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Chapter 3

IMPROVED SOL BASED CERAMIC MOULDS FOR USE IN INVESTMENT CASTING

by
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Faculty of Engineering
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CHAPTER THREE

3.1: Scanning Electron Microscopy (SEM)

Scanning electron microscopy was used to view the structure of several investment casting mould sections in order to develop an understanding of the basic refractory and binder distributions. The technique was also used to characterise the refractory materials and to evaluate the effects of metal casting and thermal cycling upon the overall mould structure.

Specialist X-ray analysis techniques, such as wavelength dispersive and energy dispersive, allowed both quantitative and qualitative elemental compositions to be determined. This was used effectively to quantify silica binder compositions, to identify phase separations within refractory materials and to analyse metal mould interactions.

The instruments used in experimental work were a JEOL 840A Scanning Electron Microscope and an ISI-100A Scanning Electron Microscope.

3.1:1 The Principles of SEM

Scanning electron microscopy uses an electron beam which scans the surface of a sample causing the emission of low energy secondary and backscattered electrons. These electrons are collected to form an image of the sample surface(117). A schematic diagram of the basic structure of the instrument is shown in Figure 39 (the electron beam path is under a vacuum).

Electrons are produced by a tungsten filament gun and focused using condenser lenses to produce a beam 5 nm - 1 μm in diameter, using an accelerating voltage of 1-30kV and an electron beam current from 10^{-6} to 10^{-12} Amps. The beam is scanned on the area of interest using scan-coils and the secondary electrons emitted produce an image of the sample on a display cathode ray tube (CRT). The magnification is altered by changing the scanned area of the sample and focus is obtained by altering the strength of the condenser lenses. The resolution of the image is affected by the gun conditions (voltage, current and source size), working distance, collection efficiency of the detector and the beam collimation (final aperture size).

SEM is particularly useful for observing surface features and fracture surfaces and is also widely applicable in general microstructural observations.

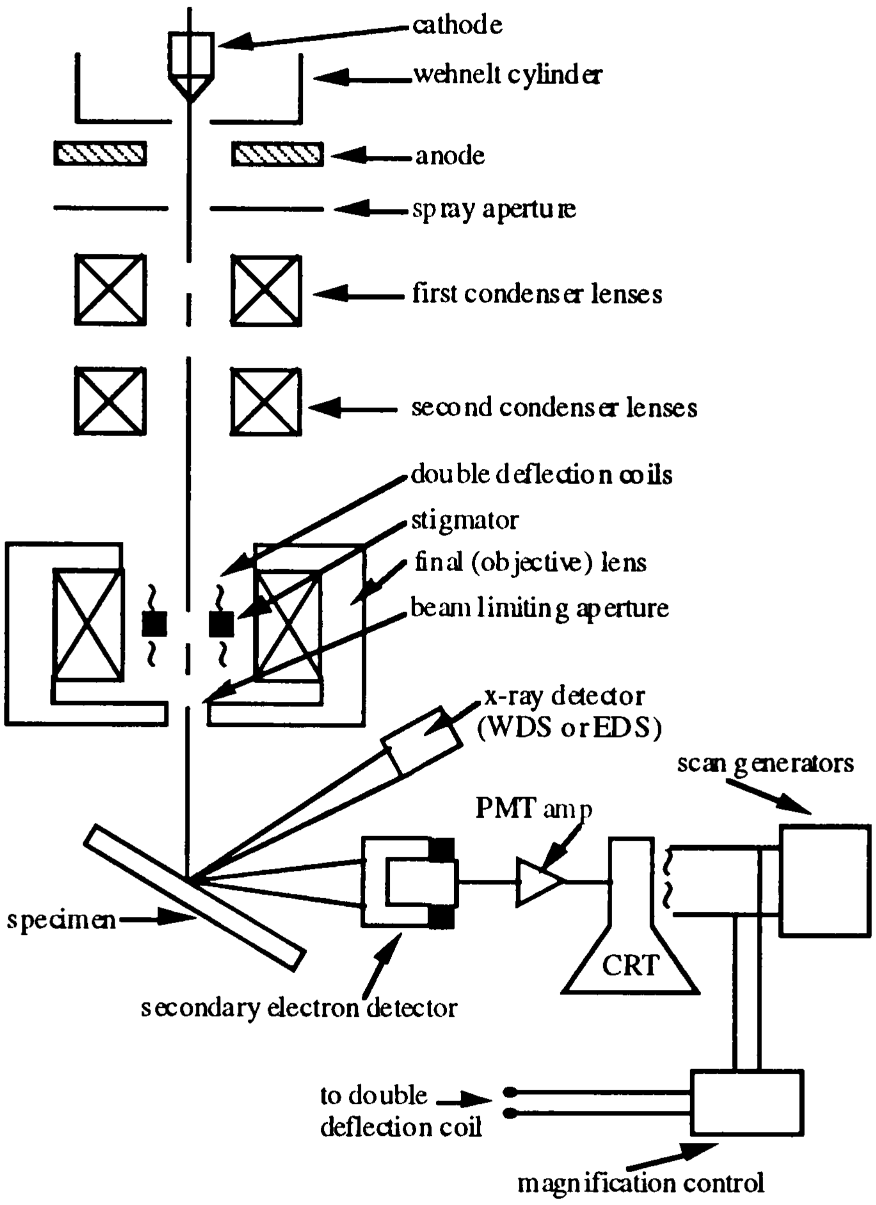


Figure 39: Schematic illustration of an SEM microscope

3.1.2: Preparation of Samples

There were three stages in the preparation of the ceramic SEM samples; small amounts of material were mounted into resin, the surfaces polished with successively finer grades of carborundum paper, finishing with a short polish using very fine silica sol particles in an aqueous medium (pH 10) or diamond paste. Finally the samples were carbon coated and mounted into aluminium stubs ready for inspection in an SEM microscope. These stages are described in detail below.

Mounting

1. Prepare the sample to be mounted and place this centrally on the mould base (Figure 40).

2. Pour sufficient plastic resin (Metset resin type FT-30-60% styrene) into a measuring/mixing jug, add the required amount of hardener (methylethylketone peroxide-50% in plasticiser) and mix well using a wooden spatula. In this case use 5 drops of hardener to 10ml of resin.

3. Pour the mixture into the moulds to the desired level and immediately add an identification label with the appropriate marking (Figure 41). For refractory sand samples a small amount of the material is added to the resin at this stage and stirred until the resin begins to harden. This ensures an even dispersion of sample throughout the resin.

4. Leave the resin to set and harden being careful not to disturb the mould.

5. When fully set eject from the mould by pressing the moulding out from the unflanged end of the mould cylinder.

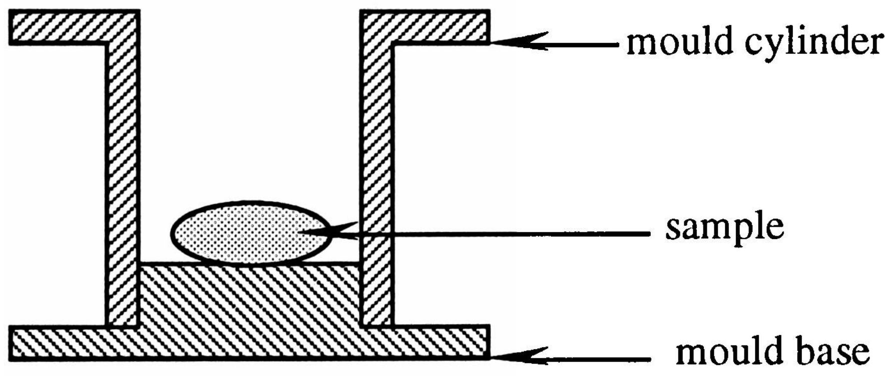


Figure 40: Cross sectional view of the mould

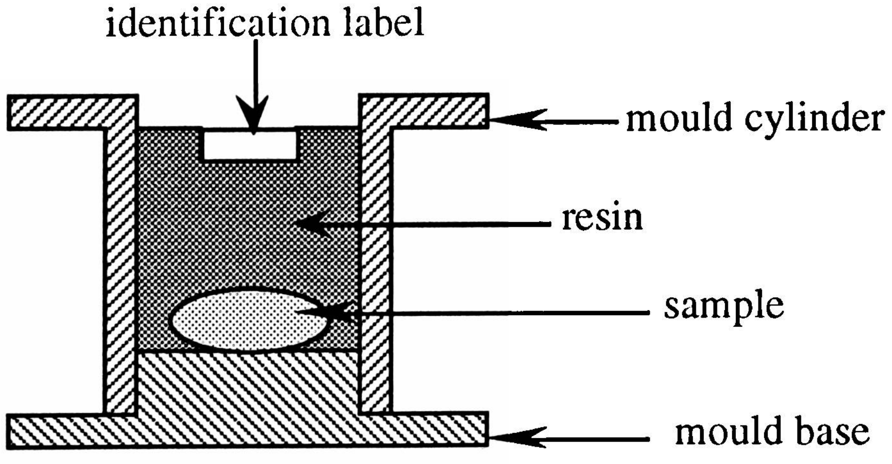


Figure 41: Cross sectional view of a filled mould.

Polishing

The ceramic samples were first polished by hand on successively finer grades of 'wet or dry' carborundum (Silicon Carbide) abrasive paper, using distilled water and a glass support surface. Grade P150, the coarsest, was the first paper used, followed by P280, P400, P800 and finishing with the finest grade P1200. At each stage the polishing was continued until all scratches produced by the previous grade were removed.

After this initial hand polishing the samples were completed using Syton W30 polishing fluid on a Multipol 2 polishing machine for 24 hours. This slow polish, using a silica sol, produces a finely polished sample surface due to the fine particle size of the polishing fluid (approx. 150 nm) and the ability of the liquid to dissolve any small portions of the sample that may become dislodged.

The preparation of metal samples was similar, with the exception that diamond paste was used as the final polish medium. Two grades of paste, 6 μm and 1 μm, were used in conjunction with a suitable polishing cloth and lapping fluid on a Mecapol polishing wheel. Again the duration of each stage was dependent upon the removal of all scratches from the previous grade.

Coating

After polishing the samples were prepared for the SEM. Non-conducting specimens, such as ceramics, provide no pathway for the removal of electrons produced by an incident electron beam. Thus, the sample surface rapidly becomes charged and the image quality decreases. In order to facilitate the removal of these excess electrons, the surface of the sample is made conducting by the deposition of a thin film of conducting medium.

To examine the surface topography, the sample can be sputter-coated with gold, but if analysis of elements is required then a conductive coating with a low atomic number is required to prevent detection of this coating by the analyser. Carbon is such a coating and a thin film is deposited by vapour deposition.

After deposition, the sample is mounted onto an aluminium stub and a small strip of silver "dag" is painted from the edge of the sample, down the side of the resin mould, to connect with the stub. This silver provides an 'electron pathway' by which the excess electrons are removed from the surface of the sample.

3.1.3: Analysis Techniques

Element and phase analysis can be carried out in the SEM due to interaction of the electron beam with the surface of the sample producing characteristic X-rays. Inelastically scattered electrons produce these X-ray patterns by two distinct routes:

1. The white X-ray continuum is produced by the gradual deceleration of an electron beam.
2. The interaction of the electron beam with inner shell electrons (K and L shells) in which an outer shell electron falls into the vacancy created and an X-ray is produced.

Specific energy transitions are involved when electrons move between energy levels, i.e. between electron shells, and the energies of the X-rays emitted are specific to the element concerned. A spectrum is thus produced which is characteristic of the element (Figure.42) making identification possible. By comparison with tabulated data and specific element standards, both qualitative and quantitative analysis can be carried out.

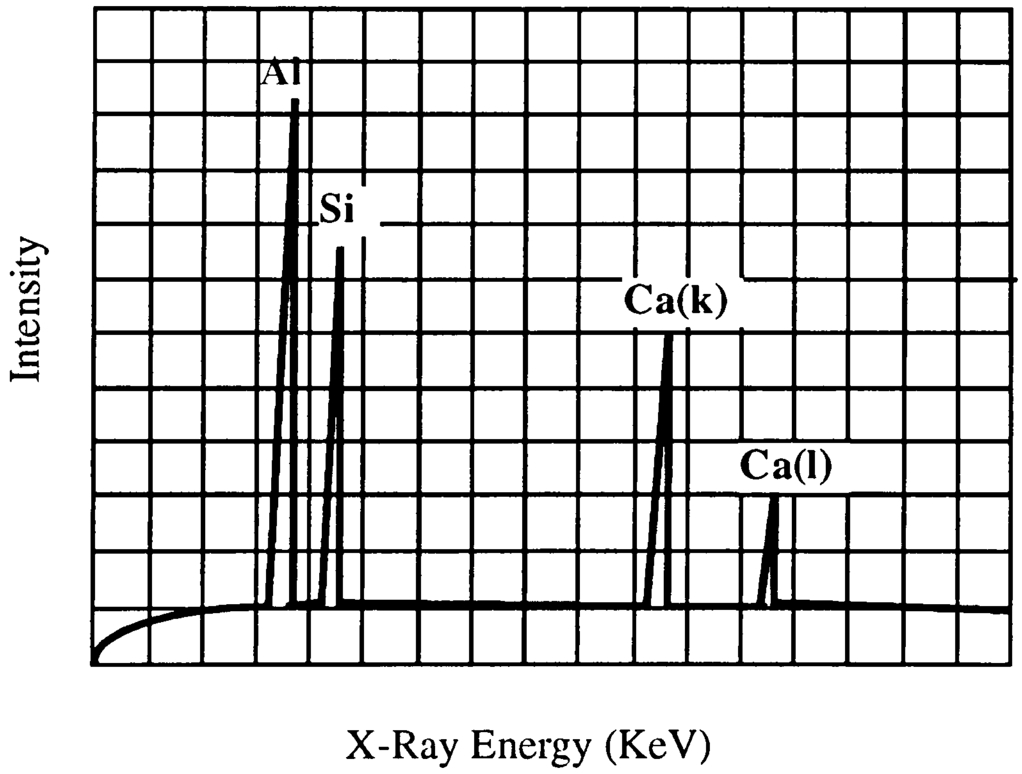


Figure 42: Characteristic X-Ray Spectrum

The following analytical techniques were used for the identification of phases within samples:

Energy Dispersive X-Ray Analysis (EDX)

The X-rays produced in a scanning electron microscope, due to interaction between the electron beam and the sample, are collected and used for EDX analysis. The detector used consists of a Lithium-drifted Silicon crystal mounted in a cryostat and separated from the microscope chamber with a thin Beryllium window (Figure 43). Each X-ray photon is detected and amplified individually using an FET (field effect transistor) and pulse processor. The pulse-train is accumulated and processed using a multi-channel analyser, to display a histogram showing X-ray energy horizontally and intensity vertically (Figure 42). The characteristic energies of these K and L line peaks can be compared with tabulated data for elements enabling an analysis to be carried out.

