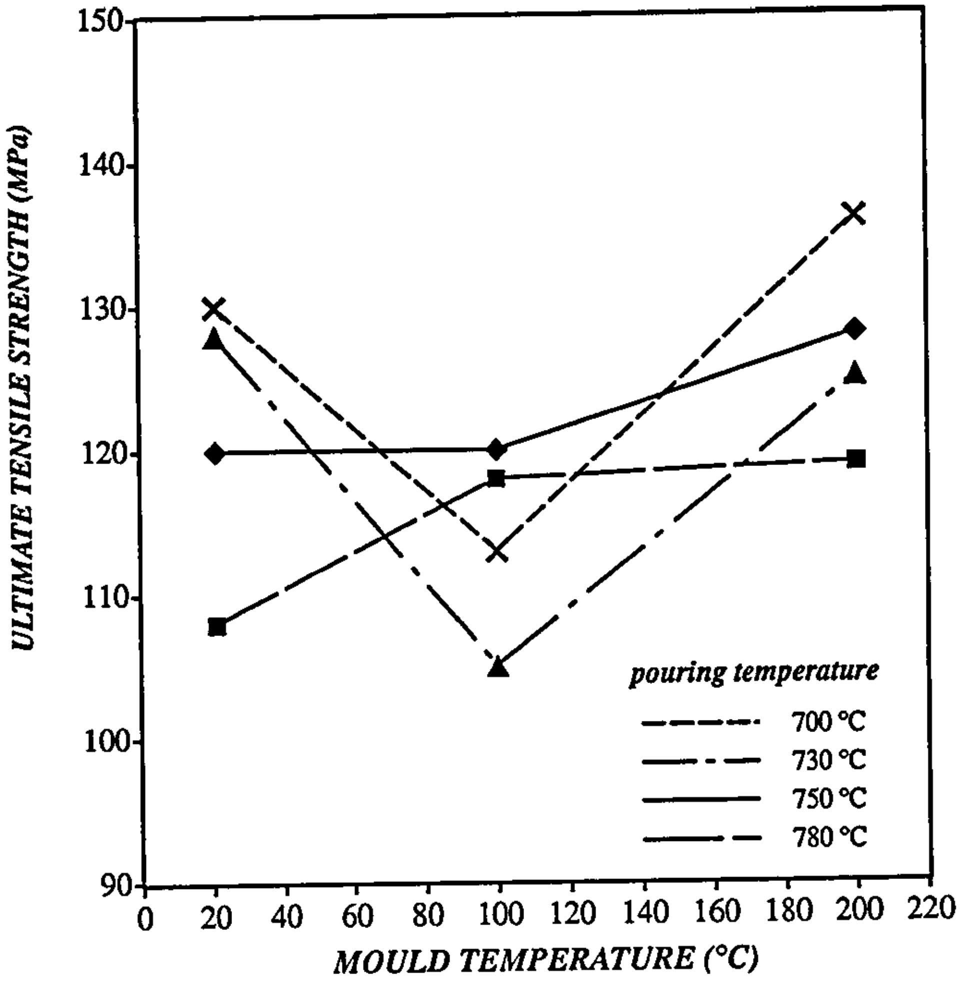

Figure 5.10 : Effect of mould preheat temperature and pouring temperature on the ultimate tensile strength of castings produced using the flux method.

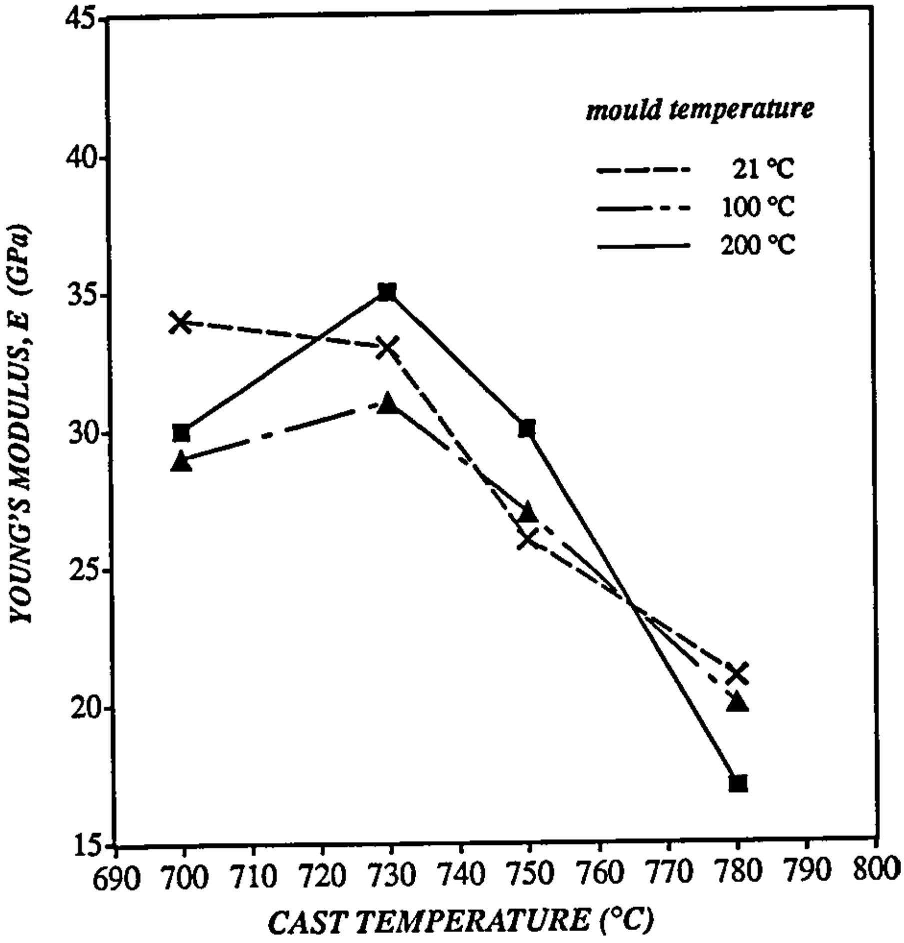

Figure 5.11 : Effect of pouring temperature and mould preheat temperature on the elasticity (Young's Modulus) of castings produced using the flux method.

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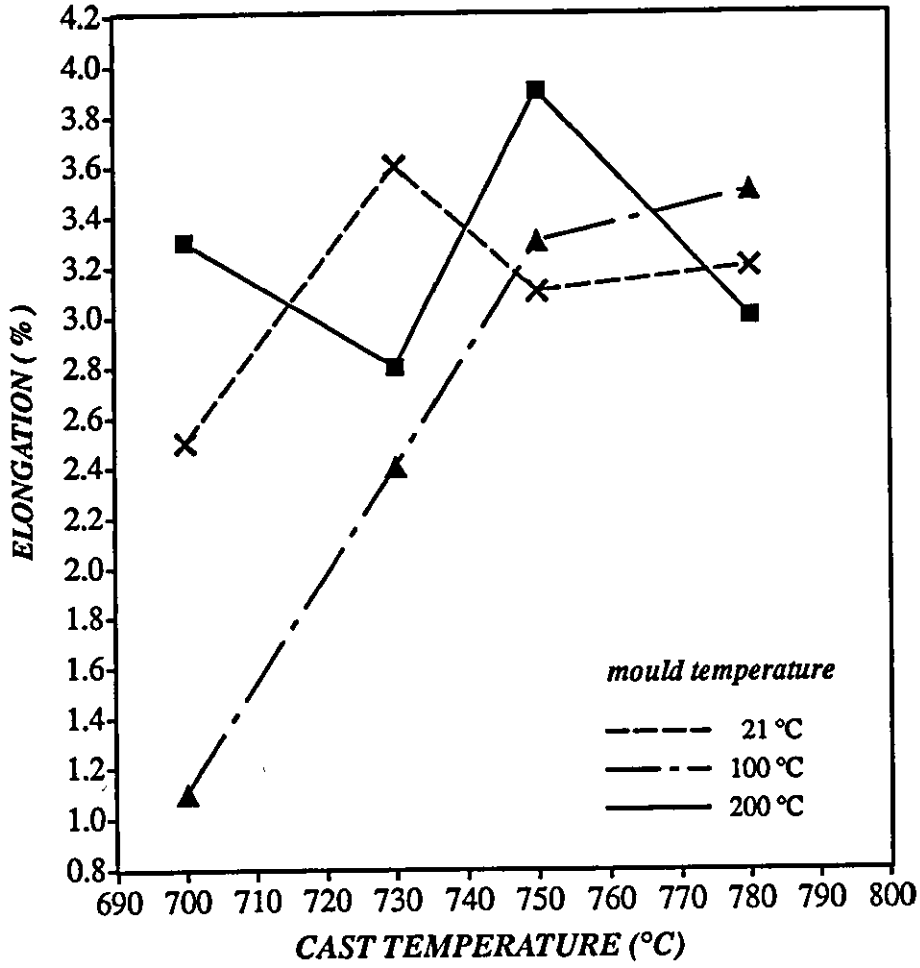

Figure 5.12 : Effect of pouring temperature and mould preheat temperature on elongation of castings produced using the flux method.

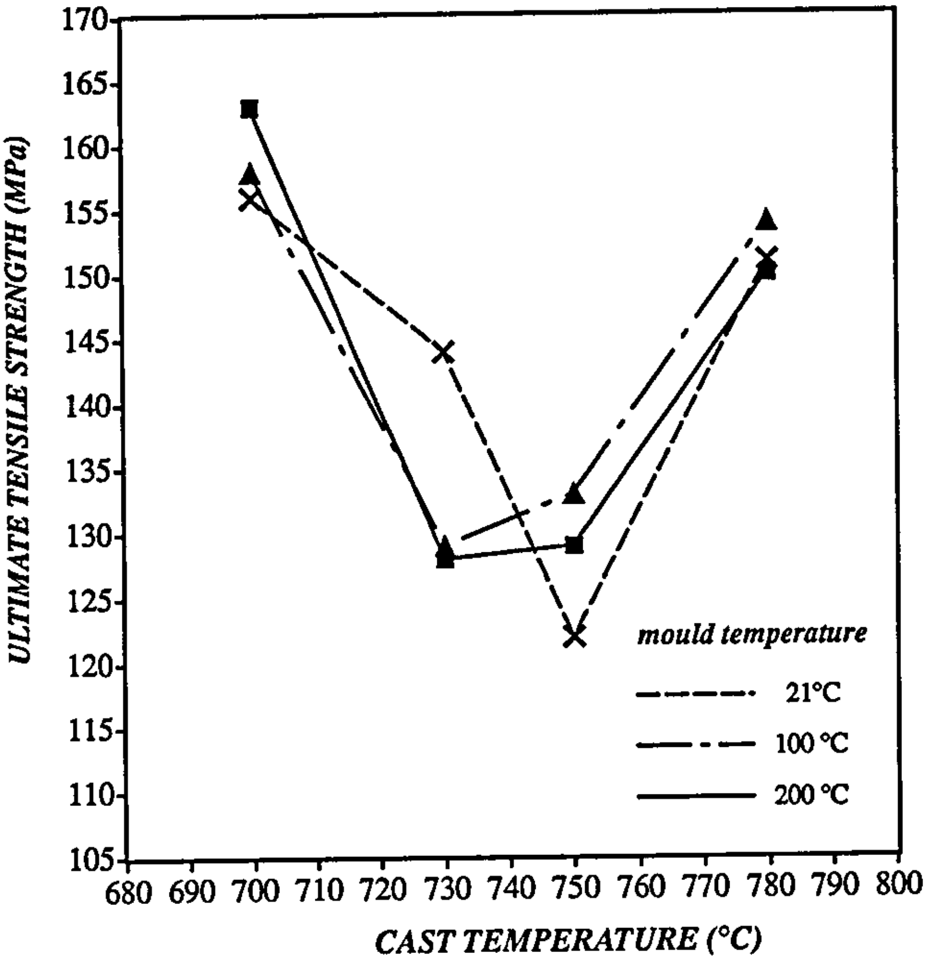

Figure 5.13: Effect of pouring temperature and mould preheat temperature on the ultimate tensile strength of castings produced using the fluxless method.

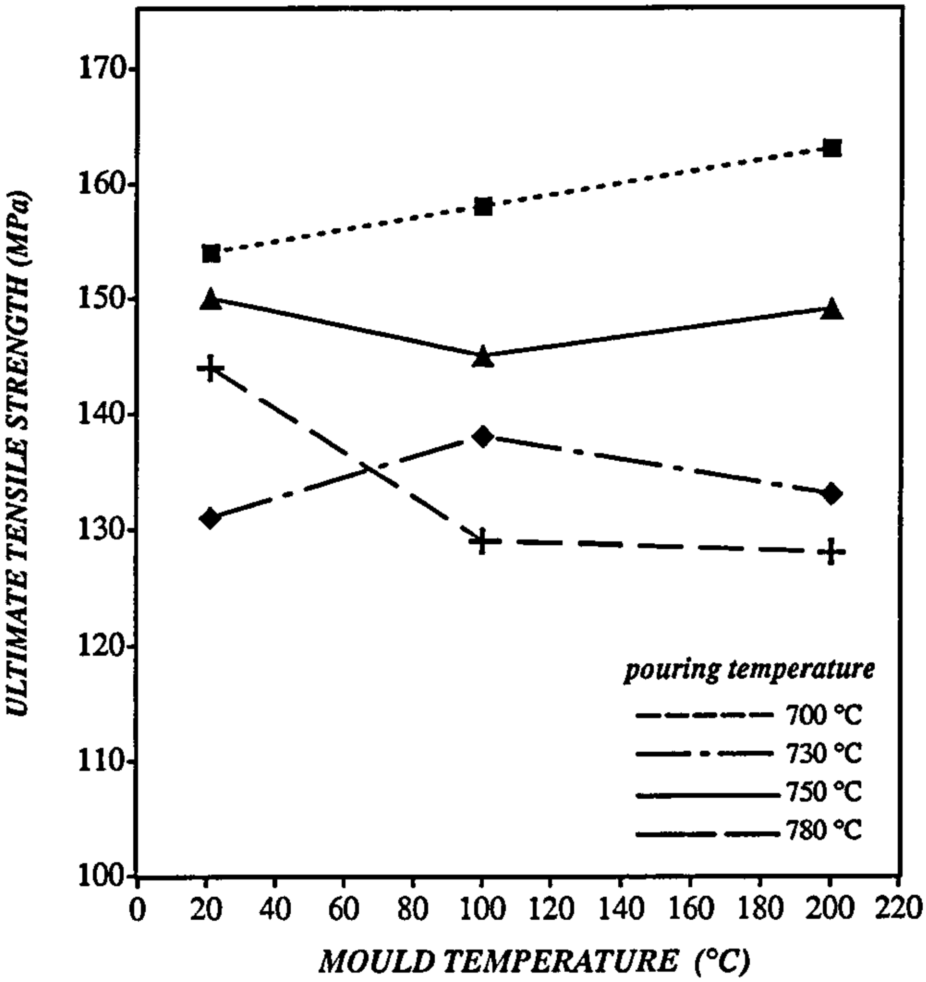

Figure 5.14: Effect of mould preheat temperature and pouring temperature on the ultimate tensile strength of castings produced using the fluxless method.

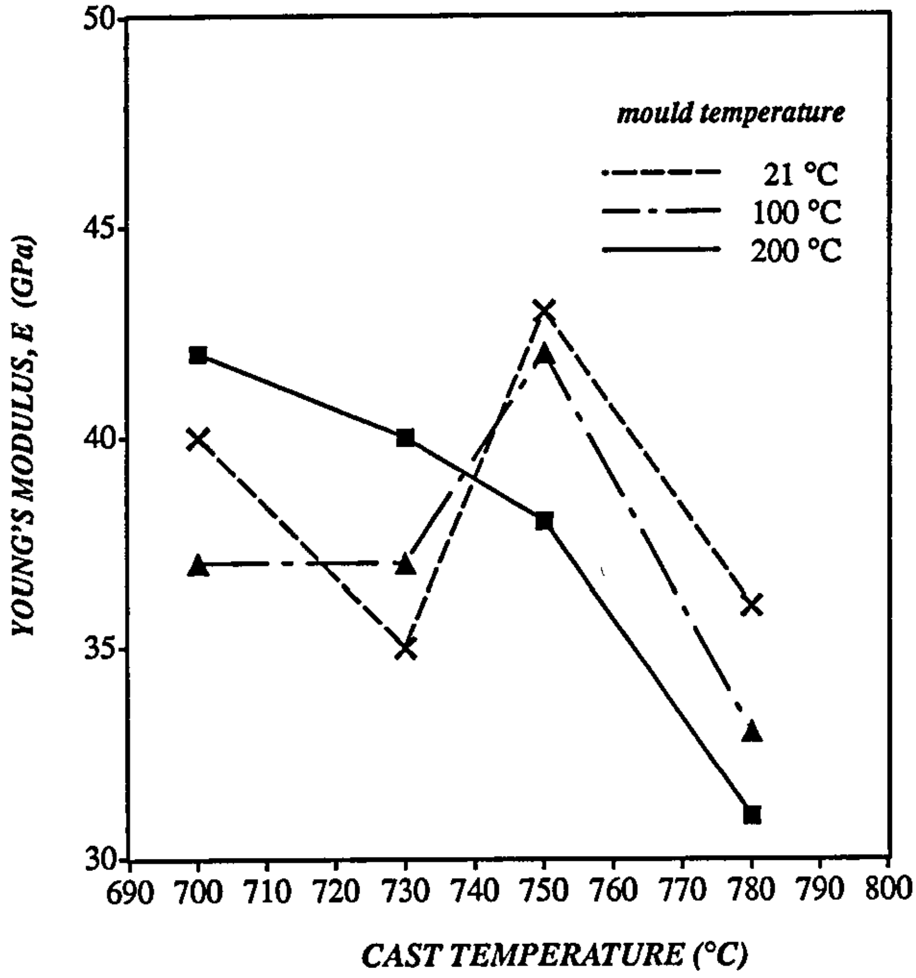

Figure 5.15 : Effect of pouring temperature and mould preheat temperature on modulus of elasticity (Young's Modulus) of castings produced using the fluxless method.

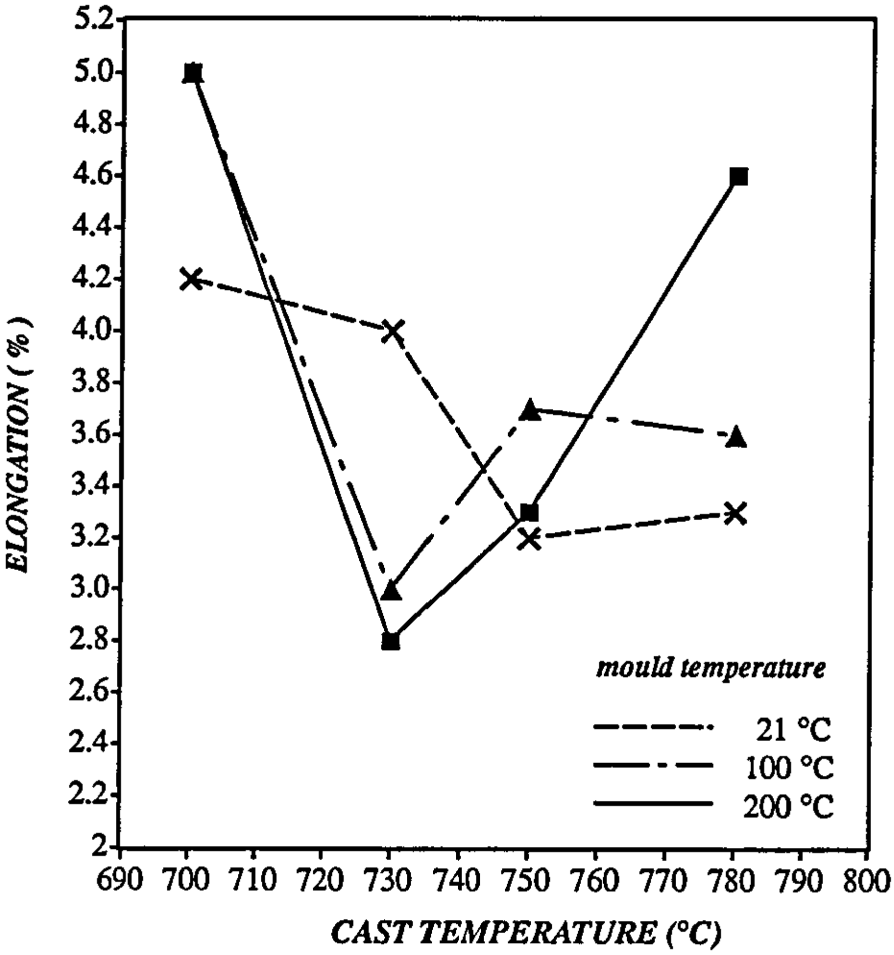

Figure 5.16 : Effect of pouring temperature and mould preheat temperature on the elongation of castings produced using the fluxless method.

CHAPTER 6

DISCUSSION

6.1 SHELL INVESTMENT MOULD

6.1.1 Preliminary Experiments

It was anticipated that the investment shell moulds built for this experiment could withstand any melt temperature. From the results tabulated in Table 5.1, it can be seen that each of the eight moulds that were poured at the temperature of 780°C, regardless of the mould temperature, exhibited cracks. Inspection of the moulds and castings revealed that all of the moulds poured at 780°C cracked at the bottom and side edges (along the front perimeter) as shown in figure 6.1.

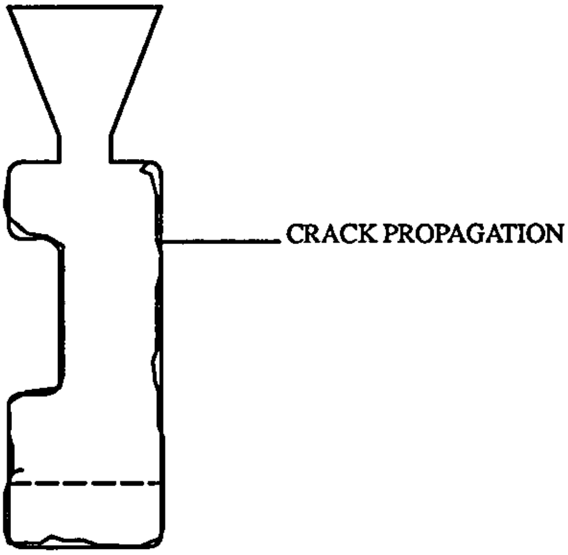

Figure 6.1 : Crack propagation at the side and bottom edges of the mould

Based upon this observation, the cracking of the shell investment moulds might be due to two possible reasons: first, the cracking might be due to the magnesium itself and the effect of pouring temperature; and second, failure due to the processing method used during mould building. These possibilities are discussed in the following section.