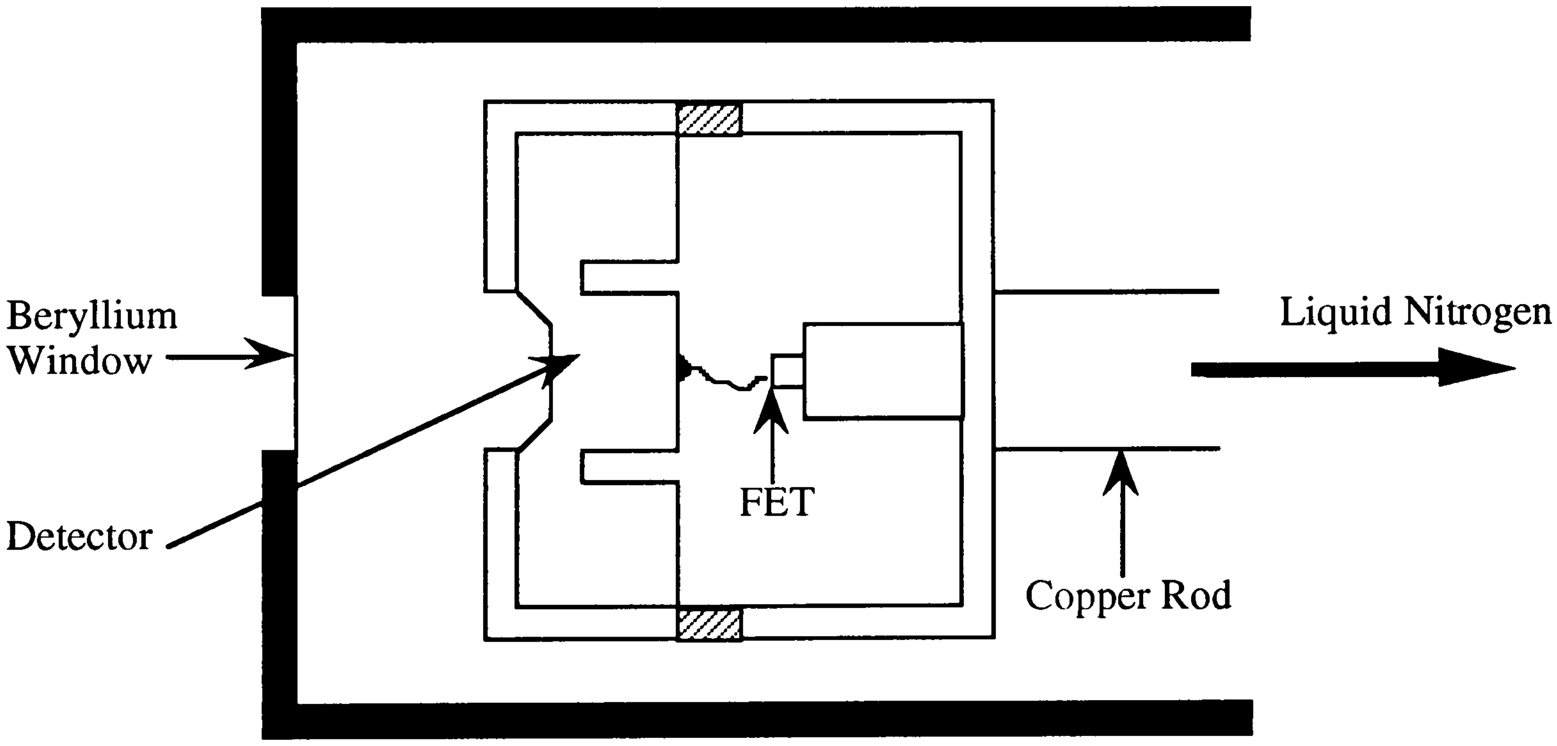


Figure 43: Si(Li) detector assembly (schematic)

Wavelength Dispersive X-Ray Analysis (WDX)

Normal EDX systems cannot detect certain ‘light’ elements (C,B,O,N) due to the presence of the Be window, which absorbs the characteristic X-rays of these elements. WDX detectors have no Be window and rely upon the use of specific crystals (such as LiF) which diffract the X-ray beam via a proportional counter. A schematic illustration of a WDX spectrometer is shown in Figure 44. The system is essentially geometric and relies on the focussing effect of the crystal and the mechanical positioning of the detector to select the required analysis peak. A variety of crystals are needed for detection of different elements, with a typical WDX system having at least 4 separate crystals.

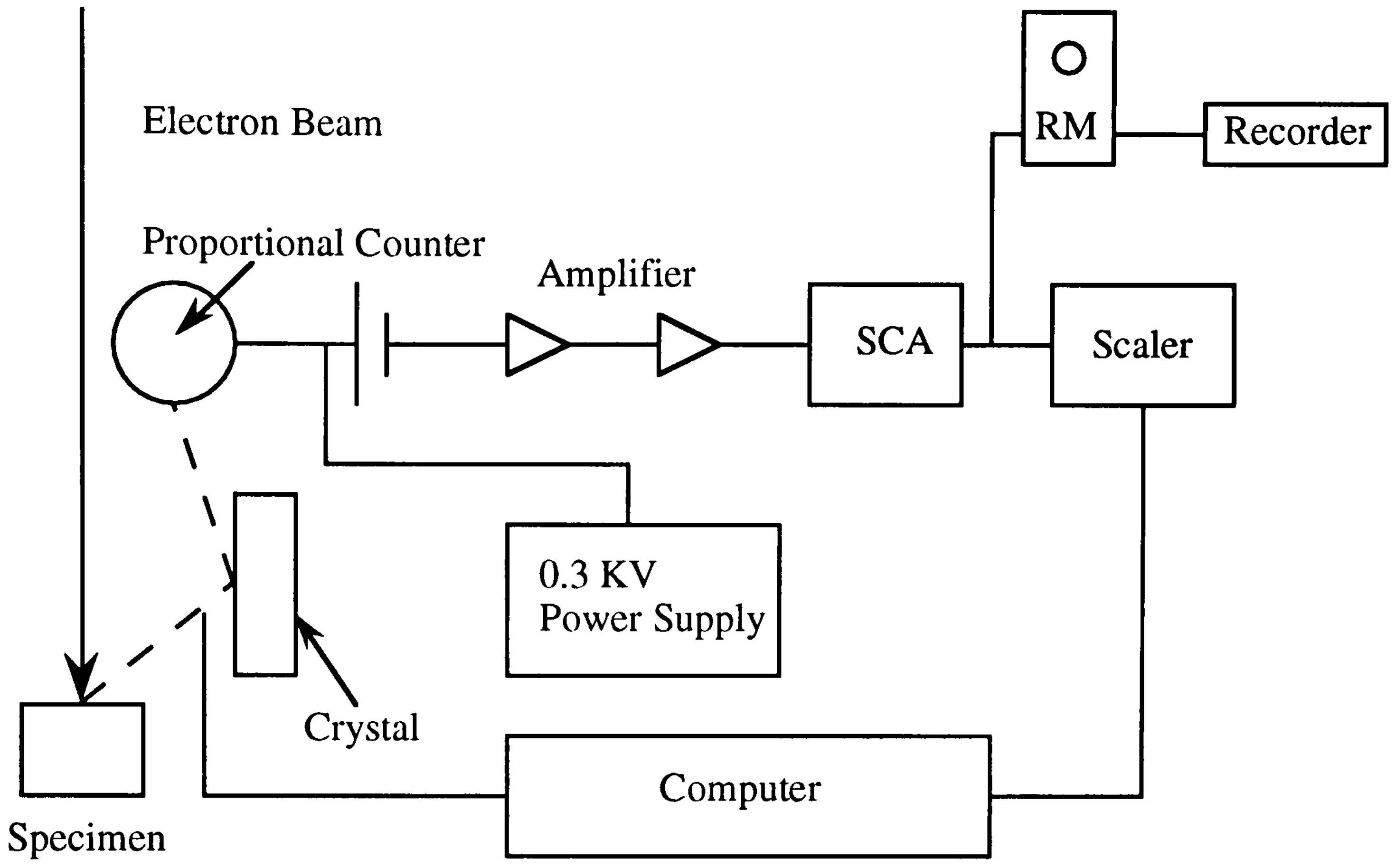


Figure 44: Schematic representation of a wavelength dispersive spectrometer

Electron Probe Microanalysis (EPMA)

EPMA is a quantitative method of analysis in which electron bombardment is again used to generate X-rays. From the wavelength and intensity of the lines in the X-ray spectrum the elements present can be identified and quantified(118). In quantitative analysis the intensities of the lines are compared with those from standard samples of known composition. The measured intensities require instrumental corrections which are dependent upon the characteristics of the measuring system. The composition is calculated from the corrected intensities by applying 'matrix corrections' (ZAF factors) which take account of the various factors governing the relationship between intensity and composition.

The main advantage of EPMA is the use of a finely focused electron beam (0.2 - 1 μm) which forms a probe allowing chemical analysis of very small selected sample areas(119). An example of an EPMA analysis readout can be seen in Table 9.

Table 9: An example of an EPMA analysis readout

ELMT	ZAF*	% ELMT	ATOMIC%
C K=1	0.249	17.800	49.018
O K=1	0.640	0.148	0.306
B K=1	0.209	0.000	0.000
Mo L=1	0.821	0.087	0.030
Ti K=1	0.963	71.366	49.277
V K=1	0.936	0.592	0.385
Cr K=1	0.906	0.159	0.101
Ni K=1	0.915	1.569	0.884
TOTAL		**91.721**	**100.000**

* matrix correction factors

3.2: Transmission Electron Microscopy (TEM)

TEM was used to study the individual nanometre sized particles that make up the sol binders in order to characterise the structure, particle size, packing arrangements and general compositions. TEM analysis was carried out on a Phillips E.M.400T transmission electron microscope.

3.2.1: The Principles of TEM

TEM is a form of electron microscopy in which a beam of high energy electrons is passed through an extremely small specimen thickness. Interaction takes place and electrons are scattered by the sample atoms, the degree of scatter being proportional to the density and thickness of the individual parts of the material through which the electron beam has passed. These scattered electrons are then incident upon a fluorescent screen, thus forming an image of the sample with the features shown as regions of light and dark(120). This is referred to as a 'bright field image'. A schematic diagram of the structure of a TEM microscope is shown in Figure 45.

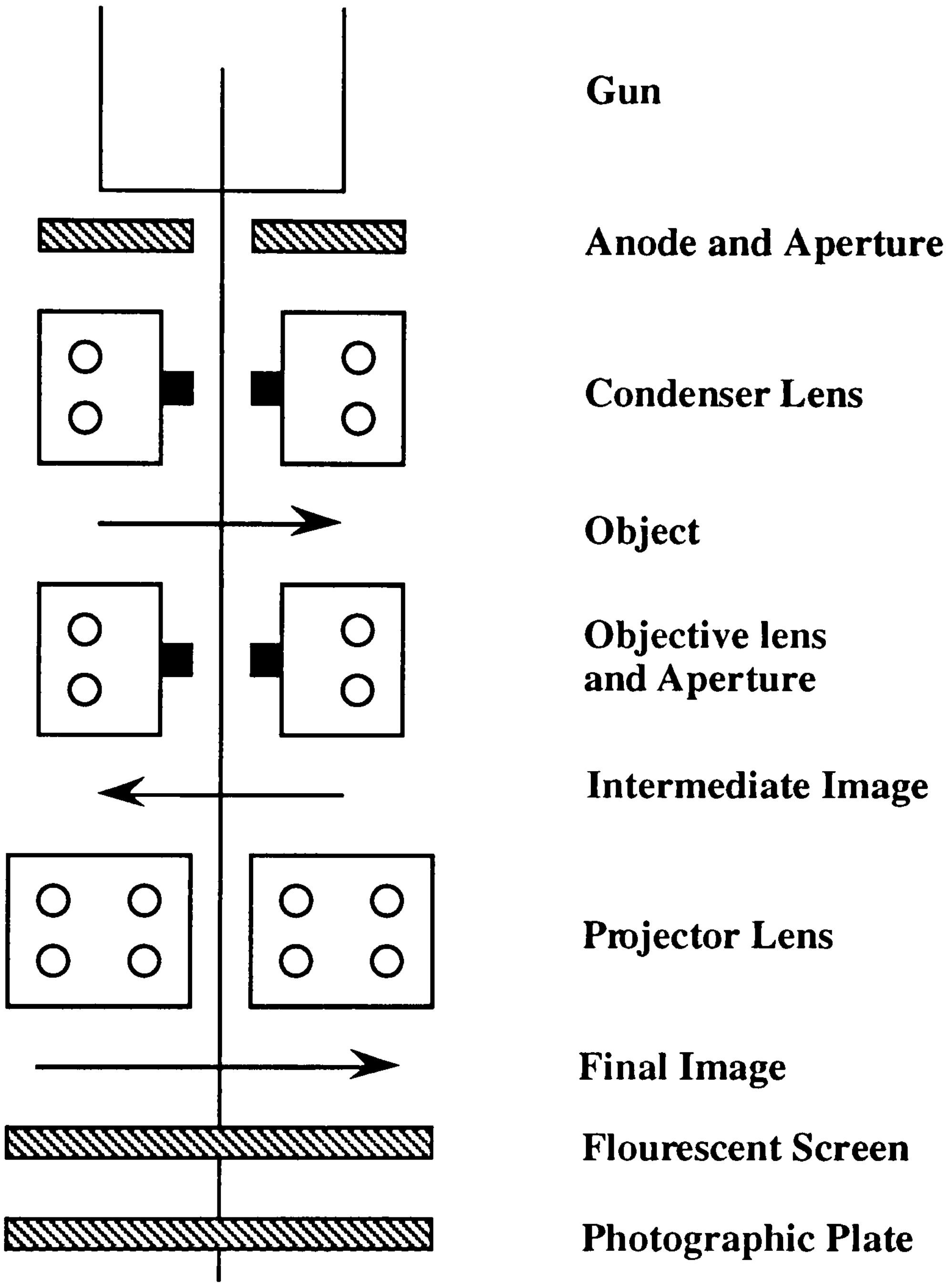


Figure 45: The transmission electron microscope

The electron source is a heated tungsten filament surrounded by a bias shield, a small hole in which allows the beam of electrons to pass through. This beam is focussed by the condenser lens, passes through and interacts with the sample to produce an electron image. The image is magnified by the objective and projector lenses and displayed on the fluorescent screen. The screen can be replaced by a photographic plate, upon which a permanent record of the sample structure can be recorded. Compositional information is also available from the electron beam, which can be utilised by the installation of EDX detectors.

3.2.2: Preparation of TEM samples

Sol samples were prepared by the gellation and drying of small droplets upon 100 mesh folding copper grids. A schematic illustration of the whole process can be seen in Figure 46. Vapour deposited carbon film was floated onto the surface of distilled water. Coating of one half of the copper grid was achieved by positioning the grid under the film and raising slowly from the water. The film then adheres to the copper and when dry provides an electron transparent holder for the sol particles.

A small droplet of the sol was placed onto the film and the water evaporated using a heat lamp. This left a thin layer of sol attached to the film. The grid was folded to hold the carbon and sol layer in place. It was necessary to dilute all the sols to approximately 5% solids content so that the solution was weak enough for the individual sol particles to be seen in the TEM.

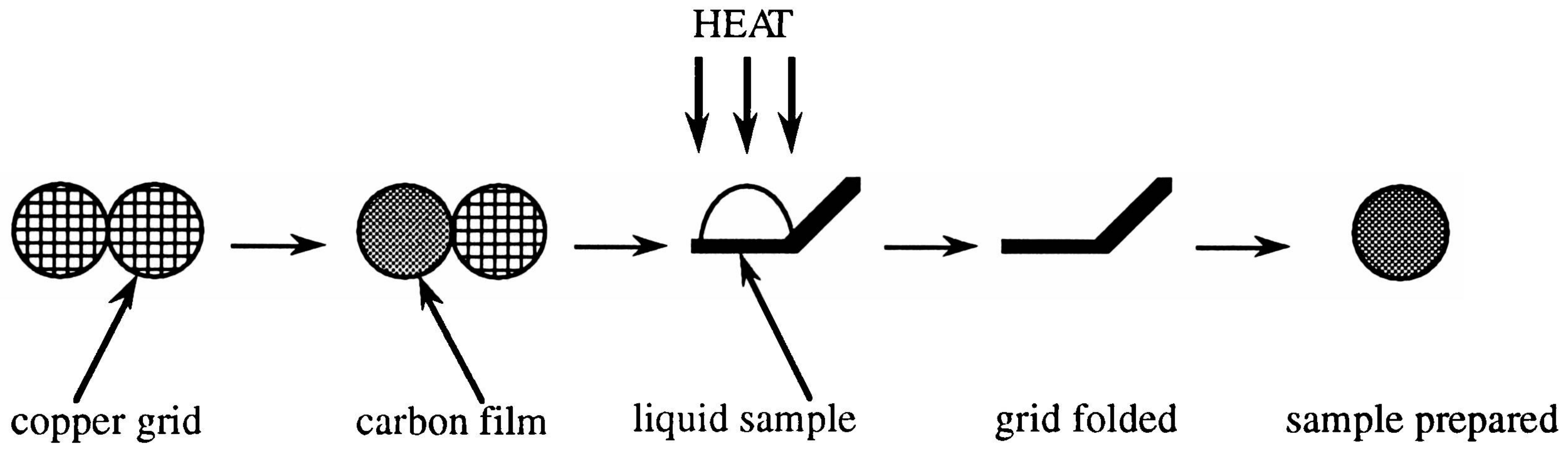


Figure 46: A schematic illustration of the preparation of a sol TEM sample

3.3: Differential Thermal Analysis (DTA)

DTA was used to investigate the thermal response of various binders during heating and cooling. The results allowed us to characterise the temperatures at which phase changes and sintering were occurring within the samples in order to predict the behaviour of the binder during firing and casting of the moulds.

3.3:1: The Principles of DTA

DTA is a technique which involves heating (or cooling) a test sample and an inert reference sample under identical conditions, and recording any temperature difference which develops between them. This allows the transitions and reactions occurring in the substance during this heating or cooling period to be observed(121).

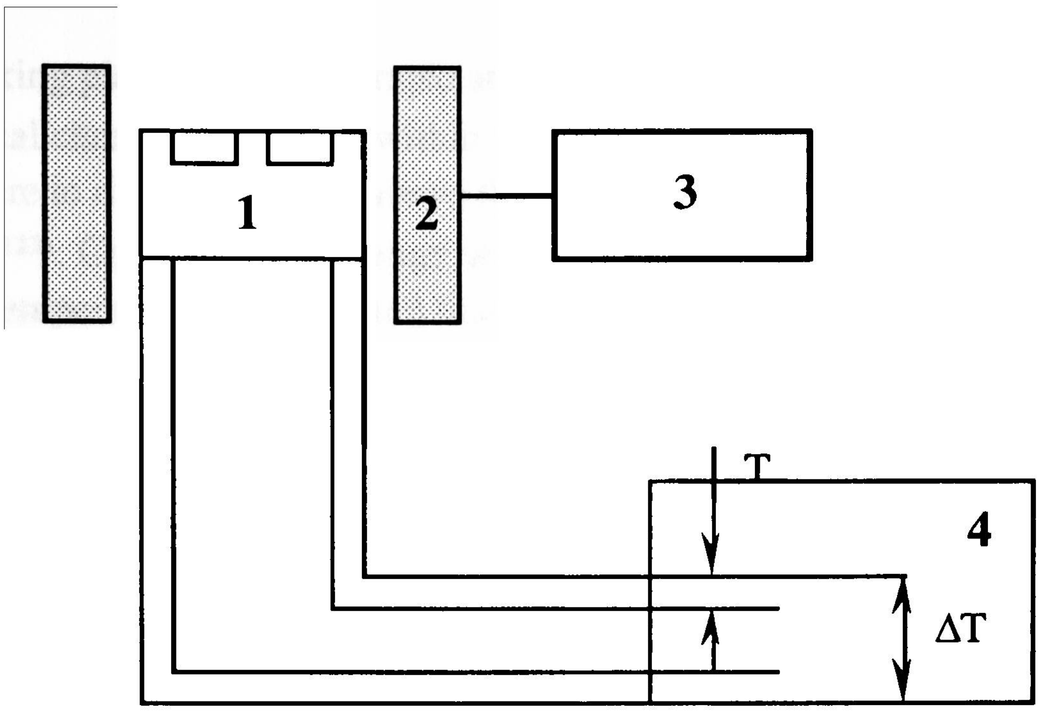


Figure 47: Block diagram of DTA apparatus; 1.Sample measuring system 2. Furnace 3.Temperature programmer 4. Recorder.

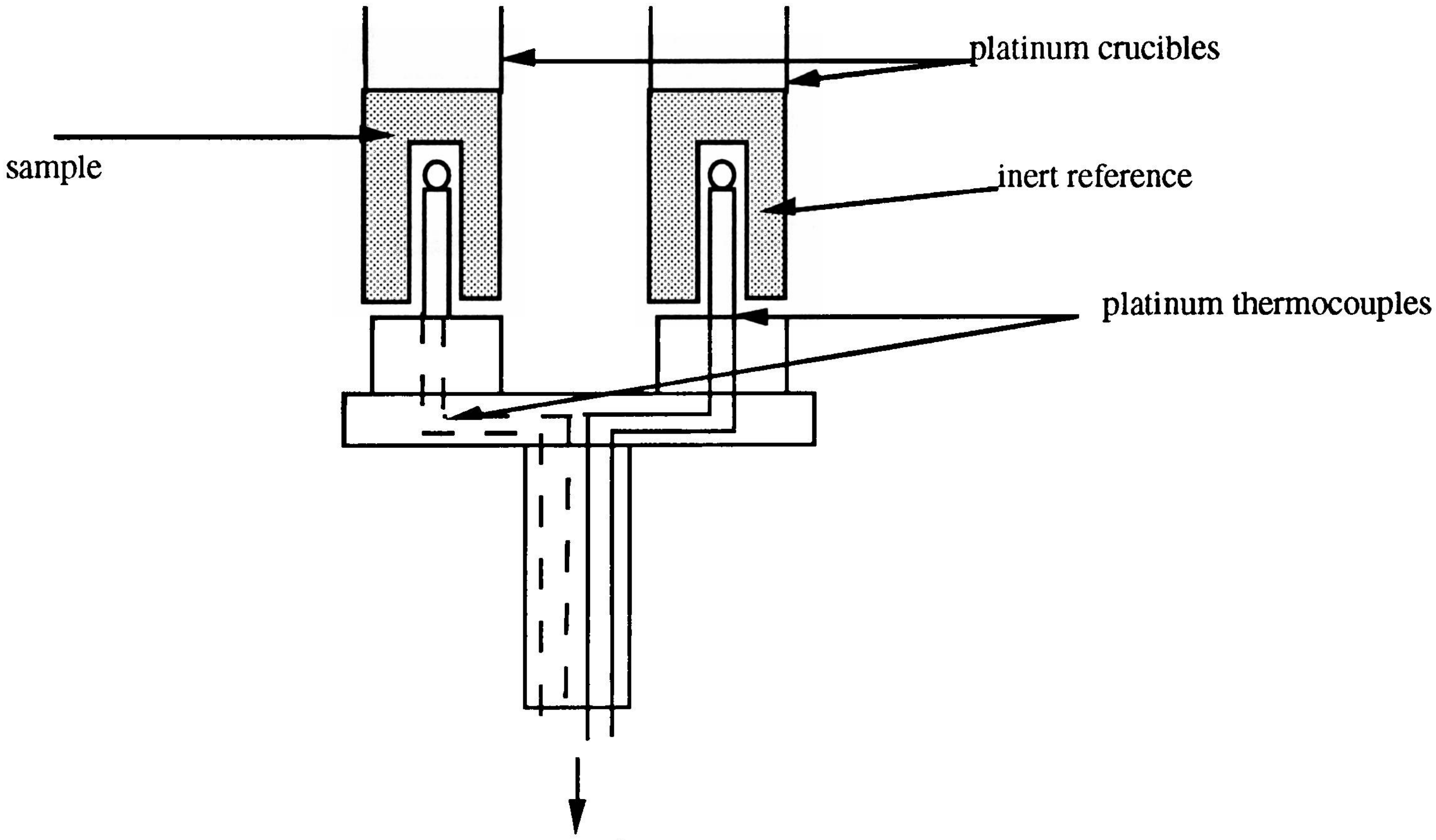


Figure 48: Section through the sample loading apparatus

The substance under investigation, together with a sample of inert material, is placed in a furnace. Figure 47 shows the block diagram of a DTA apparatus and the basic equipment design. The furnace temperature is raised at a constant rate. Differences in temperature between the sample and the reference material (ΔT) are recorded as a function of the furnace temperature. This measurement is accomplished by means of a differential thermocouple (Figure 48) placed in close contact with the sample and the reference material.

If no reaction is taking place, both the sample and the reference will be at the same temperature. Any physical or chemical changes occurring within the sample, which involve the evolution of heat, will cause its temperature to rise temporarily above that of the reference, giving rise to an exothermic peak on the DTA trace(122). Conversely, a change which is accompanied by the absorption of heat will cause the sample temperature to lag behind that of the reference, leading to an endothermic peak on the trace. A typical DTA trace is shown in Figure 49.

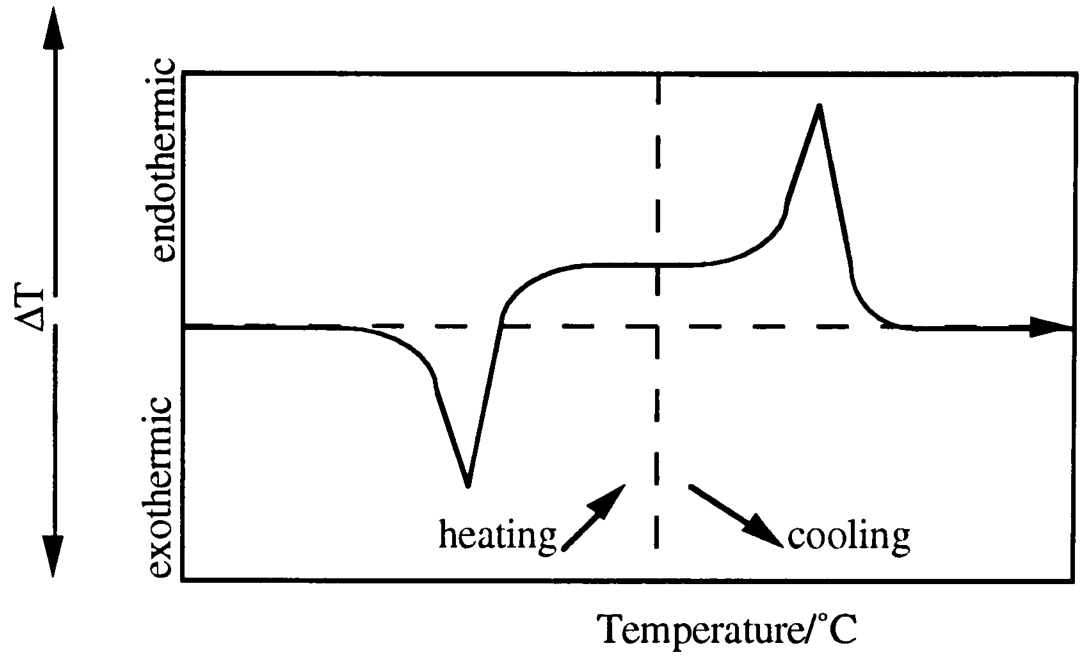


Figure 49: A typical DTA trace

It should also be noted that even when no physical or chemical process is occurring in the sample, a small and steady differential temperature normally develops between the sample and the reference material. This is due primarily to differences in the heat capacity and thermal conductivity of the two materials, but is also influenced by factors such as the sample mass, packing density and the heating/cooling rate(123). Thus, the base line of the DTA plot is rarely horizontal and rarely occurs at ΔT=0.

3.3.2: Apparatus

The apparatus used was a Linseis DTA L62 furnace (covering a temperature range of 20 to 1550^{0}C). Thermocouples (Pt-Pt/10%Rh) were attached to a Keithley 195 System Digital Multimeter, with a IEEE 488 interface which was linked to a BBC Master Series Microcomputer.

3.3.3: Experimental Procedure

The sample and the reference material (calcined Al_2O_3) were made available in powder form (by grinding with a pestle and mortar), weighed and placed into L62/94 platinum crucibles. These crucibles are designed so that the base has a small indentation in it to ensure a snug fit over the thermocouple bead. Sufficient powder should be used so that the thermocouple ends are completely surrounded and thus a nominal powder weight of 50 mg was chosen.

The platinum crucibles were fitted onto the sample carrier and the whole assembly was placed within the furnace. The furnace was programmed with the required temperature profile, running at an initial heating rate of 20°C/minute. The heating rate was chosen to resemble rates used at Deritend Precision Castings. The computer interface allows one thermocouple reading to be taken every 5 seconds and a plot of ΔT versus time was obtained from this data. A temperature versus time plot was also included to allow the temperature profile of the reference to be monitored.

3.4: Linear Dilatometry

The dimensional and mechanical properties of a material provide a sensitive indication of internal structural changes. The determination of the change in length or volume of a sample as a function of temperature constitutes the technique called dilatometry. Dilatometry was used to study phase changes and sintering behaviour of many silica and non-silica binder systems. By understanding the changes within a binder as it is heated, the structural integrity of the mould during casting can be predicted. It should be noted that the dilatometer used in the following experiments was equipped with fused silica sample holder and push-rod. As such, the maximum temperature was restricted to 1150°C to prevent devitrification of the silica at 1200°C. The technique was also used to study the effect of impurity elements upon the thermal response of the basic binder systems.

3.4.1: Principles of Dilatometry

A material which does not undergo a change in structure increases uniformly in length as the temperature is increased. However, structural changes in the material may override this expansion effect, and at particular temperatures discontinuous changes in length, or in the expansion coefficient may occur. The detection and interpretation of such changes in terms of the molecular structure of the sample is the basis of thermal analysis by dilatometry. The most common phase transition that is determined is solid_1 to solid_2(124), although solid to liquid and solid to gas transitions can be determined. The technique can also be used to detect shrinkage and sintering of a sample upon heating to elevated temperatures.

3.4.2: Equipment

The most commonly used instrument is a simple mechanical device constructed from a material of good reproducibility of thermal expansion, such as fused silica(125). A specimen of the material under investigation is placed into the specimen holder in contact with one end of the holder (Figure 50). A rod of similar material to that of the specimen holder is then brought into contact with the other end of the specimen. Conventionally, the push-rod exerts a small compressive load on the specimen to ensure mechanical contact is maintained, especially during contraction of the specimen relative to the specimen holder. The sample holder is then surrounded by a programmable furnace.

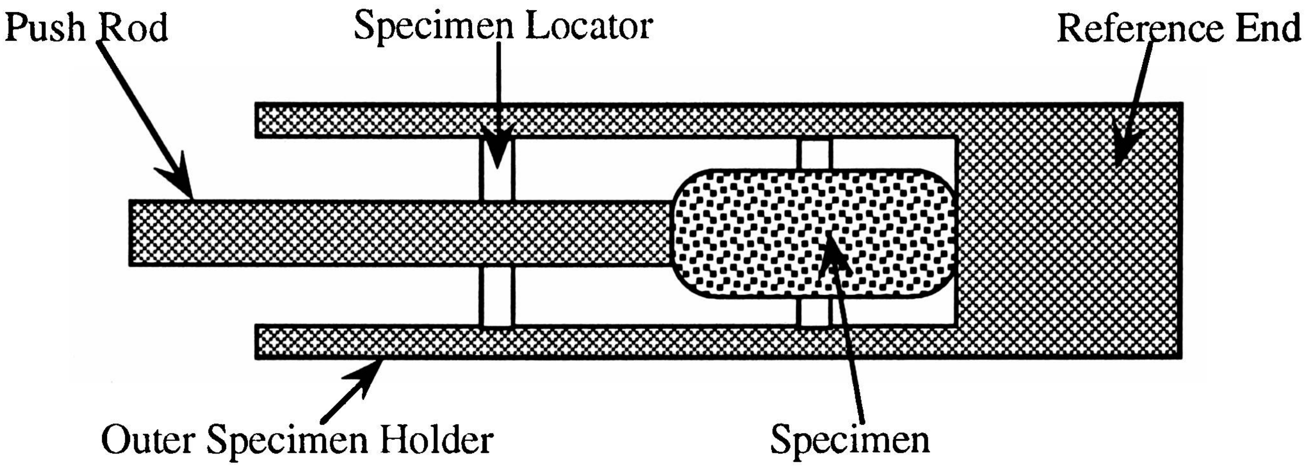


Figure 50 Specimen Holder in a Linear Dilatometer[125]

A Pt / Pt 13%Rh thermocouple is installed into the centre of the push-rod to measure sample temperature. The relative movement of the push-rod and specimen holder, as the specimen is heated or cooled, is recorded by a linear displacement transducer. The output of the transducer (ΔV) and the temperature thermocouple (ΔT) is fed into a data acquisition devise (usually a computer) and the results were obtained graphically. A schematic of the dilatometry apparatus used is shown below (Figure 51).