6.1.1.1 Magnesium alloy

6.1.1.1.1 Pouring Temperature

From the experimental results, it was postulated that, a possible reason for mould cracking might be due to the temperature of 780°C since the castings poured at 730°C did not crack. Furthermore, it is known that the reaction of magnesium and silicate is exothermic (67) and this, combined with a relatively high pouring temperature, might superheat the mould and contribute to its failure.

However, shell investment moulds have been used for casting other alloys (69,70) at much higher pouring temperatures, without failure. Since only two mould temperatures were used on a limited number of investment shell moulds, a firm conclusion could not be established.

6.1.1.1.2 Reactions Between the Mould and Magnesium

Kiesler and Moore described an interesting phenomenon in their study of metal–mould reactions with steel cast in foundry sand (11) :

'When the metal entered the mould gases liberated during the heating of the mould material exploded and generated shock waves in the metal which in turn impacted against the mould wall'.

A similar phenomenon was reported by Torok & Wilson (25) with regard to magnesium. In their explanation, they stated that magnesium when in contact with silicate reacted and

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Thermodynamic

formed Mg_2Si and O_2 i.e $2Mg + SiO_2 \rightarrow Mg_2Si + O_2$. The O_2 which was liberated accelerated the oxidation process to such an extent that extreme gas pressure and mould pressure led to an explosion. They further added that a shell mould will break or crack long before extremely high pressure is produced.

6.1.1.2 Mould Build-up

6.1.1.2.1 Drying

While in the process of building the mould, a crack may have initiated within the mould layer during drying as described by Schwartz (71):

'The rate of drying is independent of the slurry liquid content and equal to the rate of evaporation from a free liquid surface, dependant upon the temperature, humidity and air flow over the surface. The evaporation rate continues at a rate controlled by external conditions such as temperature, humidity and air flow. Once the surface liquid has been removed, the balance of the liquid must be supplied to the surface for further evaporation by wicking or capillary force'.

'As the liquid moves from the interior portion of the shell to the surface and the amount in the interior is reduced, it becomes more difficult for the liquid to come to the surface, thus reducing the evaporating rate. Eventually, the wicking or migrating of the liquid to the outer surface stops and the surface of the refractory becomes free of liquid. As this process progresses, the refractory particles are forced together by surface tension. Shrinkage may occur during drying when the particles are free of liquid. Cracking may occur if the drying is not uniform as internal stresses are formed between the dry and near dry sections of the shell. As the drying process takes place, high thermal expansion and contraction of the wax occurs'.

With regard to cracking due to wax thermal expansion and contraction, Schwartz (55) explained that too rapid drying will cause a chilling effect which causes the pattern to contract while the coating is still wet and unbonded. As the coating develops strength and shrinks to a certain extent, the wax begins to regain temperature and expand. This can cause the coating to crack.

6.1.1.2.2 Uneven Coating/Thickness

During the dipping of the wax pattern into slurry and the subsequent draining off, the tendency for an uneven coating to form is great, particularly at the bottom edge of the pattern, since excess slurry will flow under gravity to the bottom part of the pattern. This will create a thicker layer at the bottom of the mould. With an uneven coat,an uneven amount of residual moisture is left in the shell. A different rate of drying will result, which might lead to hairline cracking (71,72). An inconsistent control on slurry thickness may also cause an uneven thickness of the shell which will lead to a similar problem during dewaxing, the pattern wax expansion stresses may be sufficient to cause fracture to occur.

It was observed that after dewaxing, the moulds had cracks along the perimeter edge. These cracks were repaired by applying two coats of cement to the mould before they were fired in the kiln. Despite repair, these cracks might create a weak spot from which further cracking could develop. When pouring is conducted the hot melt tends to heat the mould causing it to expand. With the presence of the weak spot, the expansion of the mould might cause it to crack at the repaired area.

6.1.1.2.3 Pattern Design

The pattern design was seen to bear sharp edges. Since sharp edges are known to be areas where stress is highly concentrated, the moulds could fail there under shock waves or an explosion. Furthermore, the mould thickness at these edges might be less than at other parts of the mould. This would create a weak area since it is known that the thicker the mould skin the stronger the mould will be (63). During the pouring of the hot melt, different thermal expansion rates will occur due to the thickness difference and may cause failure to occur. This problem could be addressed by rounding off the pattern edges so as to avoid stress concentration and indirectly creating a uniform thickness. Another way of ensuring that the edges could have a better strength would be to apply cement at the outside edges of the mould.

6.1.2 Verification of the Cause/s of Mould Cracking

In the experiment 4.2.1.1 in which a higher pouring temperature was used, it was found that there was no breakage or cracking. With this experiment it was shown that the hypotheses that mould breakage was due to pouring at a higher melt temperature was not in line with other researchers (69,70). It was also shown that the breakage of the mould was not due to a reaction between the magnesium and the mould, or the design of the pattern.

From experiments 4.2.1.2.1 and 4.2.1.2.2, it was expected that the shell which was left for 24 hours would crack during dewaxing since it was expected that within that period, drying would not be sufficient throughout the entire shell. This could lead to an uneven expansion of the shell during dewaxing and thus crack the mould. It was also expected that when using degraded wax for the pouring cup, breakage might be seen since degraded wax is known to have a higher melting temperature (73). With a higher melting temperature, the degraded wax will melt later than the component wax, preventing the component wax from flowing out of the shell thus creating a pressure build-up which will eventually crack the shell. However, neither of these moulds exhibited cracking.

One possible conclusion that can be made is that the 'strength' of the investment casting shell is due to the controlled slurry density which was kept constant at 67–69 on the Baume scale. This produced a uniform thickness on each layer, each time the slurry was applied. This practice avoided the hairline cracking problem mentioned by other researchers (71,72). The uniform thickness minimises differential thermal expansion during dewaxing, thus preventing the shell mould from cracking.

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6.2 THE INFLUENCE OF ATMOSPHERE

6.2.1 The Use of CO_2/SF_6 Gas as an Inhibitor

It is known that a pure carbon dioxide atmosphere inhibits reaction between a magnesium alloy melt and the atmosphere and proved to be very effective at a temperature range of 621°C–676°C, provided that no contamination by air was present(28). Since, in practice, contamination by air cannot be avoided, a small amount of SF_6 gas is required to give an effective protection. In general the protection provided by CO_2/SF_6 is similar to that provided by SO_2 whereby an inert barrier is formed between magnesium and the atmosphere. For the same reason, inhibition within the mould cavity is also possible provided that certain procedures and arrangements are devised to exclude the interaction of the poured melt and the atmosphere contained inside the mould cavity.

6.2.1.1 Flushed Directly inside the Mould

6.2.1.1.1 The Shaw Process Mould

The use of a CO_2/SF_6 gas mixture with the Shaw process mould was ineffective as the castings produced exhibited defects. A possible reason was that insufficient gas was available inside the mould to give a good protection to the surface. The gas that was flushed into the mould was unlikely to remain in the mould since the design of the mould is shallow and open to atmosphere. The problem might not be expected to occur with a ceramic shell mould because its configuration would increase the chance that the gas would remain in the mould.

6.2.1.1.2 The Shell Investment Mould

It was expected that because the shell mould was a less open mould, compared to the Shaw process mould, no surface reaction would be observed on the casting. The finding, however, was not what was expected. The shell investment mould which was flushed with CO_2/SF_6 at a flow rate of 40 litres/min for 30 seconds produced a defective casting. It was very difficult to ensure that the gas that was flushed directly inside the mould remained there until pouring was conducted even though CO_2/SF_6 gas is known to be heavier than air. The gas might not remain inside the mould since it might be pushed out by the dynamic turbulence of the incoming gas. Figure 6.2 shows schematically how this might have occurred.

6.2.1.2 Flushed within an Enclosure

Although the same flow rate and flushing time were used for an enclosed mould, a different result was obtained. A clean metallic finish was produced. It would appear that such a procedure is more reliable and that it ensures that the flushing gas remains both within the enclosure and the mould.

In the initial experiments the enclosure was flushed with a flow rate of 40 litres/min and this produced promising results. However, this flow rate might not be the optimum, economic flow rate and a similar result might be obtained with a much lower flow rate. Based on the experiment conducted where three flow rates were used i.e 25 litres/min, 15 litres/min and 5 litres/min and flushed for 30 seconds, it was found that all of the castings exhibited a clean metallic surface. Among the three castings, the casting which was produced from a mould flushed with 25 litres/ min flow rate had a better surface compared to the other two castings. The other two castings, even though they did not exhibit any reaction, exhibited a dull metallic surface. This seems to indicate that a flow rate of 25 litres/ min and flushing for 30 seconds, supplies sufficient gas to inhibit any metal–mould reaction. Even though the 25 litres/min flow rate was determined as the 'optimum' rate of gas supplied in this experiment, the amount of gas supplied will vary by mould volume. As the volume of the mould is increased the expected amount of gas to be flushed is increased and vice versa.

6.3 THE INFLUENCE OF POURING TEMPERATURE

The aim of this experiment was to provide a preliminary assessment of the extent that pouring temperature affects the mould–metal reaction of magnesium with an alumino–silicate mould. No inhibitor was used. Even though the temperature was varied close to the melting temperature of magnesium alloy (650°C), reaction can still be observed especially at the side of the castings. The reaction was observed to be more severe as the pouring temperature was increased. However, as was observed in figure 5.4, the bottom surfaces of all the castings were clean but dull. This may be a function of the rate of solidification of the castings as the melt came into contact with the mould surfaces. As the melts came in contact with the bottom surface of the mould, and due to the very small quantity of the melt poured, it solidifies immediately, not giving a chance for the reaction to take place. This might also explain why as the pouring temperature was reduced, less reaction was found at the sides of the castings. With a higher pouring temperature, solidification was slower than that for the lower temperature. The increased heating of the mould due to the higher pouring temperature of the melts slows the rate of solidification which increases the time interval during which the metal can react with the mould surface.

6.4 THE USE OF POTASSIUM BOROFLUORIDE (KBF_4) AS AN INHIBITOR

It was observed that the moulds that contained potassium borofluoride did not respond as positively as expected. Potassium borofluoride is known to suppress the reaction in sand moulds and solid investment moulds (5)(6)(40) but it does not seem to work for ceramic shell moulds.

If comparison is to be made between the preparation of a sand mould and a ceramic shell investment mould, the main difference that can be observed is that in preparing an investment mould, the mould is fired in a kiln to a very high temperature, which is normally over 1000°C. Probably, due to this, potassium borofluoride tends to degrade during the firing. After firing tiny brown spots and patches could be seen all around the mould. The degradation of potassium borofluoride in the mould might have impaired its effectiveness as an inhibitor.

In the case of KBF_4 added to the SEMCO coating, the reaction seemed to occur between the coat and the melt. The SEMCO coating is water–based coating. It is a light pink heavy liquid with solid content of approximately 58%. The density of the product is approximately 1.54 kgs/litre. Alumino–silicate is used as the refractory filler material and is added to the amount of approximately 57%. Water and organic additives are added in percentages of 42% and 1% respectively, and 0.15% of a low toxicity biocide is added to prevent any bacterial growth during storage and use.

Even though 2% of KBF_4 was mixed with the coating, it was not able to suppress the mould–metal reaction. The result suggested two possibilities. It might be possible that the reaction was due to the presence of silica in the coating, where it acted as the main source of oxygen for the reaction to take place. The other possibility is that the magnesium melt might have reacted with water which was readily available in the coating. Even though the coated moulds were dried for two days, it is difficult to ensure that the coating is totally dried. Even if the moulds were totally dried before the end of the time allocated, they might pick up moisture from their surroundings. To remove the moisture content by firing the moulds would be inappropriate since in the previous experiments, in which the moulds contained various percentages of KBF_4, positive results were not obtained because of the degradation of the KBF_4 at elevated temperature.

6.5 USE OF DIFFERENT MOULD REFRACTORY MATERIALS

It was anticipated that the magnesium alloy would not be oxidised by three of the materials used – magnesium oxide, calcium oxide and calcium carbonate – since these three materials are stable compounds when compared to magnesium alloy. However, the results showed that the moulds burned away immediately after the magnesium melt was poured into the moulds. It was noticed that while building the moulds and after torching them to remove the alcohol, the moulds were weak and had to be handled very carefully. The condition of the moulds suggested that the bonding might have disintegrated during torching. After the firing process in the kiln, little change was observed in the moulds. Based on these observations it would appear that the use of ethyl silicate for bonding these materials is ineffective and, furthermore, even though these three materials are relatively stable compounds, when bonded with ethyl silicate, a reaction might take place between the magnesium alloy and the silica of the binder. This may also explain why reactions were observed with the other two materials used in the experiment, i.e graphite and silicon carbide.

The experiment using anhydrous calcium sulphate was also unsuccessful. The surfaces of the castings exhibited roughness indicating that reaction had taken place. Generally it was observed that the reaction was more severe as the pouring temperature was increased. It was expected that this material would produce better results since such material was successfully used by other researchers (5)(6) in casting magnesium alloy in block moulds, but with an addition of borofluoride compound. However, Emley (1) stated that the addition of an inhibitor was not necessary in many instances since the mould is fired at red heat before use and the solidification rate of the metal is reasonably fast.

Based on Emley's statement, the reaction might have been due to the unheated mould. It is possible that while the moulds were in the process of cooling down to room temperature they picked up moisture from the atmosphere. The moisture present in the moulds may then have reacted with the magnesium melt, producing a rough surface on the casting surfaces.

The decrease in severity of the reaction as the pouring temperature was decreased was probably due to the speed of solidification of the metal. As the pouring temperature was decreased the rate of solidification was increased. This allowed less time for the reaction to take place when compared to the relatively slower rate of solidification produced by a higher pouring temperature.

6.6 THE ULTIMATE TENSILE STRENGTH (UTS)

6.6.1 The Castings Produced from Flux Method Melts

The mechanical properties of the specimens tested are shown in table 5.1 and are graphically presented in figures 5.9, 5.10, 5.11, and 5.12. As can be seen the highest UTS value was obtained at a pouring temperature and preheat mould temperature of 700°C and 200°C respectively. The UTS value at that particular combination was 136 MPa with 3.3% elongation. In figure 5.9, the trend shows that UTS decreases with an increase in pouring temperature. UTS was at its lowest value at a preheated mould temperature of 100°C. The scatter of data points conceals any trends in most of the graphs plotted.

The values obtained are low when compared to those obtained from sand castings produced in the same alloy (see appendix C). The best UTS value obtained of 136 MPa is 4 MPa below the minimum specified limit. The UTS values for the whole of this group of castings was, on average, 20 MPa below the minimum specified limit even though the percentage of elongation is at par.

A number of factors can affect the mechanical properties of a magnesium alloy casting and these are considered below.

6.6.1.1 Test Specimen Preparation

The most common ways of preparing test bars are by casting to shape or machining from specified test bar shapes. Another way of producing test bars is to cast them using a sprue common with the production casting which will ensure that it is from the same melt and produced under the same conditions as the casting.

Meier (74) observed that machining had a significant effect on tensile properties. The factors included the accuracy of dimensional measurements of the cross section in the gauge length i.e the uniformity of cross section as well as the degree of surface smoothness. However, when taking into consideration the variations due to diameter range and differences between melts, he concluded that the tensile properties of cast–to–shape and machined bars differ only very slightly.

The use of undersize test bars may produce a significant difference in results from those obtained on standard test bars. The reason is the exaggerated effect of small local discontinuities or other minute defects on the results of mechanical tests on undersize test bars.

6.6.1.2 Test Bar Shape

Another important factor is the shape of the test bar cut out of a casting. Although in many cases the shape depends on the dimensions of the casting, it was considered necessary to compare properties obtained on round and flat bars cut out from the same locations of a casting.

The results obtained by Meier (74) in his study on two types of aluminium–magnesium containing alloys, showed that round bars gave much higher tensile test results than did flat bars cut out from the same location.

6.6.1.3. Gauge Length

Another variable which will affect the test results is the gauge length. The effect is particularly on the ductility i.e elongation. Elongation to fracture normally increases with decreasing gauge length (75). This effect is due to the localization of strain during necking, which occurs after maximum load. Meier (74) stated that comparison of elongation values should only be made if measured on test bar specimens with gauge length–to–diameter ratios of constant value.

6.6.1.4. Holding Time

Holding time i.e the time of keeping the alloy in the molten state before pouring, has been claimed to be of critical importance for magnesium alloys. Published data (42)(74) on various Mg–Al–Zn alloys, show that prolonged holding times cause grain coarsening and reduced mechanical properties. Regarding zirconium–containing alloys, it has been observed that longer holding times cause settling out of zirconium and, therefore, larger grain size and lower mechanical properties.

6.6.1.5 Section Thickness

Section thickness per se has no influence on the properties but contributes to changes in the solidification conditions where the quantity of heat given off by a casting during cooling can be dissipated only through the casting surfaces which are in contact with the mould, which in turn affect the alloy structure (grain size).

A study conducted by Couture and Meier (75) found that the mechanical properties of Mg–Al–Zn round bar and plate castings, decreased with increasing bar diameter and plate thickness respectively. This finding supported those of other researchers (76-78) cited by Couture and Meier.

6.6.1.6 Melt Composition

Alloy content in the melt, in particular zirconium, must be closely controlled since it is an essential factor which will affect the mechanical properties of a magnesium–zirconium alloy. Zirconium does not affect the property per se, but it grain refines the metal and in turn produces improved mechanical properties. To achieve effective grain refining, zirconium must be supersaturated in the melt. Meier (42)(74) in his study on Mg–Zn–Zr alloys, found that about 0.6% of soluble zirconium content is essential to achieve high strength.

It is believed that grain refinement by zirconium is caused by the precipitation of zirconium–rich particles as the melt temperature drops. The precipited particles act as nuclei for crystallization. It is, therefore, important that excess zirconium, well above the theoretical value, is present in the melt.

Other elements in the melt, for example Rare Earths, will affect the creep resistance. The suggested working limits are between 2.9% to 3.3% rare earth metals with a nominal of 3.1% (24). With high rare earth content, it may be found that the alloy becomes somewhat brittle.

Zinc is added to the alloy to improve the founding characteristics and also results in a slight improvement in tensile properties. With increasing zinc content, the creep strength in the range of 200°C to 250°C and elongation values are reduced slightly (24).

6.6.1.7 Presence of Discontinuities

6.6.1.7.1 Microshrinkage

The presence of microshrinkage is probably an unavoidable phenomenon, particularly for magnesium alloy. The presence of any form of microshrinkage will act as a nucleating site for crack propagation. This means that both strength properties (ultimate tensile strength) and the percentage elongation will be affected. Every microshrinkage pore that appears in the casting can be considered as a microcrack. This means that in service there is a strong possibility of crack nucleation at these sites.

6.6.1.7.2 Inclusions

Inclusion defects are not uncommon in magnesium alloy castings. Inclusions are mainly due to flux and oxide formed on the surface of the melt when exposed to the atmosphere and they are carried over with the metal stream during pouring. The study by Couture and Meier (75) considered the pouring of magnesium alloy at 850°C, which is significantly higher than the highest temperature reported in this research. However, they demonstrated that high melting temperature promotes oxidation and the likelyhood that castings will contain dross inclusions which will reduce their properties substancially.

6.6.1.7.3 Microporosity

Campbell (79) reported that UTS and elongation in a Mg–Zn alloy system are reduced due to porosity. The reduction is more serious in Mg–11.5 Al alloy especially when the porosity is in the form of layers perpendicular to the applied stress.

6.6.2 Observation and Analysis

From this consideration of possible factors, the most likely causes of low UTS values are as follows.

6.6.2.1 Variation of Dimensions due to Machining of Test Specimen

It was recalled that, during the machining of the specimens, two different turning machines were used. The first was a manually controlled machine where the accuracy depends on the skill of the operator. This method produced a variation in dimensions. The second machine was semi–automatically controlled and produced almost consistent dimensions. It is possible that two sets of specimens with different dimensions were obtained. A variation in dimensions was possible not only because two different machines were used but because they can arise from the same machine when operation is skill–dependent. The suspicion is supported by the literature (74) which stated that variations in the gauge length of tensile test specimens will produce a variation in test results.

7

6.6.2.2 Alloy Content

The composition of the melt may have influenced the large variation in the results. Different percentages of elements present in the melt, particularly zirconium, will give different values. During the casting of the specimens a long holding time, of about 3 $^1/_2$ hours, occurred between each pouring. This was because of the difficulty in maintaining the mould temperature within a range of ± 10 °C. This may have affected the zirconium content. It was found that (42)(74) a longer holding time for zirconium–containing alloys, caused settling out of zirconium and, therefore, larger grain size and lower mechanical properties. It was suggested that the holding period should not be longer than 40 minutes as undue loss of zirconium may result (24). Unfortunately, the composition of the castings could not be analysed since no composition testing facility is available in the University.

6.6.2.3 Inclusions

Visual inspection of the fractured specimens revealed a tell tale sign of oxide inclusion (see figure 6.3). It was found that some 40% of the specimens tested had inclusions at their fracture surfaces, particularly within the specimen gauge length. The oxide formed from the exposure of the melt to the atmosphere and flux used may have been the source of the inclusions. The cause of this effect was puzzling since it was expected that, even though no filtering method was employed, the dross or oxide being less dense than the molten metal, would float on top of the casting i.e beyond the crucial dimensions of the casting. These inclusions would be machined out and thus not affect the properties.

To eliminate or reduce the effect of inclusions on the UTS value, two possible methods were put on trial. The first method used a mild steel strainer to prevent oxide from entering the mould. The strainer was approximately 25mm x 25mm wide and was fitted at the neck of the mould. After the mould knock out, two out of four specimens were observed to be defective due to cold shut. It was noticed that, while pouring was being performed, the mild steel strainer clogged with oxide film which prevented the melt from entering the mould smoothly. It is considered that, with the existing mould design, the use of a mild steel strainer is unsuitable since the neck diameter is rather small at about 30mm. This created turbulence of the melt flow resulting in inclusions in the castings.

The other method employed was casting with a melt prepared by the fluxless method. The castings produced were radiographically examined and found to have some inclusion defects. Based on the exposure intensity, the inclusions occurred close to the surface of the castings. This can be seen from the exposure intensity of the defect on the negative compared to the aluminium wire placed on top of the x–ray film cartridge (see appendix B). Generally, casting by using the fluxless melting method was found to be more convenient in the sense that it produces a reasonably good casting with less problems than were encountered with the use of mild steel strainer.

6.6.3 The Castings Produced from Fluxless Method Melts

The results presented in figure 5.13 show that the ultimate tensile strength is relatively high at a pouring temperature of 700°C and at all mould temperatures i.e 21°C, 100°C, and 200°C. This can also be clearly observed in figure 5.14. The highest average UTS value was observed for the combination of pouring and mould temperatures of 700°C and 200°C respectively, and was 163 MPa with an elongation of 5%, values which were far above the standard sand castings of the same alloy (see appendix C) i.e by 16% for UTS and 67% for elongation. The UTS was lower at pouring temperatures of 730°C and 750°C, at all mould temperatures. As the pouring temperature was increased to 780°C, the UTS increased but was lower than that obtained at a pouring temperature of 700°C. The increase in UTS occurred for all three mould temperatures.