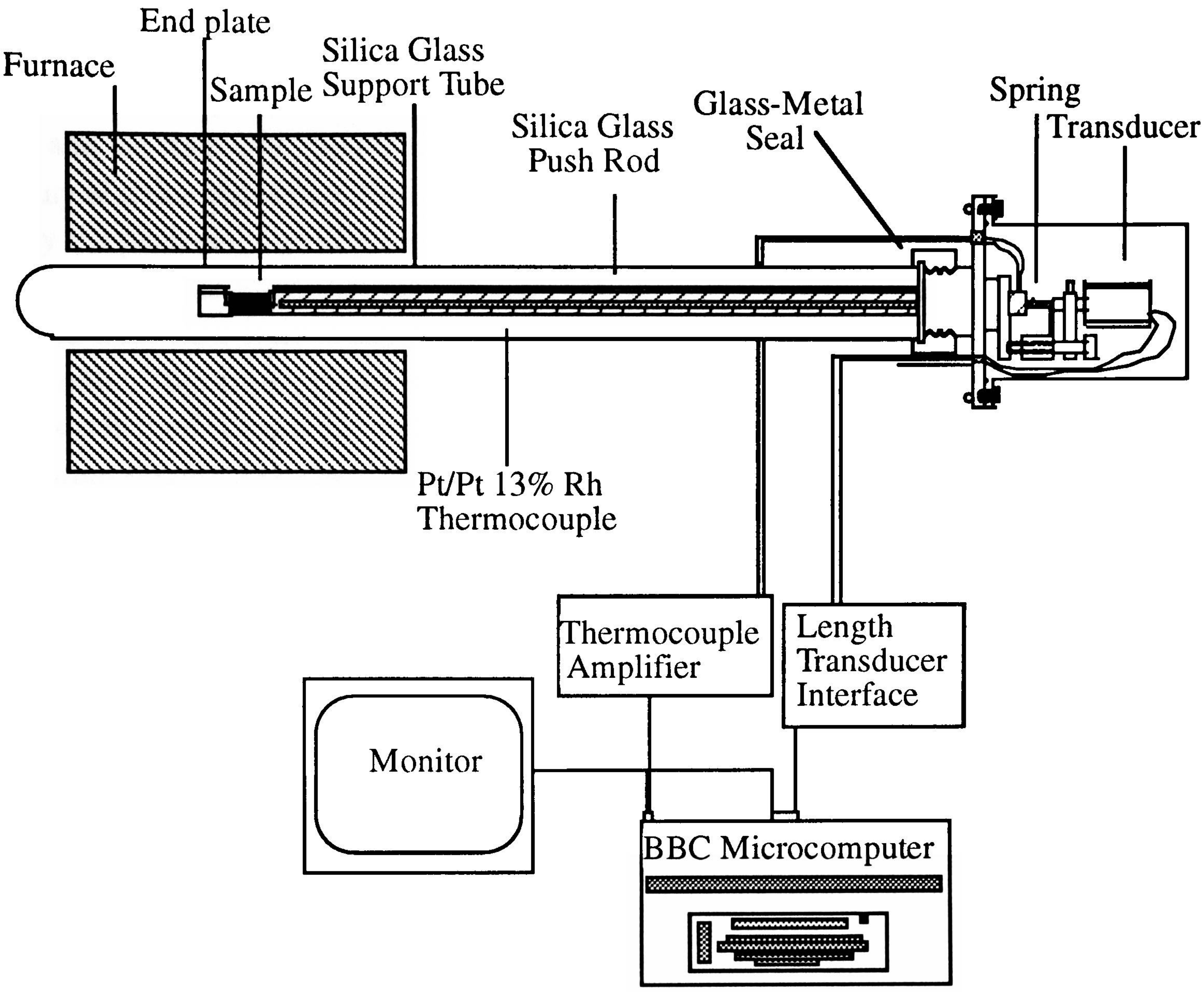


Figure 51: The linear dilatometer

3.4.3: Sample Preparation

Dilatometry requires solid disc or rod shaped samples. Thus, the powdered and dried sols were produced in disc form by pressing in a 7 mm diameter die, using a load pressure of 41.9 kg and the minimum amount of silicone oil as the release agent.

It was found that as pressed discs were too weak to withstand the compressive pressure of the push-rod, so an addition of 5% by weight of Polyethylene Glycol (PEG) was used as a room temperature binder. Approximately 3g of powdered sample was mixed with the appropriate amount of PEG in 20 ml of distilled water. The resulting mixture was redried at 55°C for 2 hours. PEG, being a water soluble organic, burns out harmlessly at 100°C and has no effect upon the thermal response of the samples.

3.4.4: Experimental Procedure

The length of the sample disc was measured and the disc placed into the specimen holder between the push-rod and the rigid end. Platinum foil discs were placed between the sample and the push-rod to prevent any reaction that would lead to devitrification of the fused silica. The pressure applied to the sample by the push-rod was altered to the same level for every sample by moving the rod in or out until an output voltage of 3.5 mV was achieved. All relevant sample dimensions and room temperature were then entered into the computer and the furnace programmed with the appropriate thermal profile. Two basic profiles were used; one to simulate the temperature regime imposed upon a mould as it is fired (Figure 52) and the other to simulate the temperature experienced by the mould during casting (Figure 53). Once the furnace was programmed and brought into position around the sample holder, the data recording programme was initiated and only terminated when the temperature had returned below 200°C.

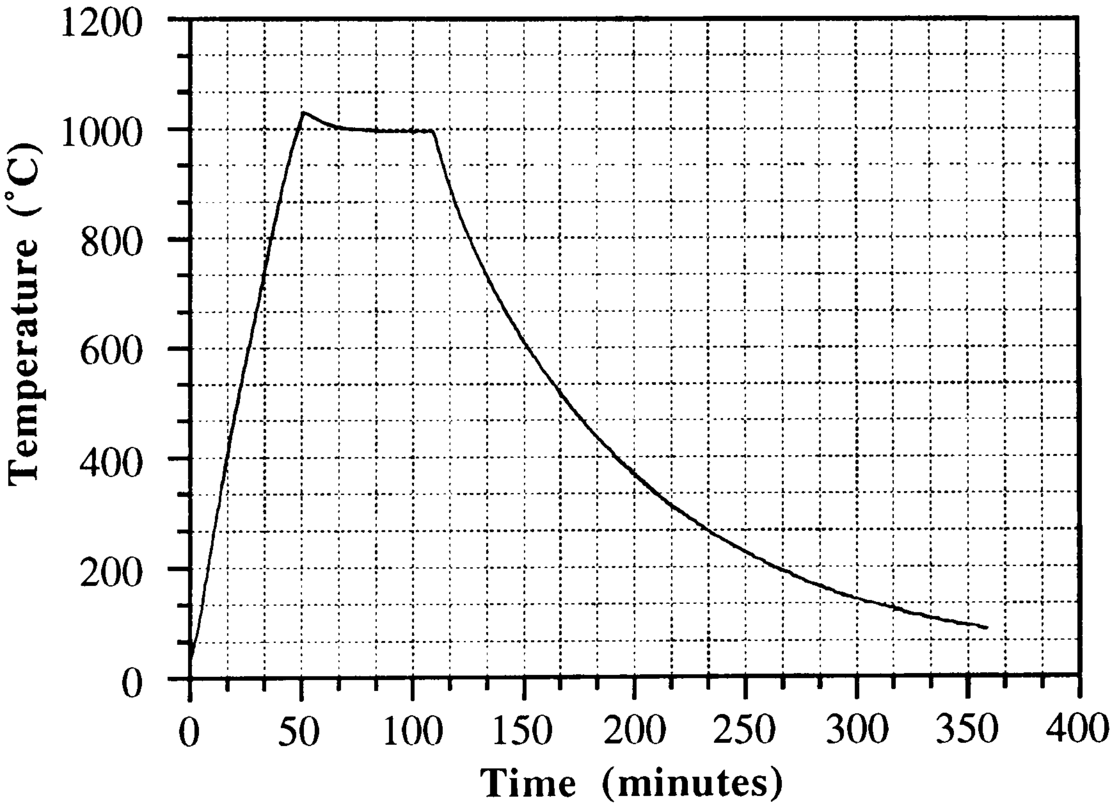


Figure 52: Dilatometry Firing Temperature Profile

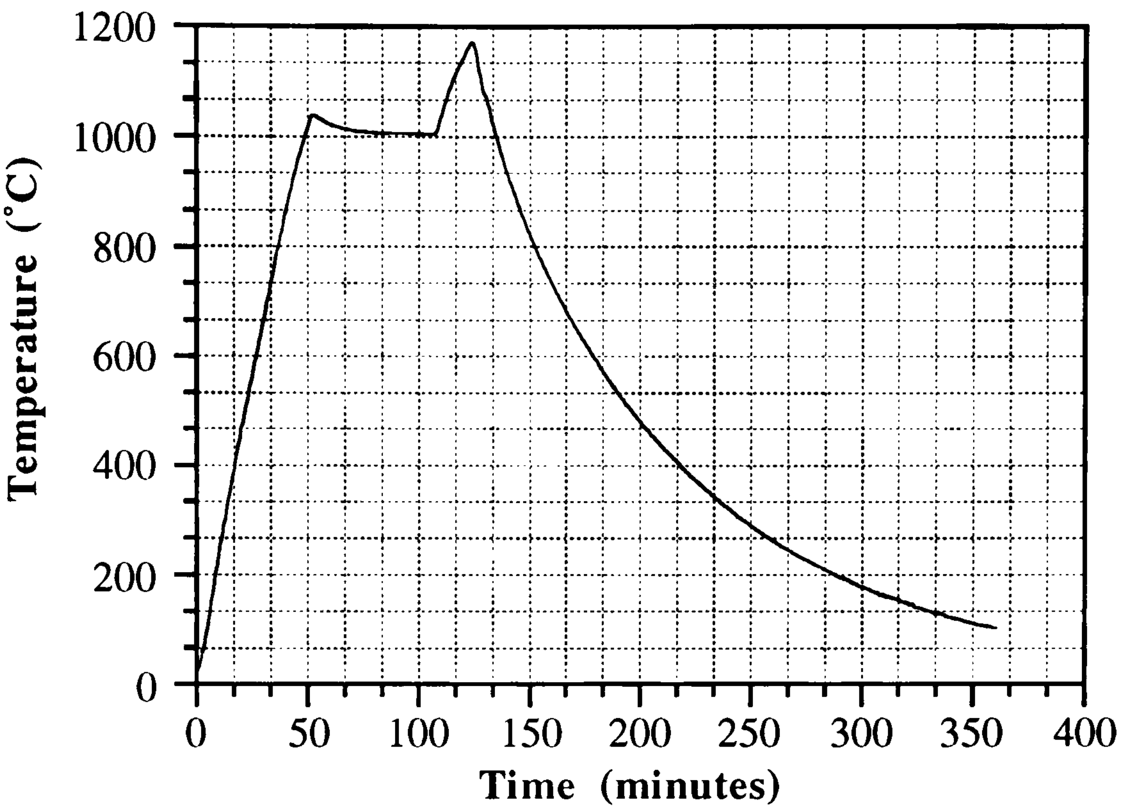


Figure 53: Dilatometry Casting Temperature Profile

3.5: Image Analysis

Image analysis was used to characterise four samples of zircon sand obtained from various sources around the world. This examines the possibility that refractories from different sources contain different amounts of separated phases within the particles. The results were processed to allow each sample to be given a value for % phase separation (by area). Statistical analysis was then used for a comparison of the data obtained.

3.5.1: Principles of Image Analysis

Image analysis is a versatile technique that has found applications in a very wide range of scientific disciplines. It is a metallographic technique used for quantification and classification of images contained within a two-dimensional section. A wide variety of measurements such as grain size, percentage porosity, area fractions and particle sizing can be carried out and the technique can even be used to quantify volumes in three dimensions using geometric probabilities[127].

The principal stages in image analysis can be considered as; image capture, segmentation, object detection, measuring and analysis (these are the operations in the boxes down the centre line of Figure 54). The image to be analysed is input into a television camera either directly from a microscope or from a macroviewer and light table. This image is digitalised, coupled with an analog-digital converter and displayed on a monitor. Automatic selection of features is possible by such criteria as ratio, length, area, width or combinations of these. By the use of a digitiser tablet and light pen the required area can be selected for study , allowing the analysis of particular areas or images whose contrast is not good enough for automatic processing. The relevant parameters are measured and processed in an image processing unit. Sizing, statistical analysis, plotting of distributions etc. is performed by a computer equipped with data analysis software.

3.5.2: Apparatus

The system used was a VIDS 2 video interactive display system which comprises an Apple 11e 64k computer, TV scanner, video interface card, CalComp 2000 series digitizer, light table, software measurements pack (VISCALC) and a high speed printer. The system combines the video output from the TV camera with the graphics display of the computer so that measurements are made directly from the TV image. VIDS 2 is a software based measuring system utilising a graphics tablet which allows area fractions to be calculated within operator defined boundaries.

3.5.3: Experimental Procedure

Four samples of zircon sand, obtained from Cookson Ceramics Ltd., were examined to determine the amount of phase separation contained within each. Conventional chemical analysis detects the presence of small amounts of the individual impurity elements (such as Fe and Al) but this tells us nothing about the type or physical distribution of the separated phases. Since the impurity elements appear to leach into the binder from the surface of refractory particles, it is important that the separation is categorised relative to area and amount.

SEM samples were made up according to the method described in Section 3.1.2. Using a constant magnification of 200x, an area of the section was chosen at random and a photograph taken. Nineteen other areas directly adjacent were also photographed. These areas were deemed to be representative of the overall sample so that statistical analysis could be used to determine an average value for the amount of phase separation in each sample.

Each micrograph was traced onto a transparent sheet and the areas of phase separation highlighted accordingly (Figure 55). The tracings were placed onto a light table and an image obtained using the TV camera. A grid was used to calibrate the system and to allow every individual photograph to be split into four sections (the calibration used was 0.2631 cm per picture point). The regions of zirconium silicate and phase separation were highlighted using the light pen and the information fed into a software package which calculated area fractions using this calibration parameter. The four sections were analysed separately and the values obtained were subsequently combined to give total areas of zirconium silicate and phase separation. The amount of separation relative to the overall area of zirconium silicate was expressed as a percentage giving twenty individual results. Using statistical analysis (see Appendix 1), the average percentage phase separation (by area) was calculated.