Other properties are graphically presented in figures 5.15 and 5.16.

The findings seem to counter the general understanding of the relationship between solidification rate, grain size and strength. With a higher solidification rate, grain size is expected to be smaller thus increasing the tensile strength. The study shows a trend of high UTS at extreme pouring temperature compared to the intermediate pouring temperatures. The investigation conducted by Herrick (6) indicated that increasing the investment mould temperature from 70°F to 650°F (21°C to 343°C) and the pouring temperature from 1240°F to 1360°F (671°C to 738°C) had a deleterious effect on the average mechanical properties of all the magnesium alloys he investigated.

From microstructural observation and the results reported for grain size in Table 5.4, it is difficult to identify a clear relationship. Grain size is relatively small in the castings produced at the pouring temperature of 700°C compared to that in castings produced at the pouring temperatures of 730°C, 750°C, and 780°C. The values of grain size obtained for the castings produced with a pouring temperature of 750°C appeared to be the most consistent.

Typical microstructures of castings are shown in figures 6.4, 6.5, 6.6 and 6.7.

As the findings show, there is a degree of variability in the data particularly at the pouring temperatures of 750°C and 780°C. The variability ranges from 9% to 22% (see Table 5.4).

6.6.3.1 Variation at a Pouring Temperature of 750°C

From the data, the casting produced at the pouring temperature of 750°C in the second experimental group shows marked differences compared to the two castings produced from different melts but cast at the same pouring temperature. One possible explanation is that, since the second casting was cast from a different melt, there is a possibility that the melt might have received a different melt treatment. However, it was observed that the first and the third experimental group castings which were also from different melts showed similar UTS results.

Metallographic examination of the castings, produced with a pouring temperature of 750°C, and at all mould temperatures, revealed that the casting from the second experimental group had a smaller grain size than those from the other two groups (see figures 6.8, 6.9, and 6.10).

This observation suggests two possibilities. First, the alloy composition, in particular zirconium, might have varied for the first and the third melts. This could have been due to the solubility of zirconium in the melt. In order to produce the grain refining effect, zirconium must be 'sufficiently' soluble in the melt. Previous research on sand cast magnesium alloy, showed that, as the percentage of zirconium solubility increased so did the UTS (42)(74). The same research showed that zirconium may settle out if the melt is subjected to a longer holding time. This could cause larger grain size and lower mechanical properties. A coarse grain size indicates that the soluble zirconium level is low and is a reason for the low properties (24).

Second, since grain size depends on the rate of solidification, it is possible that variation of the mould and pouring temperature could have led to the difference in rate of solidification in the second experimental group casting, thus affecting the grain size. The tolerance for temperature variation for pouring and mould temperatures in the study was ±10°C. Should pouring be conducted at the extreme ends of both mould and pouring temperature ranges, variation in grain sizes might have occurred.

6.6.3.2 Variation at a Pouring Temperature of 780°C

A quite significant variation was also observed for the casting poured at a pouring temperature of 780°C, in particular at a mould temperature of 100°C. At that particular temperature combination, a non–metallic inclusion was observed (see figure 6.11). The inclusion may have resulted from the oxide formed on the surface of the melt. This oxide may have entered the casting while pouring was conducted. However, inclusions were not observed in the castings produced at different mould temperatures. This might have been due to the pouring sequence. At that particular temperature, the melt was first poured into the mould preheated to 100°C and followed by those 21°C and 200°C. The arrangement was due to the quantity of melt in the crucible and the position/height of the mould which hindered pouring. Even though the fluxless method was employed during the preparation of the melt, a thin film of oxide still formed on the surface of the melt. When the melt was poured into the mould preheated to 100°C the oxide film flowed into the mould leaving a clear patch of melt at the spout of the crucible. This enabled a clean metal to be poured into the other moulds.

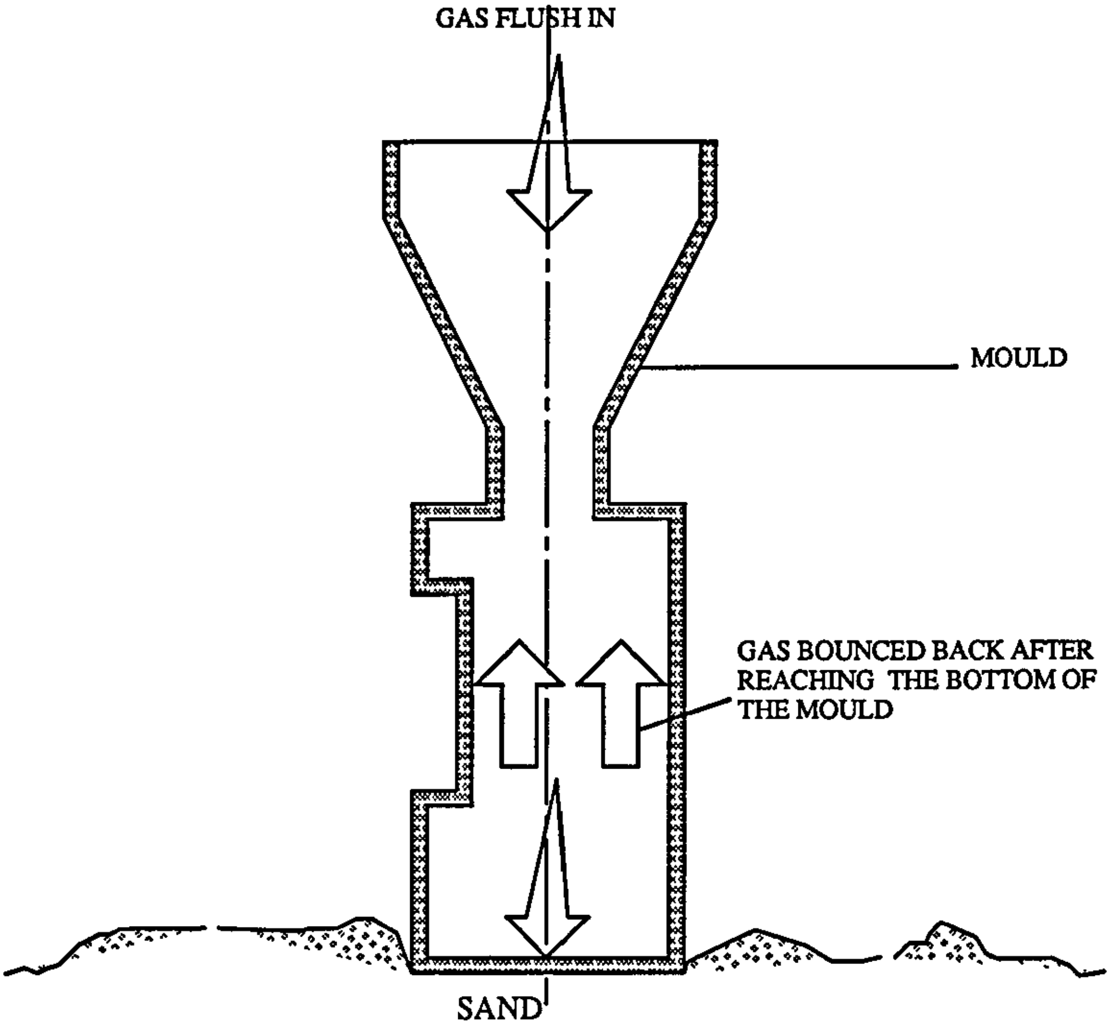

Figure 6.2 : Flushing of gas in a sample ceramic shell mould

Figure 6.3 : Inclusion on one of the fractured surfaces of a casting produced using flux method melting

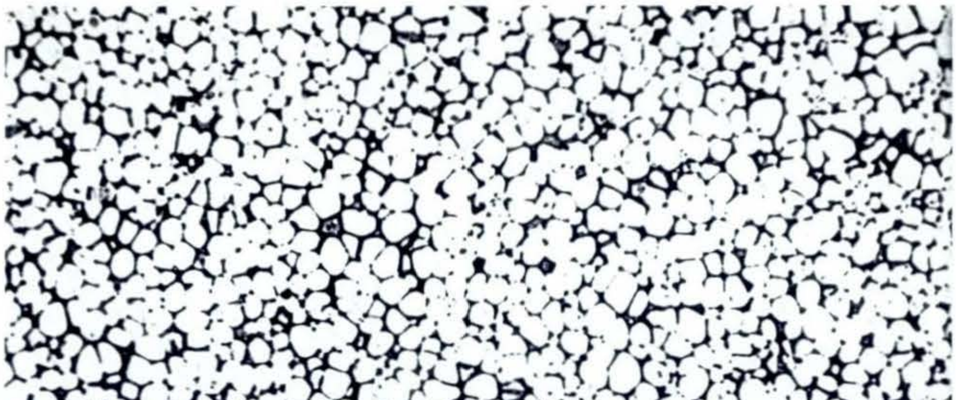

Figure 6.4 : Grain structure of a casting poured at 700°C with a mould temperature of 100°C

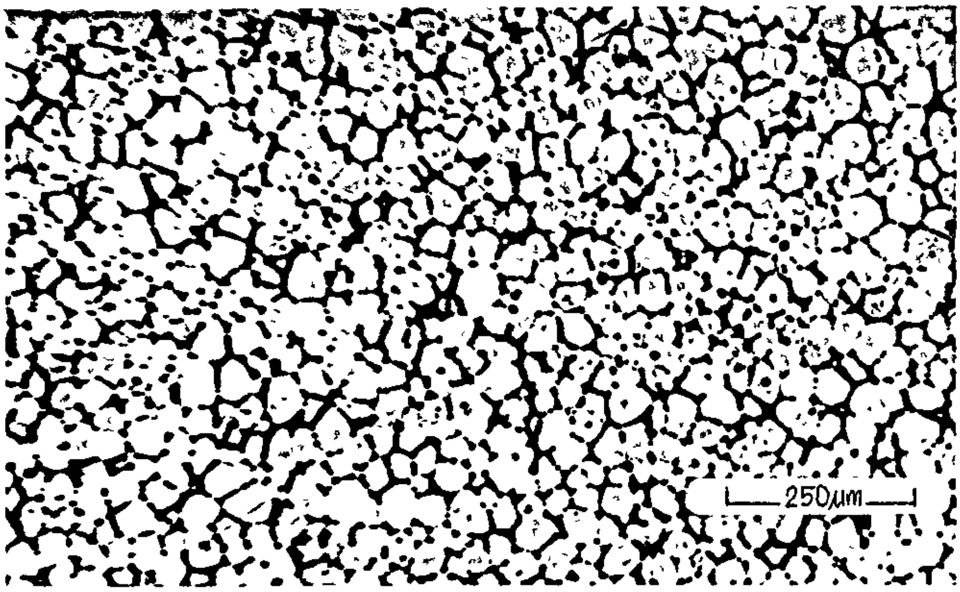

Figure 6.5 : Grain structure of a casting poured at 730°C with a mould temperature of 100°C