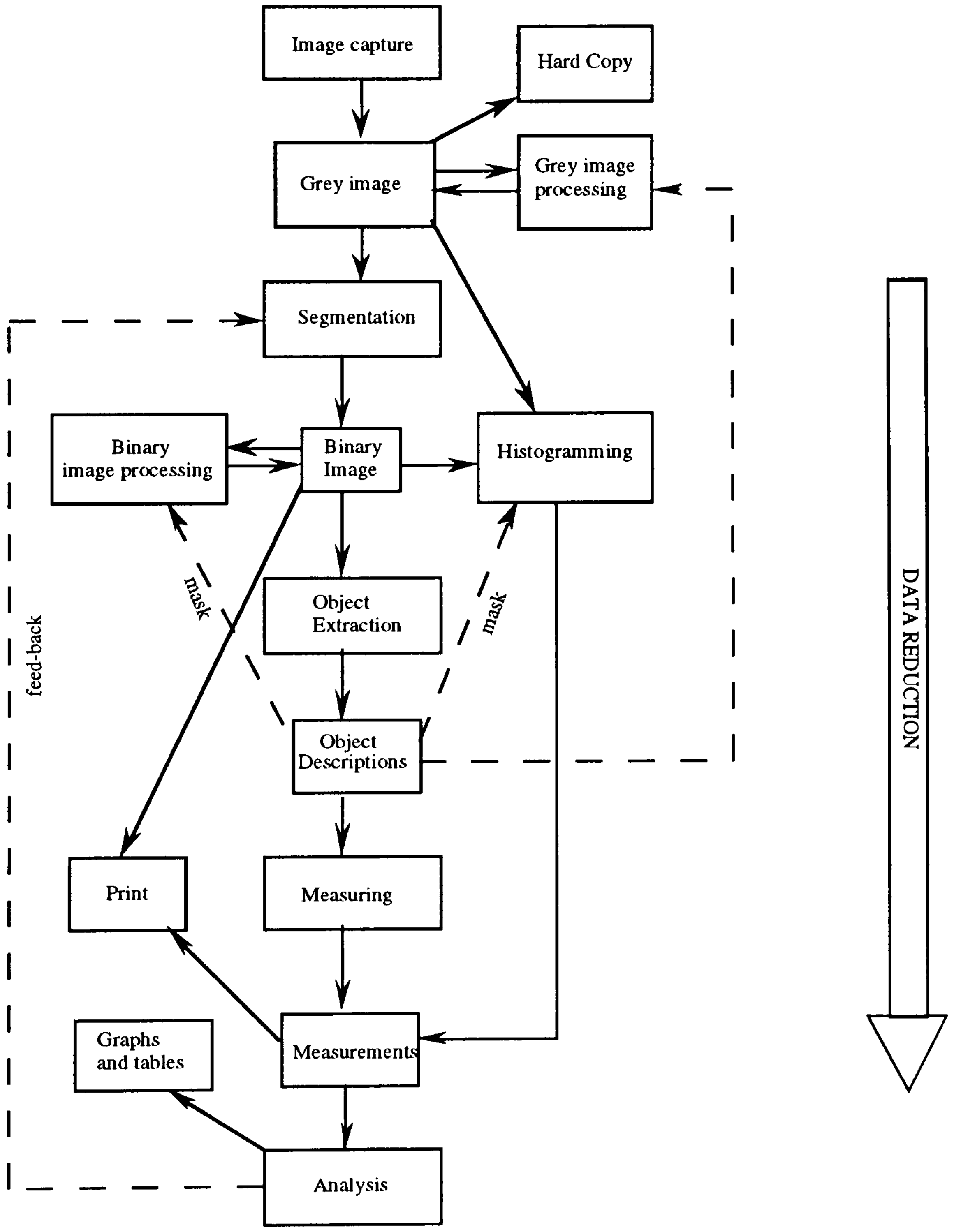


Figure 54: The principal stages of image analysis(127)

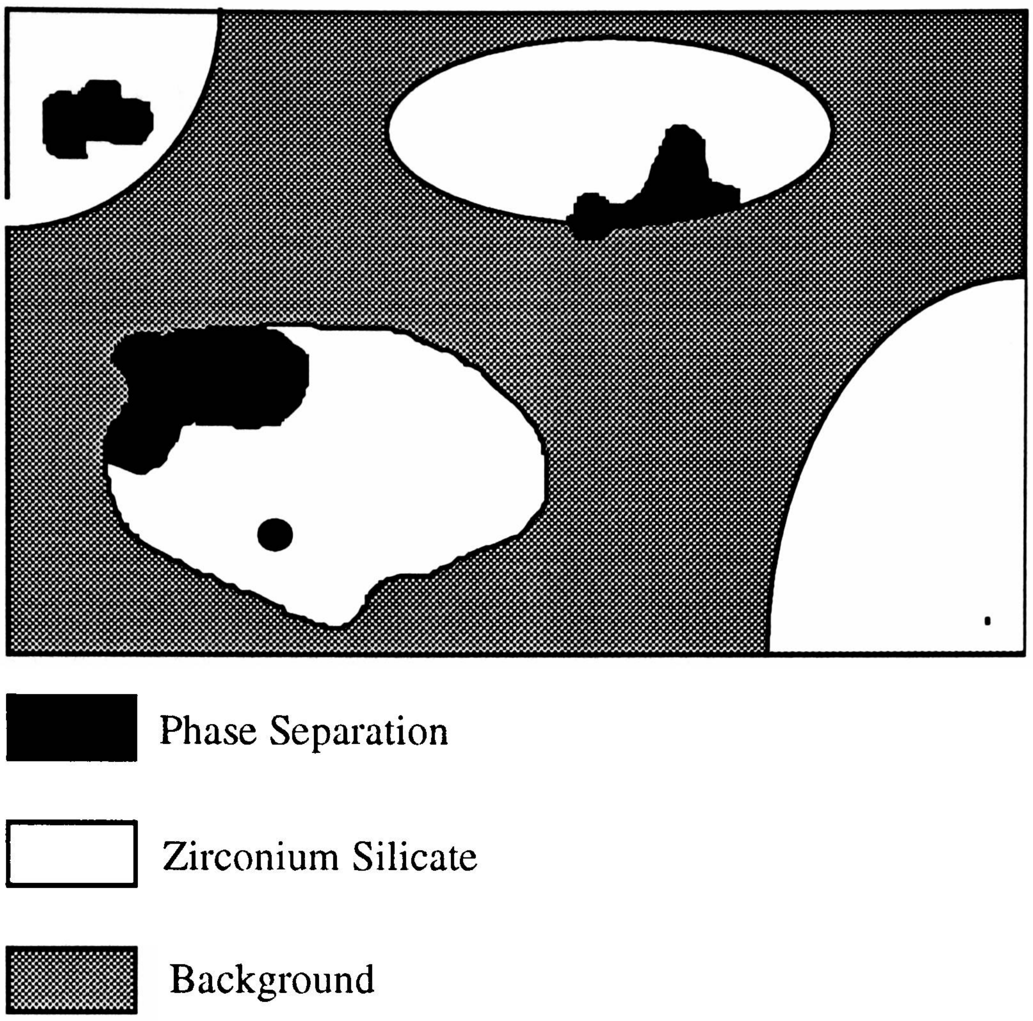


Figure 55: Traced image used for area analysis

3.6: Atomic Absorption Spectrometry (AAS)

AAS is a technique used for the analysis of water based elements. It was used to identify impurity elements within silica binders after incorporation into ceramic slurries. All experiments using AAS were carried out at Midland Research Ltd.

3.6.1: Principles of AAS

A A S is an analytical method for the determination of elements, based upon the absorption of radiation by free atoms(129). The principle of AAS is the way in which atoms respond to the input of energy. When energy e.g. thermal,electromagnetic, chemical or even electrical, is input into a material, collision processes transfer this energy to the electrons of the atoms. Electrons in the ground state i.e. when the electrons are at their lowest energy level, are 'excited' and moved to higher energy levels. Transitions between the energy levels correspond to the absorption of quantised amounts of energy, in the form of electromagnetic radiation, which are determined by Bohr's condition (Equation 17);

$$\Delta E = E_1 - E_2 = hf \qquad \textbf{Equation 17}$$

ΔE = energy absorbed
E_1 = energy of initial electron state
E_2 = energy of final electron state
h = Planck's constant
f = frequency of electromagnetic radiation

When the energy input has ceased the atoms are left in a metastable state with electrons in higher energy levels than they would normally occupy. The electrons return to their normal energy levels and in doing so emit light energy equal to ΔE (Figure 56). This light energy is manifest in the form of a spectrum, which consists of radiation of a number of discrete wavelengths. Individual elements absorb and emit different amounts of energy at different wavelengths. Therefore each element has a characteristic light spectrum associated with it.

In AAS the reverse process is utilised and light of a frequency associated with a particular element is passed through a vapour containing the sample atoms. If the sample contains atoms of this element then the light is absorbed in the process of excitation and an absorption spectrum will be produced. The absorption spectra of most elements are extremely simple and, as there is very little possibility of coincidence of lines, atomic absorption is a highly accurate means of analysis.

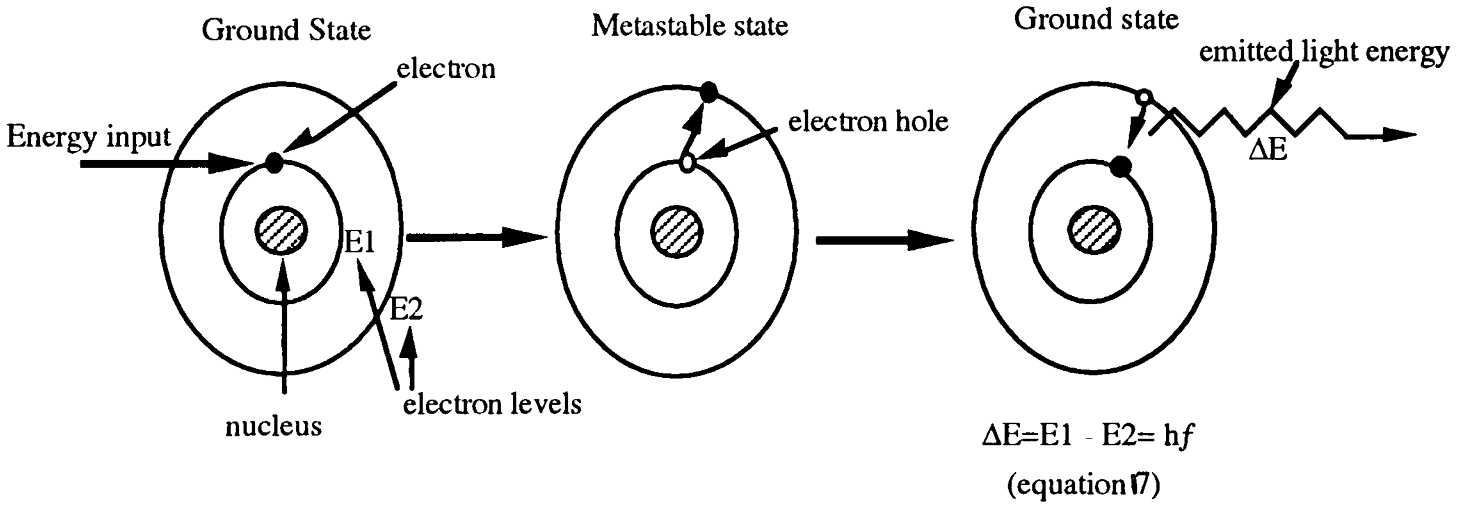


Figure 56: Schematic illustration of energy absorbance and emittance by atoms[128]

3.6.2: Preparation of Samples

Slurry samples were allowed to settle over a period of time into two distinct layers; the top layer consists of the liquid binder and the lower layer being the remaining ceramic filler material. The binder was decanted and allowed to gell and dry in a drying cabinet for 8 hours at 55°C. Since AAS requires a liquid medium, the samples were redissolved in hydrochloric acid and diluted to approximately 5% (molarity) before examination.

3.6:3: Experimental Procedure

The basic atomic absorption spectrometer is shown in Figure 57. Light from the source lamp, generating a sharp line spectrum characteristic of the desired element, passes through the flame into which the sample solution is sprayed as a fine mist. The region of the spectrum in the immediate neighbourhood of the frequency line to be measured is selected by the monochromator. The isolated frequency line falls onto the detector (a photomultiplier), the output of which is amplified to drive a readout device. The intensity of the line is measured with and without the sample passing into the flame. The difference between these readings is a measure of the absorption and therefore of the amount of the element present in the sample solution.