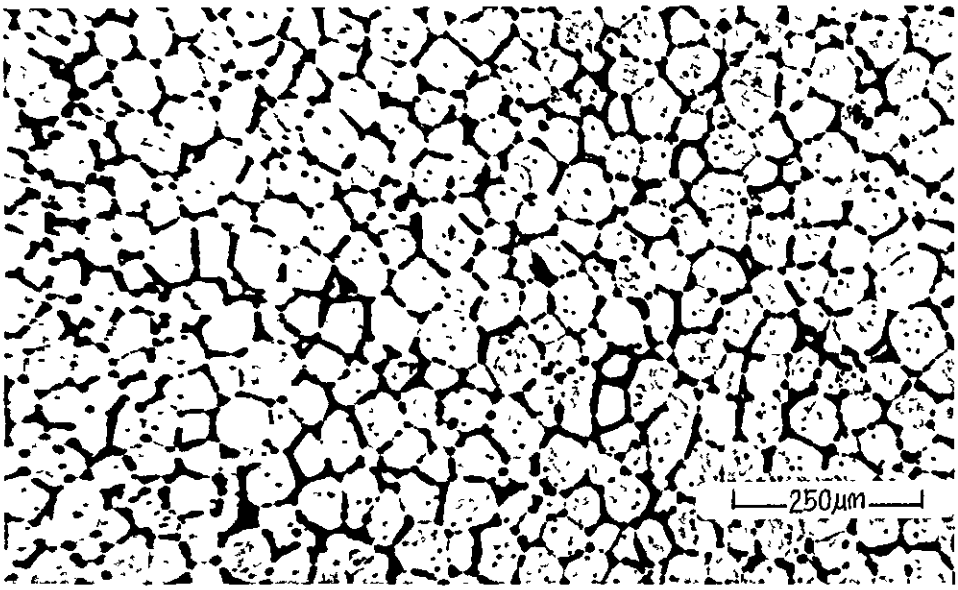

Figure 6.6 : Grain structure of a casting poured at 750°C with a mould temperature of 100°C

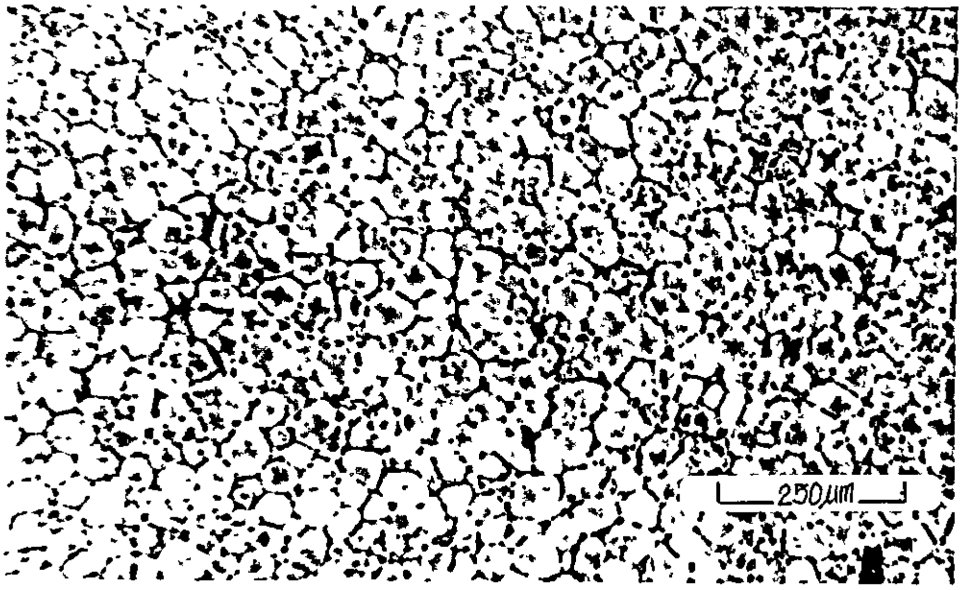

Figure 6.7 : Grain structure of a casting poured at 780°C with a mould temperature of 100°C

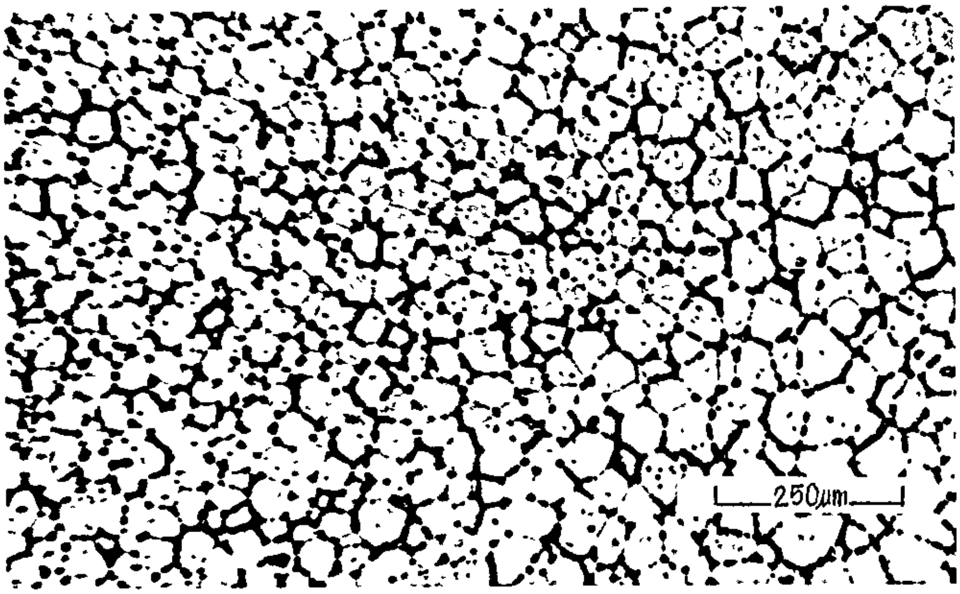

Figure 6.8 : Grain structure of the first batch casting poured at 750°C with a mould temperature of 21°C

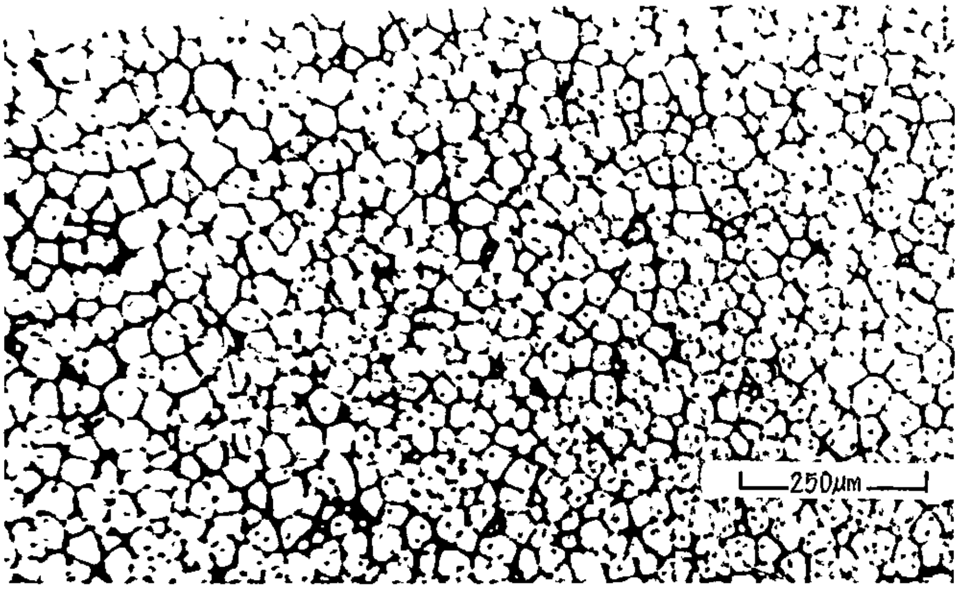

Figure 6.9 : Grain structure of the second batch casting poured at 750°C with a mould temperature of 21°C

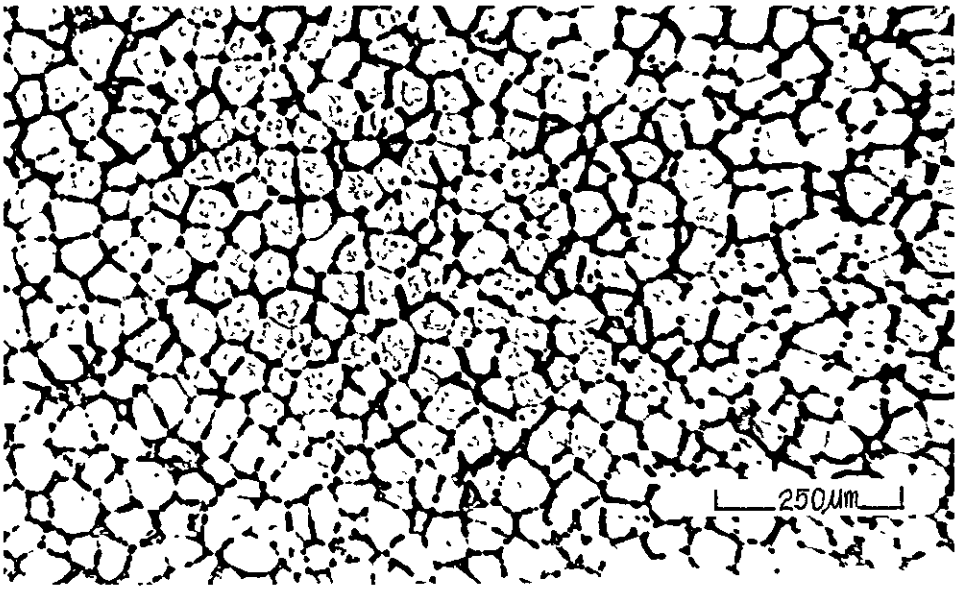

Figure 6.10 : Grain structure of the third batch casting poured at 750°C with a mould temperature of 21°C

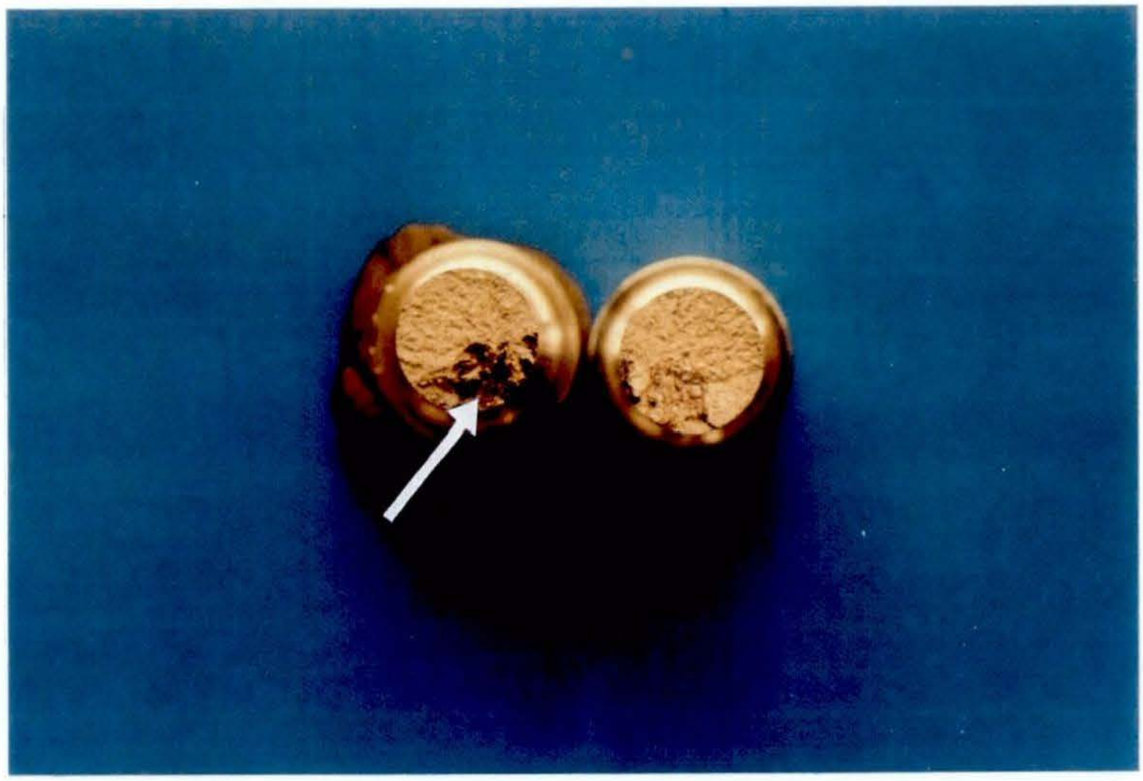

Figure 6.11 : Inclusion observed on casting poured at 780°C with a mould temperature of 100°C

CHAPTER 7

CONCLUSIONS

Based on the experiments conducted, the following conclusions can be drawn:

1. Magnesium alloy (ZRE1) can be successfully poured into shell investment moulds with the use of CO_2–2% SF_6 as the protective gas. In order for the protection to be effective, the mould must be flushed within an enclosure. The minimum amount of gas required for melt protection using the fabricated enclosure was 25 litres/min. flushed for 30 seconds.