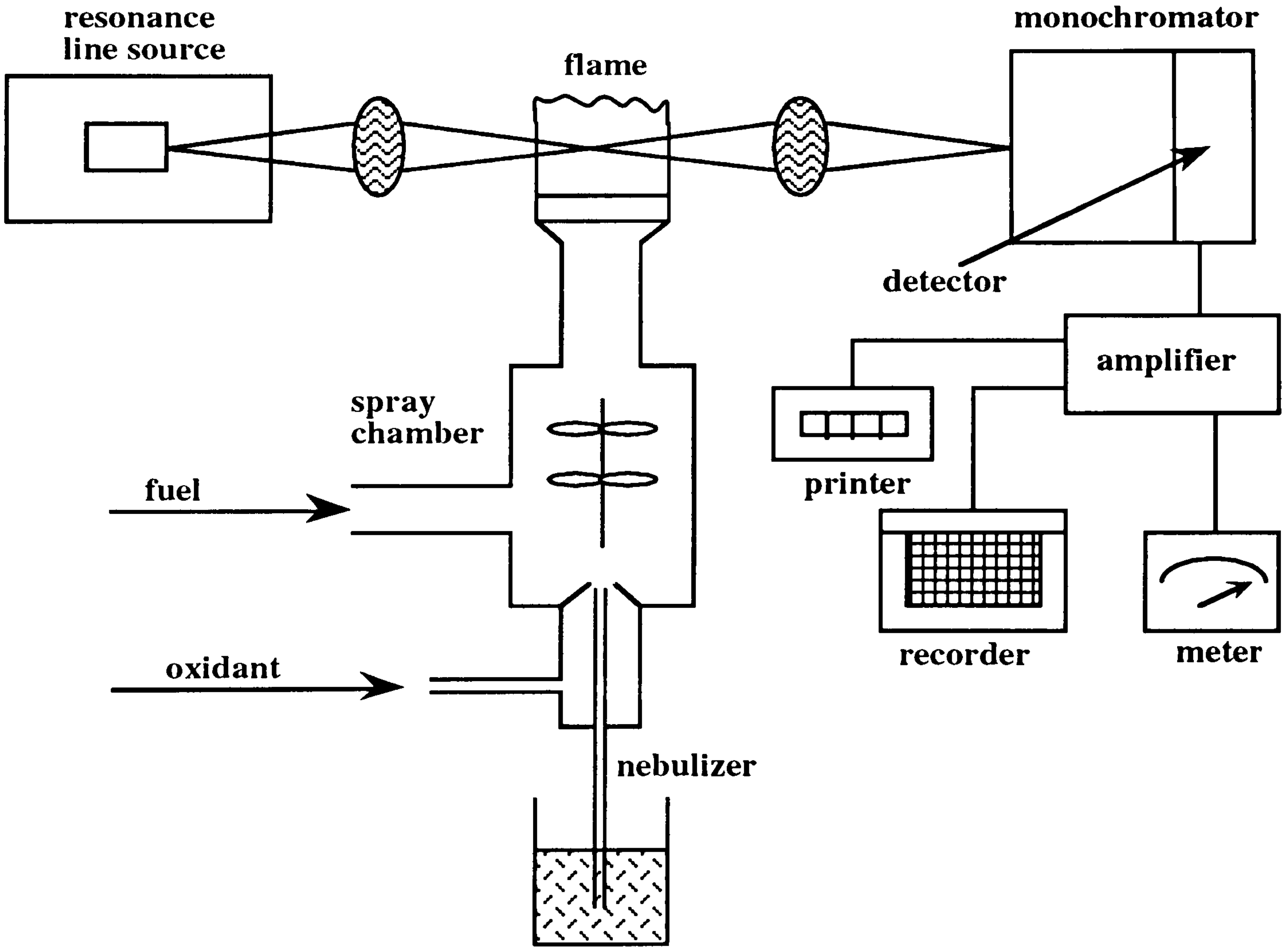


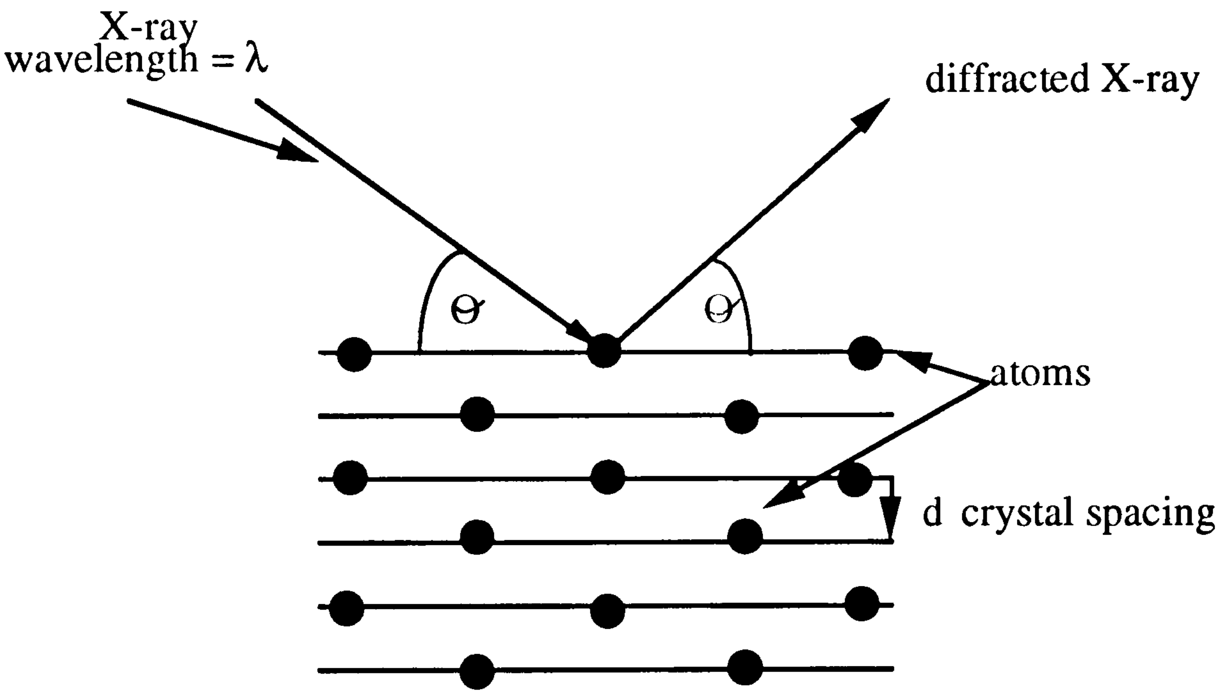
Figure 57: Atomic Absorption Spectrometer[128]

3.7: Powder X-Ray Diffractometry

Powder X-ray diffractometry was used to determine the phase structures of the various ceramic fillers, powders and binders that are used to produce an investment casting mould. X-ray diffractometry was carried out on a Phillips PW 1050 powder diffractometer with a counting period of 2 seconds and step size of 0.05° (2Θ). The radiation used was CuK_α (wavelength = 1.5405 nm). Though x-ray analysis of this nature can also be carried out with a Debye-Scherrer powder camera, diffractometry was chosen because of equipment availability and the rapidity of phase identification using computer analysis packages.

3.7.1: The Principles of X-ray Diffractometry

An X-ray diffractometer is an instrument for studying the crystallinity of solids, by using the diffraction patterns produced by the x-rays as they interact with the materials(129).



Θ= angle of diffraction

Figure 58: X-ray diffraction from a set of crystal planes

Most solid materials exhibit a degree of crystallinity and hence show some symmetry and regularity. In a section of crystal the atoms are arranged on a set of parallel planes (Figure 58). According to Bragg's Law (Equation 18) if the value of λ and θ are fixed then the atomic spacing (d) for the crystal can be calculated.

$$\lambda = 2d \sin \theta \qquad \textbf{Equation 18}$$

λ = wavelength of radiation
d = crystal interplanar spacing
θ = angle of diffraction
= angle of incidence

The essential features of a spectrometer are shown in Figure 59. The sample is rotated about the axis through a series of angles (0-180°). The x-rays produced by the source are incident upon the crystal at an angle O. The X-ray is diffracted by a set of crystal planes within the sample and the intensity of the diffracted beam measured directly by an electronic counter.

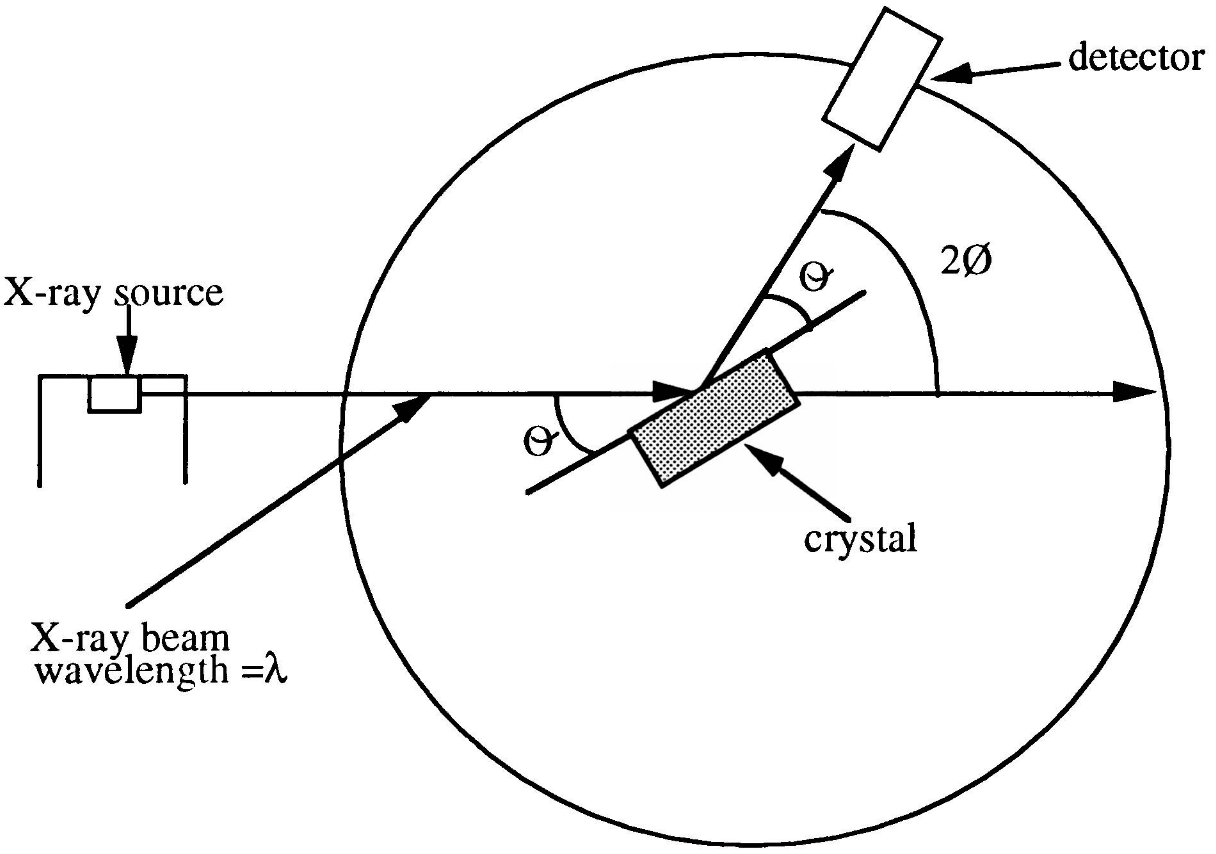
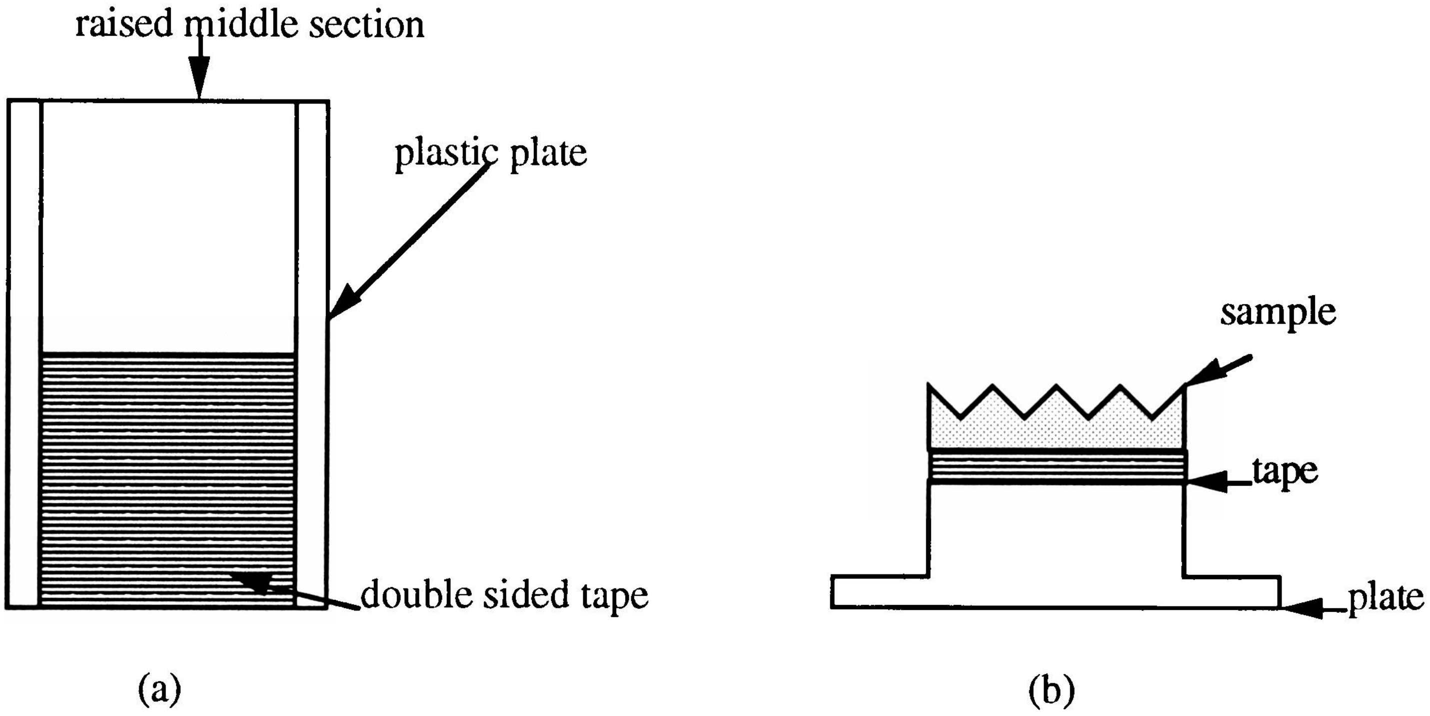


Figure 59: The X-ray spectrometer

3.7.2: Experimental Procedure

The sample to be examined must first be ground to a powder with a pestle and mortar. The powder must be extremely fine (10μm or less) to give an accurate representation of the overall structure of the sample. A sample holder was prepared by placing a strip of double sided tape on one half of the plate (Figure 60(a)). The sample powder was sprinkled onto this tape, smoothed down and compacted gently with a glass slide (Figure 60(b)).

The sample holder was then inserted into the diffractometer (sometimes called a goniometer) and the machine sealed to prevent the leakage of X-rays. The diffractometer was set to scan between a range of incident angles and the results were recorded on a Master Series Microcomputer in the form of a trace graph showing detector count rate versus the angle of rotation.



(a) (b)

Figure 60: Powder X-ray diffraction sample

3.7.3: Diffraction Patterns

A plot of diffracted intensity versus the angle of rotation (2θ) is obtained (Figure 61). When no diffraction occurs the plot is continuously horizontal, with an absence of the characteristic peaks which occur when the x-rays are diffracted by a set of crystal planes. The sample to be measured is ground to a fine powder which can be considered to be thousands of small crystals (crystallites). The random orientation of these crystallites in the specimen ensures that every possible reflection plane is represented and occurs parallel to the specimen surface in at least some of the crystallites.

The patterns produced is characteristic for that material (a 'fingerprint') and can thus be used as a means of identification. When analysing a mixture of materials or phases, diffractometry shows the typical peaks of all the components. The peaks can be categorised and used to identify the separate components. For an amorphous material there are no regular crystal planes and therefore diffraction does not occur. Such an x-ray pattern has a characteristic amorphous halo, thus allowing the identification of an amorphous material or phase (see Section 2.3.2).

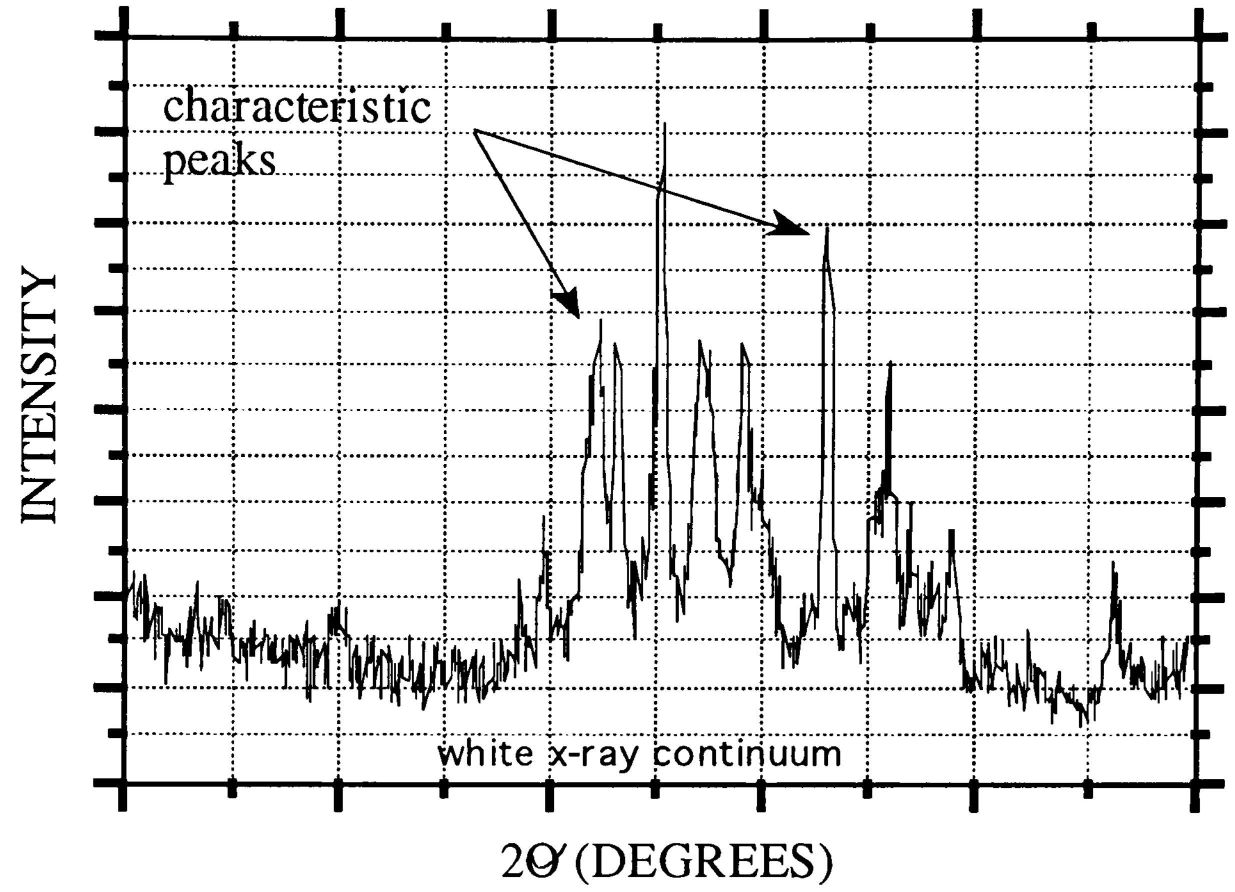


Figure 61: Powder X-ray Diffraction Trace

3.8: Binder Processing

In order to study the phase structure (X-ray diffraction) and thermal responses (linear dilatometry and DTA) of the binders it was necessary to produce them in powder form. The binders were gelled, dried and subjected to thermal treatments or mixed with other ceramic constituents to produce particular compositions.

3.8:1: The Gellation and Drying of Binder Samples

There are two types of silica binder commonly used in the investment casting industry, water based colloidal sols and alcohol based ethyl silicates. Both binders can be gelled by evaporation of the solvent, but ethyl silicate can be gelled much more rapidly by a chemical setting technique. Non-silica water based binders, used for experimentation, were treated as silica sols and gelled by evaporation.

A small sample of the binder was placed into a plastic dish. The water based binders were placed into a drying cabinet at 55°C and allowed to dry for at least 10 hours. Evaporation of the water caused the sol particles to bind together in a network to form the gel structure. The alcohol based binders were gelled by the addition of a few drops of ammonia solution (NH_4OH) to the liquid. Ammonia is a base catalyst which promotes accelerated hydrolysis and condensation reactions, leading to the polymerisation of the ethyl silicate to form the gel network. The gel was then placed into the drying cabinet for a few hours to remove the remaining alcohol from the structure.

3.8.2: Preparation of Binder compositions.

Binder compositions were prepared in both the liquid and the solid state. Examples of the calculations required to determine the relative additions of other elements to the binders can be found in Appendix 10. To study the effect of sodium content on the crystallisation behaviour of silica sols, sodium was added to the binder in the liquid state in the form of NaOH. Binder compositions found within fired moulds were replicated in the solid state in order to study the effect of impurities upon phase and thermal response. Atomic percentages were used and the components were weighed accordingly. Once weighed the dry powders were shaken in a pot to ensure even mixing of the components. Firing of both compositions was carried out in a platinum crucible, thus ensuring that no reaction took place between the components and the heating vessel.

3.9: Preparation and Maintenance of Experimental Ceramic Slurries

It was necessary, as part of the investigations undertaken, to manufacture mould and slurry binder samples using both silica and non-silica based binders. Since the bulk slurries used at Deritend Precision Castings are based on silica binders, it was necessary to produce and maintain separate experimental slurries on a small scale in the laboratory. In order for the samples to be representative, the slurries were formulated and maintained in the same manner as the slurries presently in use at Deritend Precision Castings.

3.9.1: Experimental Slurries in The Laboratory

Ceramic slurries require continuous stirring or agitation to prevent the larger refractory constituents from settling out over a period of time. The apparatus used can be seen in Figure 62. The stirrer was a Citenco model KQTS9, with a speed range of 0 to 600 rev/min and a constant torque of 172 mN.m. These stirrers are only available with stainless steel paddles so, as there was a possibility that the acidic slurries might attack the steel and impair the results, a perspex paddle was designed and made (Figure 63).

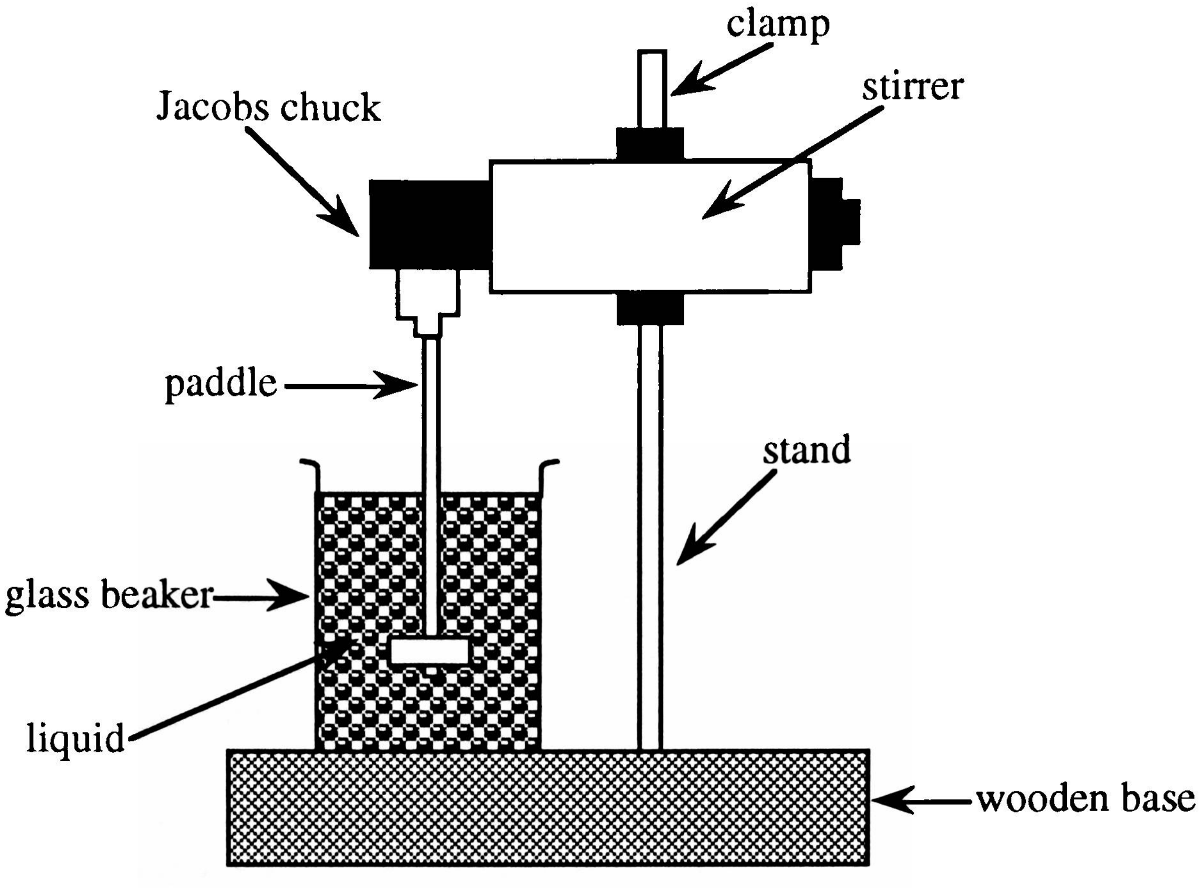


Figure 62: Apparatus used in slurry preparation

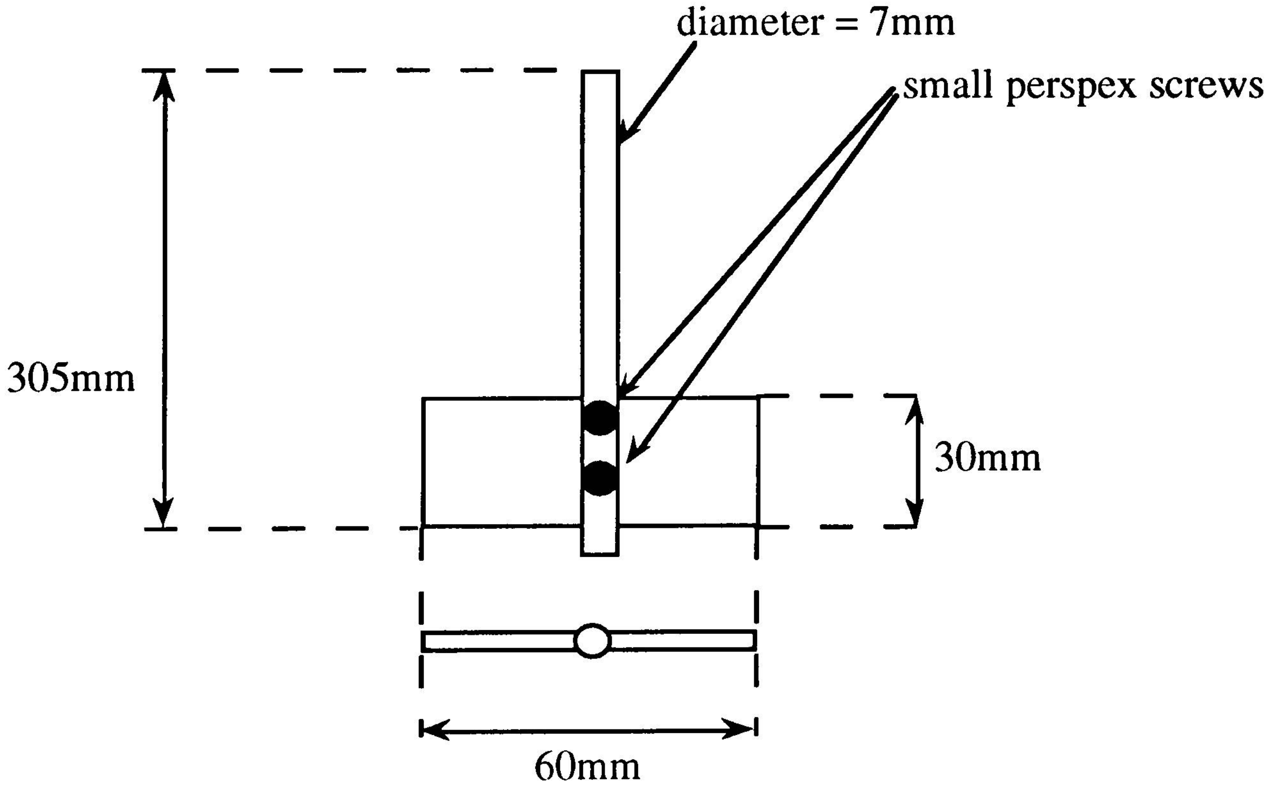


Figure 63: Perspex paddle design

Constituents were prepared according to the formulation schedule contained in Table 10 and mixed thoroughly. Note that when a water based slurry is used as a primary coat, only one type of filler is used, but when the slurry is required for use as a secondary coat the fillers are mixed as for the alcohol based slurry. The stirring rate was set at a sufficient level to prevent settlement of the filler, and the whole system was left to stabilise over a period of time. When first mixed, the slurry contained a large amount of air, which would have detrimental effects on the mechanical properties of the finished mould. Most of the slurries required at least 24 hours for complete removal of this air, though thorough initial mixing of the slurry components reduced the amount of air in the slurry.

Table 10: Water based primary and alcohol based secondary coat slurry formulations currently in use at Deritend Precision Castings (see Appendices for a full description of all the individual binders, fillers and constituents).

COMPONENT	WATER BASED SLURRY	ALCOHOL BASED SLURRY
Water Based Sol	10 litres	nil
Alcohol Based Sol	nil	10 litres
Zircosil 200	nil	17.5 Kg
Zircosil 200M	46 Kg	17.5 Kg
Octanol	30 ml	nil
Synperonic NX	60 ml	nil
Isopropyl Alcohol	nil	added to decrease viscosity accordingly
Distilled Water	added to decrease viscosity accordingly	nil

The control limits presently placed upon slurries are summarised in Table 11. Again it should be noted that when a water based slurry is to be used as a secondary coat then the viscosity should be reduced to that of the alcohol based slurry. Within the laboratory it was not possible to maintain strictly all of these controls due to limited facilities. As such, only pH and viscosity were monitored. The pH was controlled using hydrochloric acid and the viscosity by the addition of appropriate amounts of either water or isopropyl alcohol.

<u>Table 11:</u> Water based primary and alcohol based secondary coat slurry control limits currently in use at Deritend Precision Castings.

CONTROL	WATER BASED SLURRY	ALCOHOL BASED SLURRY
Temperature (°C)	22 +/- 2	20 +/- 2
pH	dependant upon the system	dependant upon the system
Humidity (relative percentage)	30% max	55% max
Viscosity	75 - 85 seconds *	10 - 12 seconds +

* Zahn viscosity cup ASTM D4212 4.39 mm diameter orifice

\+ Viscosity cup BS 3900 Part 6 : 1971 3.97 mm diameter orifice

3.9.2: Experimental slurries at Deritend Precision Castings

Slurries prepared and used at Deritend were contained and stirred in a scaled down replica of the large tanks used in normal production (Figure 64). Stirring of the slurry is achieved by rotation of the outer drum with the central paddle remaining fixed. This design of tank is commonly used within the casting industry because it promotes homogeneous mixing and prevents the formation of unstirred areas where settling can occur.

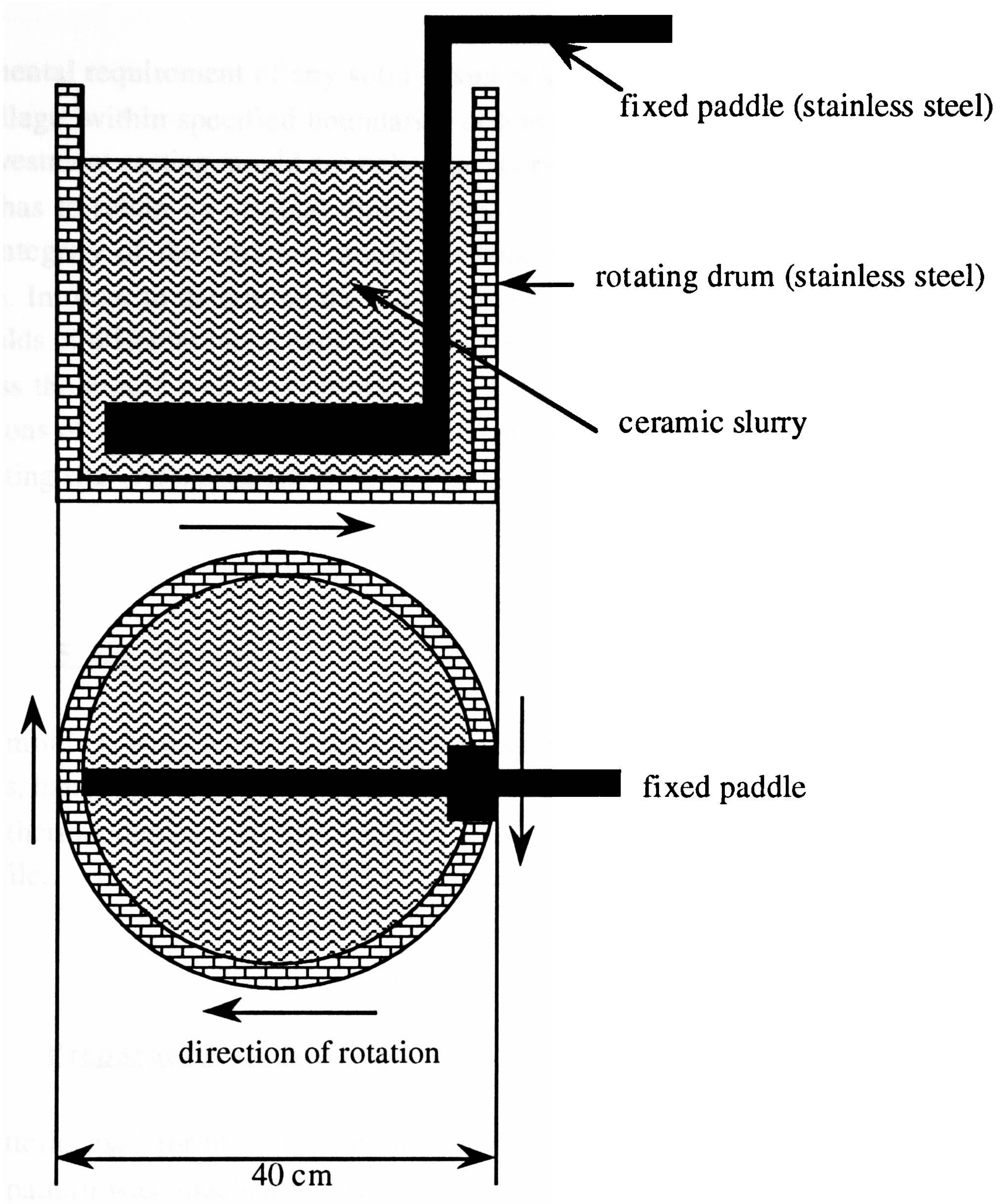


Figure 64: Experimental slurry tank at Deritend Precision Castings

The slurry formulations and control limits were identical to those used to produce slurries on a small scale in the laboratory (Section 3.9.1), with the exception that the stirrer speed is fixed and is determined by the tank design. Since the tank is within a controlled environment, the humidity and temperature was controlled and monitored.