2. The problem of mould cracking was avoided by controlling the slurry density (viscosity) at 67–69 on Baume scale. By ensuring a constant slurry viscosity, an even ceramic shell thickness could be obtained. An even shell thickness provided a uniform thermal expansion and this avoided shell cracking.

3. Potassium borofluoride (KBF_4) was not effective as an inhibitor for ceramic shell investment casting. This may be because the substance degraded during high temperature firing thus losing its function as a protective material.

4. The use of magnesium oxide, calcium oxide, calcium carbonate, graphite, silicon carbide, and anhydrous calcium sulphate as alternative mould refractory materials to alumino–silicate did not prove effective. The first three mould refractory materials disintegrated when the magnesium melt was poured into the moulds. Castings produced in graphite and silicon carbide moulds showed signs of heavy reactions on their surfaces whilst castings produced in anhydrous calcium sulphate exhibited an increase in reaction severity as the pouring temperature was increased.

5. Pouring and mould preheat temperatures were found to affect the ultimate tensile strength of the magnesium alloy under investigation with the extreme temperatures i.e 700°C and 780°C producing higher UTS values than the intermediate temperatures.

6. The highest average value of UTS was 163 MPa. This was obtained from castings poured using metal melted by the fluxless method at pouring and mould temperatures of 700°C and 200°C respectively. The average percentage elongation for this particular temperature combination was 5%. Comparatively, the values obtained are approximately 16% and 67% higher than the standard UTS and percentage elongation values respectively, quoted for sand castings produced in the same alloy. The UTS value was at its lowest for the pouring and mould temperature combination of 730°C and 200°C respectively and was recorded at 128MPa.

The UTS value recorded from the castings produced using metal melted by the flux method was approximately 3% below the standard sand cast UTS value. The best recorded UTS value was 136 MPa at the pouring and mould temperature combination of 700°C and 200°C respectively, with the percentage elongation at par. The pouring and mould temperature combination of 730°C and 100°C respectively produced the lowest recorded UTS value of 105 MPa.

7. In the attempt to control inclusions, the fluxless method was found to be a more effective alternative than the use of a mild steel strainer The mild steel strainer fitted at the neck of the mould clogged and hindered the smooth flow of liquid melt into the mould. This produced defective castings due to cold shut.

CHAPTER 8

SUGGESTIONS FOR FURTHER WORK

The objective of the research was to develop a suitable mould material or inhibitor procedure to overcome the reaction between the alloy and the oxide of the refractory material. Further research could be conducted to refine and extend the current work.

1. In this study the use of fluxless melting was not found to be effective in producing a clean casting. It is proposed that melting and pouring could be conducted in an enclosed environment in order to exclude the atmosphere from the melt thus minimising the oxidation of the melt surface. Argon might be used instead of CO_2/SF_6 because it is much cheaper.

3. Since the study concentrated on a narrow range of mould temperatures, further study could be undertaken with an extended range of mould temperatures, especially higher temperatures, because in practice higher mould temperatures are normally used to produce thin section castings.

4. The effect of holding time was not considered during this research. However, variations in holding time may have influenced the results. Further research could be pursued to establish the effect of holding time on UTS values.

5. In the study, a simple gating arrangement was used. This arrangement may not have ensured that adequate feeding was obtained and this may have some effect on the UTS value. A study should be conducted to optimise the gating and feeding arrangements.

6. Variations in section thickness occur in most casting designs. The present study was limited to a casting of uniform thickness. A study could be conducted to establish the effect of section variation on the mechanical properties of magnesium alloy.

7. As was stated in Chapter 6, the UTS value may have been affected by grain coarsening which might been have due to the temperature conditions or specifically the rate of solidification. The rate of solidification is a function of the rate at which heat is removed from the casting. A study could be conducted to determine whether the thickness of the ceramic shell affects the rate of heat transfer and, therefore, the mechanical properties of the casting.

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APPENDIX A

Table A1 : Tensile test results of castings produced by the fluxed melting method

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
700	21	1	9 04	132	30	3	3
		2	9 08	131	35	3	3
		3	9.05	126	38	2	3
		4	9.03	–	33	3	3
700	100	1	9.04	123	27	2	2
		2	9.05	122	27	1	1
		3	9.06	80	28	1	1
		4	9.07	126	35	1	2
700	200	1	9.15	135	30	3	3
		2	9.15	146	32	4	4
		3	9.13	128	27	3	3
		4	–	–	–	–	–

Table A1: Tensile test results for castings produced by the fluxed melting method (cont$^{\underline{n}}$)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
730	21	1	9.05	138	37	4	–
		2	9.03	137	53	1	3
		3	9.08	131	34	2	3
		4	9.07	141	38	3	3
		5	9.04	–	19	–	–
		6	9.12	122	34	4	3
		7	9.13	116	27	4	3
		8	9.13	120	24	4	3
		9	9.05	124	37	1	2
		10	9.07	126	38	2	2
		11	9.06	134	36	3	2
		12	9.07	124	32	2	2
730	100	1	9.01	91	37	1	1
		2	9.05	80	43	1	1
		3	9.07	116	31	2	2
		4	9.07	114	40	2	2
		5	9.04	115	22	6	3
		6	9.07	114	26	3	3
730	200	1	9.07	106	26	3	3
		2	9.08	120	31	3	3
		3	9.08	120	8	4	4
		4	9.07	118	26	3	3
		5	9.08	131	34	1	2
		6	9.08	143	40	4	3
		7	9.05	115	50	1	3
		8	9.04	150	41	4	4

Table A1: Tensile test results for castings produced by the fluxed melting method (contⁿ)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
750	21	1	9.14	118	20	4	3
		2	9.08	112	19	4	1
		3	9.13	111	20	4	3
		4	9.18	102	22	3	3
		5	9.13	130	31	3	1
		6	9.12	130	26	2	2
		7	9.13	134	35	3	2
		8	9.12	124	35	2	2
750	100	1	9.06	124	21	3	4
		2	9.05	113	22	3	3
		3	9.07	130	22	6	2
		4	9.05	111	20	4	3
		5	9.10	119	37	2	2
		6	9.06	124	29	2	2
750	200	1	9.12	115	25	5	3
		2	9.14	122	23	5	3
		3	9.14	124	20	5	3
		4	9.14	118	19	4	2
		5	9.06	124	42	2	3
		6	8.80	142	35	3	2
		7	9.04	148	45	5	3
		8	9.07	133	31	3	1

Table A1: Tensile test results for castings produced by the fluxed melting method (contn)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
780	21	1	9.02	129	21	4	4
		2	9.15	110	20	3	3
		3	9.14	86	21	3	2
		4	–	–	–	–	–
780	100	1	9.07	118	22	3	4
		2	9.07	122	20	3	3
		3	9.05	122	20	5	3
		4	9.05	111	18	3	3
780	200	1	9.04	119	16	4	3
		2	8.99	120	15	4	3
		3	9.00	118	18	4	2
		4	9.05	118	19	4	3

Table A2 : Tensile test results for castings produced by the fluxless melting method

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
700	21	1	9.08	161	44	5	3
		2	9.08	160	36	5	3
		3	9.08	157	45	5	3
		4	9.08	159	36	6	4
		5	9.08	139	49	3	4
		6	9.06	156	43	4	4
		7	9.09	158	38	3	3
		8	9.07	120	34	2	2
		9	9.08	145	33	3	3
		10	9.08	111	32	2	2
		11	9.07	143	44	4	3
		12	9.07	164	42	4	5
700	100	1	9.06	152	33	4	4
		2	9.07	158	35	5	4
		3	9.07	159	37	5	4
		4	9.07	163	44	5	4
700	200	1	9.08	160	35	5	5
		2	–	–	36	–	–
		3	9.09	162	45	5	5
		4	9.07	163	51	5	5
		5	9.08	161	41	5	3
		6	9.07	170	43	6	4
		7	9.08	163	42	5	5
		8	9.07	163	38	5	4

Table A2:Tensile test results for castings produced by the fluxless melting method (cont$^{\underline{n}}$)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
730	21	1	9.07	141	33	4	3
		2	9.07	145	26	4	3
		3	9.09	140	39	4	3
		4	9.07	148	42	4	3
730	100	1	9.08	141	33	4	2
		2	9.08	136	33	3	3
		3	9.07	110	46	2	2
		4	–	–	–	–	–
730	200	1	9.08	116	45	2	2
		2	9.09	144	41	4	4
		3	9.11	120	42	2	2
		4	9.08	130	30	3	3

Table A2:Tensile test results for castings produced by the fluxless melting method (cont$^{\underline{n}}$)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
750	21	1	9.09	116	48	3	3
		2	9.09	129	48	4	3
		3	9.10	121	36	3	3
		4	–	–	–	–	
		5	9.10	152	48	4	3
		6	9.08	148	36	4	3
		7	9.08	148	39	4	2
		8	9.09	127	42	2	2
		9	9.09	113	42	3	2
		10	9.07	–	43	–	–
		11	9.11	135	35	3	3
		12	9.07	117	41	2	2
750	100	1	9.12	134	40	4	3
		2	9.12	128	41	4	3
		3	9.13	134	35	4	3
		4	9.11	134	42	4	3
		5	9.10	145	41	4	3
		6	9.11	146	53	3	3
		7	9.09	152	46	4	4
		8	9.11	143	49	3	3
		9	9.08	93	49	2	2
		10	9.11	129	38	3	3
		11	9.09	135	38	4	2
		12	9.10	135	30	5	2

Table A2:Tensile test results for castings produced by the fluxless melting method (cont$^{\underline{n}}$)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
750	200	1	9.09	127	37	4	3
		2	9.09	129	39	4	3
		3	9.10	131	47	4	3
		4	9.08	91	37	1	2
		5	9.09	141	38	3	3
		6	9.09	141	48	3	4
		7	9.09	142	38	4	4
		8	9.08	123	29	2	2
		9	9.11	–	40	–	–
		10	9.08	128	30	3	4
		11	9.13	131	31	4	3
		12	9.08	108	42	4	2
780	21	1	9.11	109	36	1	2
		2	9.11	157	39	5	5
		3	9.10	144	33	4	4
		4	9.10	113	41	1	2
		5	9.09	156	36	5	4
		6	9.09	149	36	4	3
		7	9.09	137	29	3	3
		8	9.08	148	33	3	4

Table A2:Tensile test results for castings produced by the fluxless melting method (contⁿ)

Pouring Temp. (°C)	Mould Temp. (°C)	Specimen	Dia. (mm)	UTS (MPa)	Modulus, E (GPa)	% Elong.	% R.A
780	100	1	9.10	160	39	5	5
		2	9.11	136	33	4	4
		3	9.10	165	26	6	5
		4	–	–	–	–	–
		5	9.08	90	27	2	1
		6	9.07	138	35	3	3
		7	9.09	140	34	3	4
		8	9.08	129	33	2	3
780	200	1	9.09	169	34	6	5
		2	9.09	133	29	4	4
		3	9.10	149	37	5	5
		4	–	–	–	–	–
		5	9.08	163	38	6	3
		6	9.10	160	33	5	4
		7	9.10	145	32	3	4
		8	–	–	–	–	–
		9	9.08	142	28	4	3
		10	9.09	140	34	4	3
		11	9.08	143	24	5	4
		12	9.07	135	20	4	3

APPENDIX B

Figure B1 : Inspection of specimens cast using fluxless method by radiographic examination.

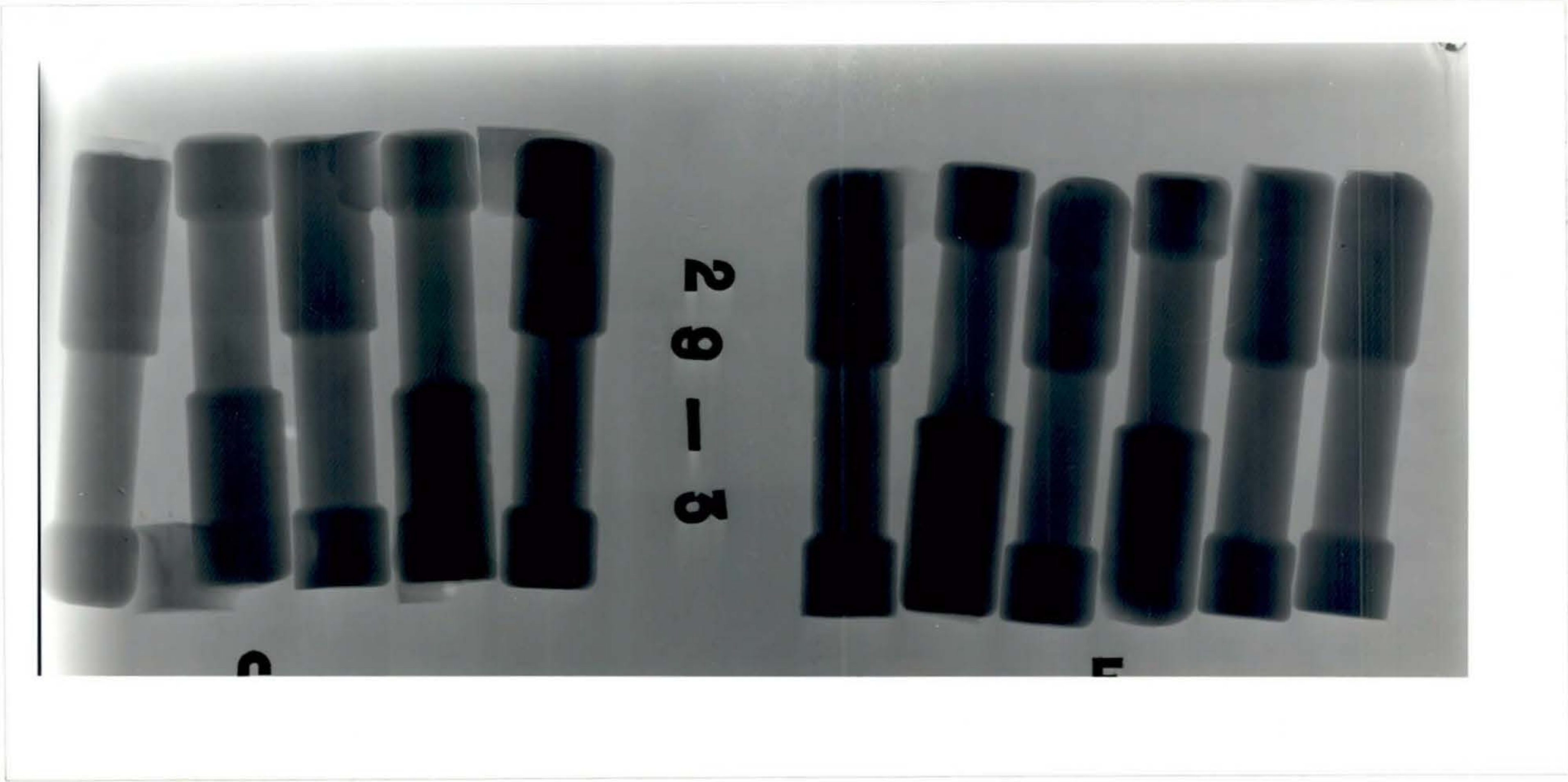

Figure B1 : Inspection of specimens cast using fluxless method by radiographic examination (Cont$\underline{^{d}}$)

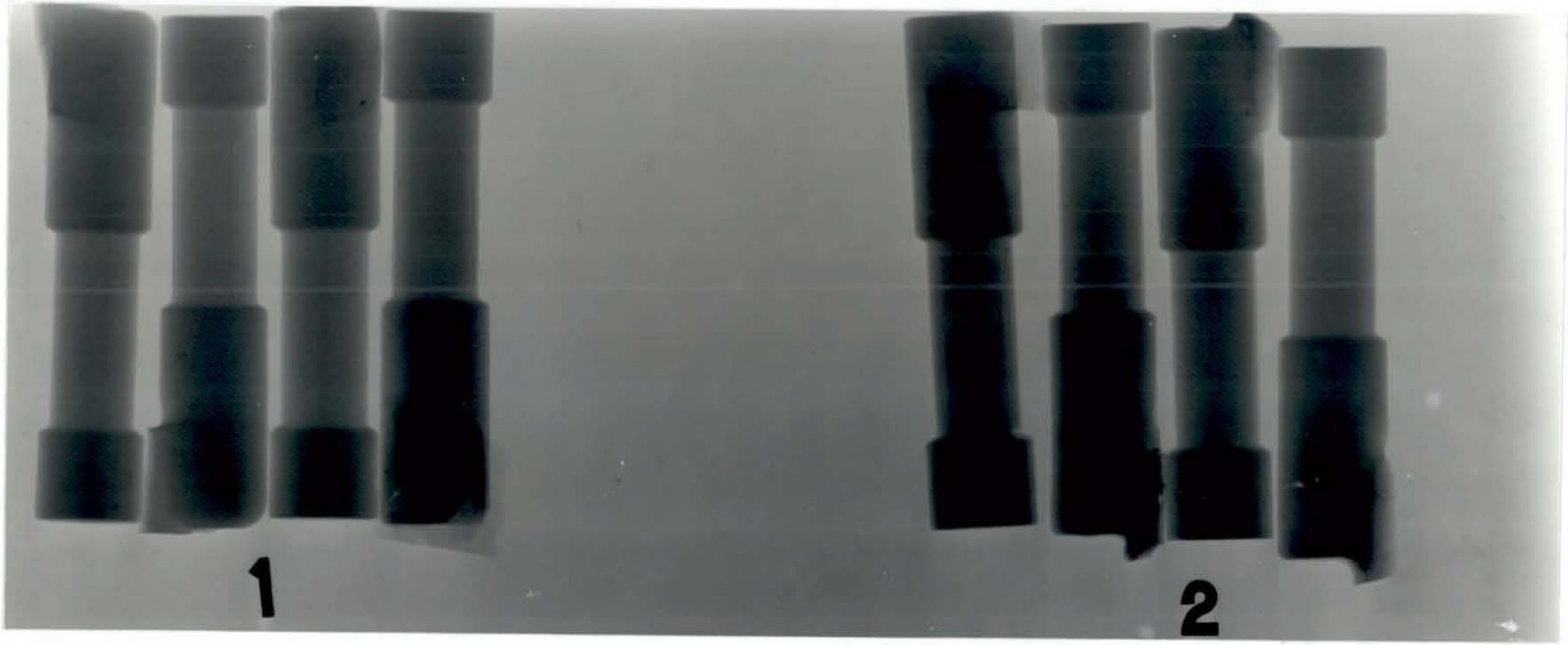

Figure B1 : Inspection of specimens cast using fluxless method by radiographic examination (Cont^{d})

APPENDIX C

Table C1 : Magnesium–Zirconium alloys properties (Smithells Metals Reference Book)

Inherently fine grained (0.015–0.035 mm chill cast)			
Elektron designation	Z5Z	RZ5	ZRE1
ASTM designation	ZK51	ZE41	EZ33
Specifications BS 2970: 1989	MAG4TF(GP)‡	MAG5TF(SP)	MAG6TF(SP)
BSS L series	2L 127	2L 128	2L 126
Equivalent DTD			
Composition % (Single figures indicate maximum)			
Zinc	3.5–5.5	3.5–5.5	0.8–3.0
Silver			
Rare earth metals		0.75–1.75	2.5–4.0
Thorium		--	
Zirconium	0.4–1.0	0.4–1.0	0.4–1.0
Copper	0.03	0.03	0.03
Nickel	0.005	0.005	0.005
Iron			
Silicon			
Manganese			
Material properties			
Founding characteristics	Good in sand and permanent moulds§	Good in sand and permanent moulds	Excellent in sand and permanent moulds
Tendency to hot tearing	Marked	Some	Little
Tendency to micro porosity	Very appreciable	Virtually none	None
Castability¶	B	A	A
Weldability (Ar-Arc Process)	Not recommended	Moderate	Very good
Relative damping capacity**	B/C	B/C	B
Strength at elevated temperature††	C	B	A
Resistance to creep at elevated temperature	Poor	Moderate	Good up to 250°C
Corrosion resistance	Moderate	Moderate	Moderate
Density, g cm^{-3} (20°C)	1.81	1.84	1.80
Liquidus, °C	640	640	640
Solidus, °C	560	510	545
Casting temperature range, °C	720–810	720–810	720–810

Table C1 : Magnesium–Zirconium alloys properties (Smithells Metals Reference Book)(cont$^{\underline{d}}$)

Heat treatment			
Solution			
Time, h			
Temperature °C			
Cooling			
Precipitation			
Time, h	16	2 followed by 16	8
Temperature, °C	180	330 180	200
	Air cool	Air cool after each	Air cool
Post weld stress relief			
Time, h	2	Precipitation	10
	330	treatment affords	250 max
	to precede precipitation treatment	s/relief	Air cool
Mechanical properties sand cast SI units (Imperial units in brackets)			
Tensile strength min, MPa (tonf in^{-2})	230 (14.9)	200 (13.0)	140 (9.1)
0.2% proof stress min, MPa (tonf in^{-2})	145 (9.4)	135 (8.7)	95 (6.2)
Elongation, % ($5.65\sqrt{S_0}$) min	5	3	3
Mechanical properties chill cast SI units (Imperial units in brackets)			
Tensile strength min, MPa (tonf in^{-2})	245 (15.9)	215 (13.9)	155 (10.0)
0.2% proof stress min, MPa (tonf in^{-2})	145 (9.4)	135 (8.7)	110 (7.1)
Elongation, % ($5.65\sqrt{S_0}$) min	7	4	3
Applications	High strength plus good ductility Not suitable for spidery complex shapes	For high strength pressure tight applications	High degree of pressure tightness at room and elevated temperatures