3.10: Temperature Profile of Moulds During Casting

The fundamental requirement of any solid vessel designed to contain a liquid is to hold the liquid, without spillage, within specified boundaries of temperature and pressure for a required period of time. An investment casting mould not only must attain these requirements until solidification occurs, but it also has to retain its original dimensions during a prolonged high temperature cycle. The structural integrity of the ceramic mould constituents is dependent directly upon the operating temperature. In order to determine the temperature regime that a mould undergoes during casting, several moulds of different metal capacity were manufactured and the temperature attained at several points across the mould wall was monitored as function of time. The thermal gradients across the mould sections of both large and small components was investigated, to determine the relationship between casting size and maximum mould temperature.

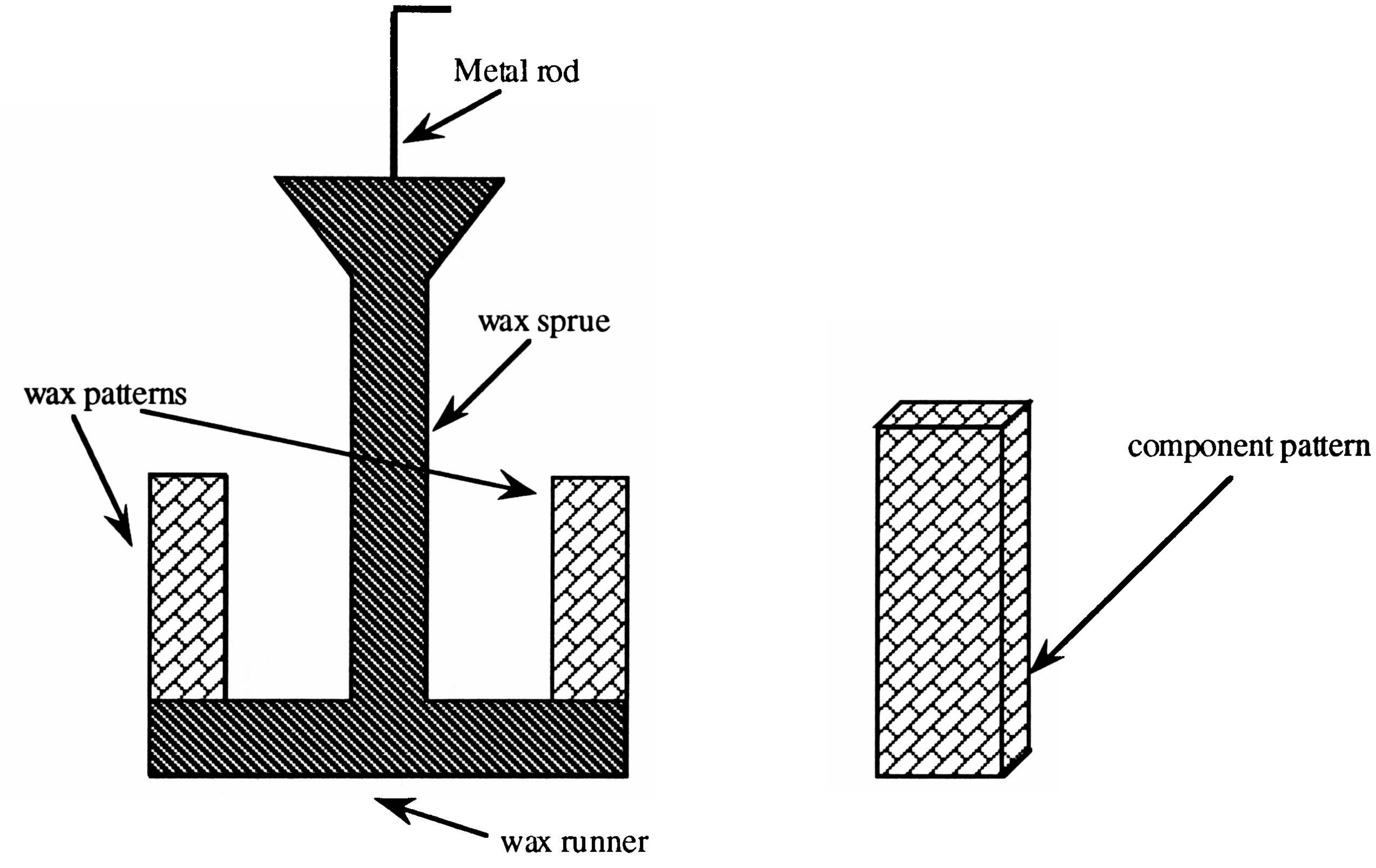
3.10.1: 5 Kg Moulds

Two small moulds, both with a 5 kg metal capacity, were manufactured using standard slurry formulations, hand dipping procedures and stucco sequences to duplicate a typical air-cast shell. Pt / Pt 13% Rh thermocouples were incorporated into the structure, between the coats, to produce the thermal profile.

3.10.1.1: Preparation of Moulds

The wax patterns used for this experiment were designed and assembled as shown in Figure 65. A component pattern was attached to each of the three runners. Care was taken that no holes, sharp edges or unattached joints were left in the wax. The components chosen were simple oblong shapes, possessing flat areas where the thermocouples could be attached, to simulate the large flat areas where bulging occurs in conventional moulds (see Section 2.7.1). The wax was degreased using acetone to ensure that all residual silica from the wax injection process was removed (silica is used as the mould release agent).

Primary and secondary slurries were prepared using the formulations in Appendices 7 and 8. The first thermocouple was embedded into the wax of the component pattern, to record the metal temperature during casting and metal cooling. The mould was then built up by dipping the wax into the slurry, and the stucco applied by rotation of the wax underneath a sieve containing the stucco material. The primary coat was allowed to air-dry for 12 hours, whereas the secondary coats were chemically set in ammonia gas for 5 minutes and then allowed to air-dry for 10 minutes. The thermocouples were placed after each stucco coating using ceramic cement to hold the ceramic sheath in place. Locations of all the thermocouples are shown in Figures 66 and 67. The moulds were dewaxed in a high pressure steam autoclave and fired at 1,000 °C (+/- 50°C) for 60 minutes.



(a) (b)

Figure 65: (a) wax runner and pattern assembly (b) component pattern

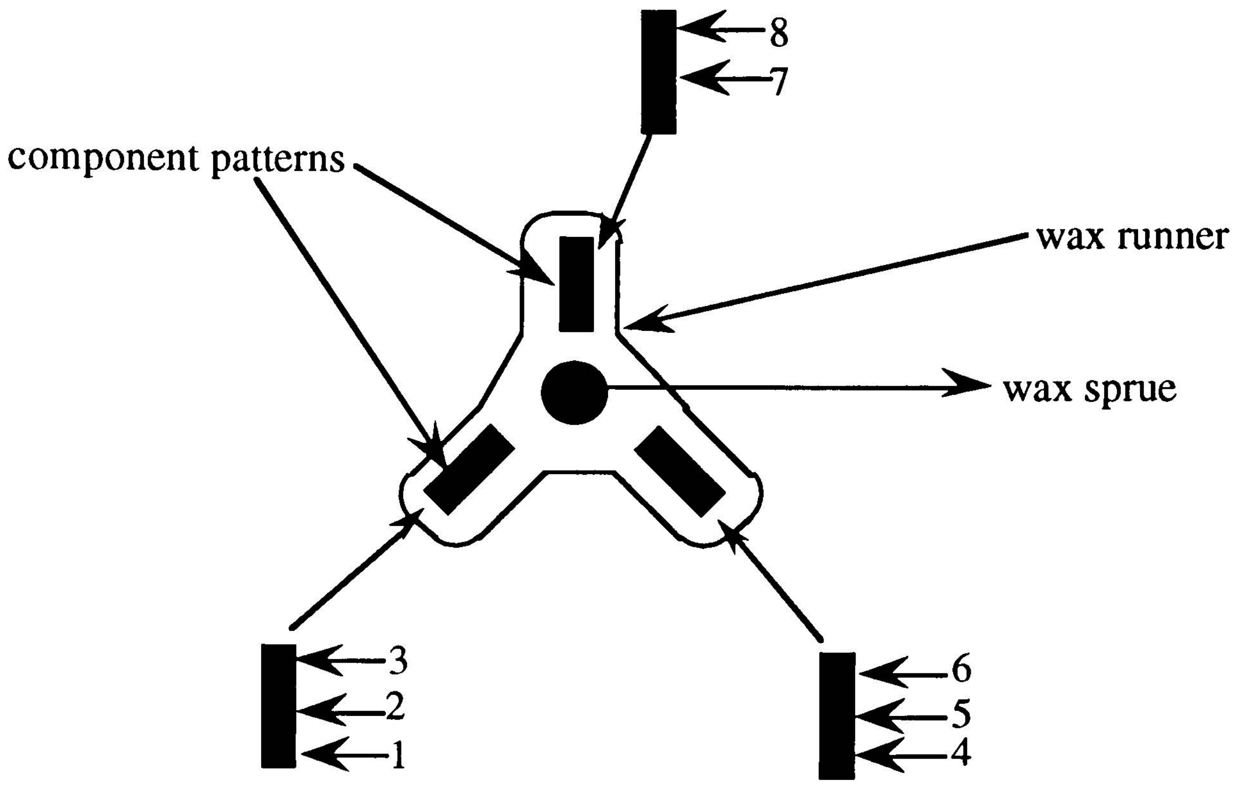


Figure 66: Relative thermocouple positions

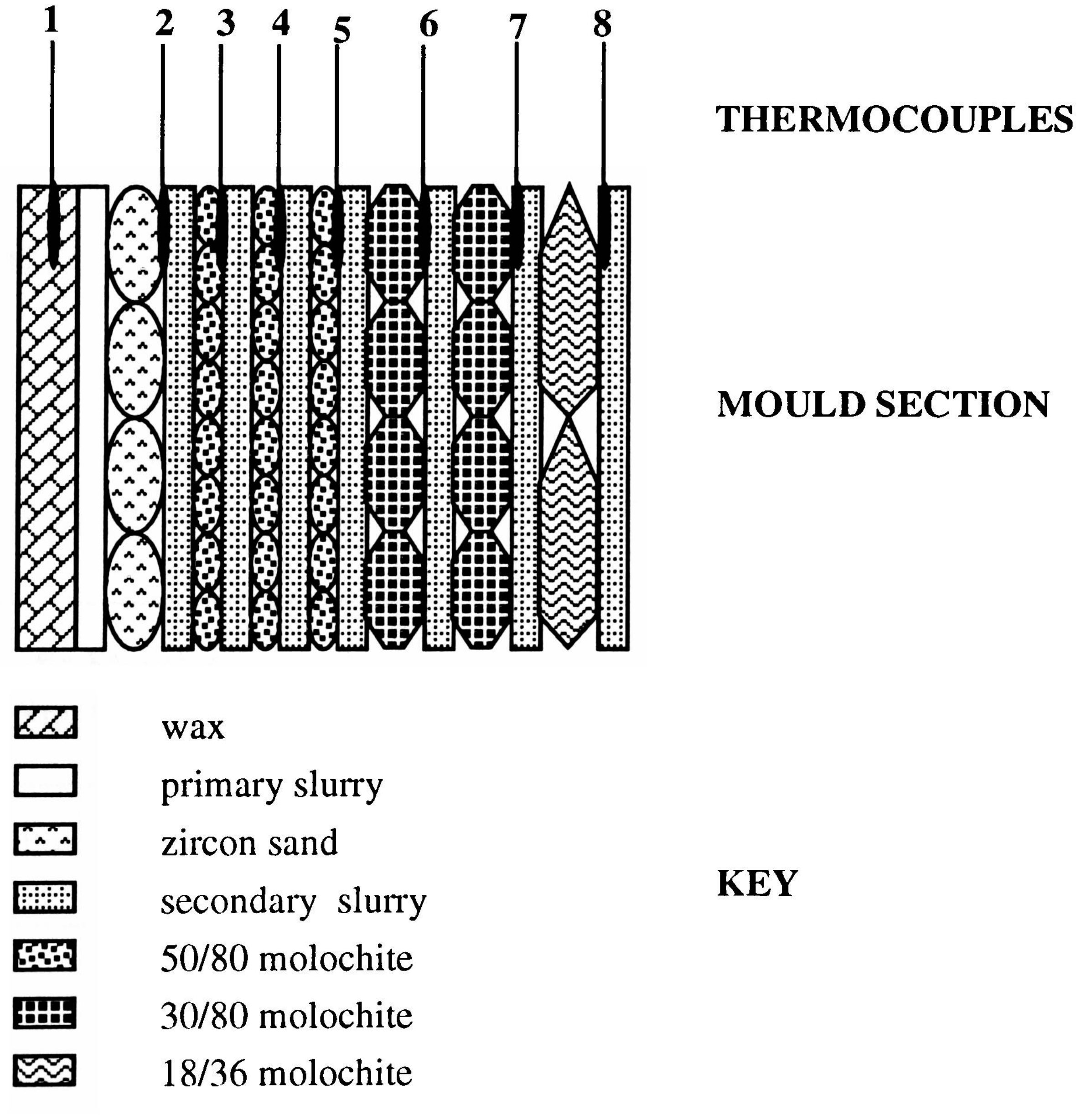


Figure 67: Slurry / stucco/ thermocouple schedule (5 Kg mould)

3.10.1.2: Casting Procedure

There are at present two main groups of air-castable metals which produce gross defects and possible quality control failure of a casting. These are low alloy and martensitic stainless steels (typical compositional analyses are shown in Table 12). Low alloy steels tend to produce hot tearing of the metal during cooling, and martensitic stainless steels produce a surface reaction called 'spotted dick'. Both of these defects are discussed in more detail in Section 2.7. These were the metals chosen for casting, so that the type and extent of metal mould interaction together with the associated defects could also be studied.

It should also be noted that the martensitic stainless steel contains 16.5% chromium. The high proportion of this element produces a single freezing point and stabilises the high temperature ferrite phase. The alloy remains single phase through to room temperature, which removes the possibility thta the cooling curves are being distorted by the evolution of latent heat during a phase change.

The melting and casting parameters for both types of steel was identical and are summarised in Table 13. The shell moulds required support during casting, and this was achieved by placing them into a metal flask and surrounding them with a refractory aggregate (sand or granular alumino silicate). A cross-sectional view of this arrangement is shown in Figure 68.

Table 12: Typical compositional analyses of low alloy and martensitic stainless steels (**Note** that the remaining element is Iron).

Element (wt%)	C	Si	S	P	Mn	Ni	Cr	Mo
Martensitic Stainless Steel	0.18	0.8	0.015	0.015	0.5	2.6	16.5	0.05
Low Alloy Steel	0.26	0.5	0.010	0.015	0.5	0.2	3.1	0.5

Table 13: Melting and casting parameters for low alloy and martensitic stainless steels

	Low Alloy Steel	**Martensitic Stainless Steel**
Shell Temperature (° C)	1000 +/- 50	1000 +/- 50
Deoxidant	aluminium granules and CSM *	aluminium granules and CSM
Weight of Deoxidant (g) (for 5Kg metal)	10g Al 20g CSM	10g Al 20g CSM
Pouring Temperature (° C)	1580	1580

* CSM is a standard mixture (by weight) of 16-20% Calcium, 53-59% Silica and 14-18% Manganese.

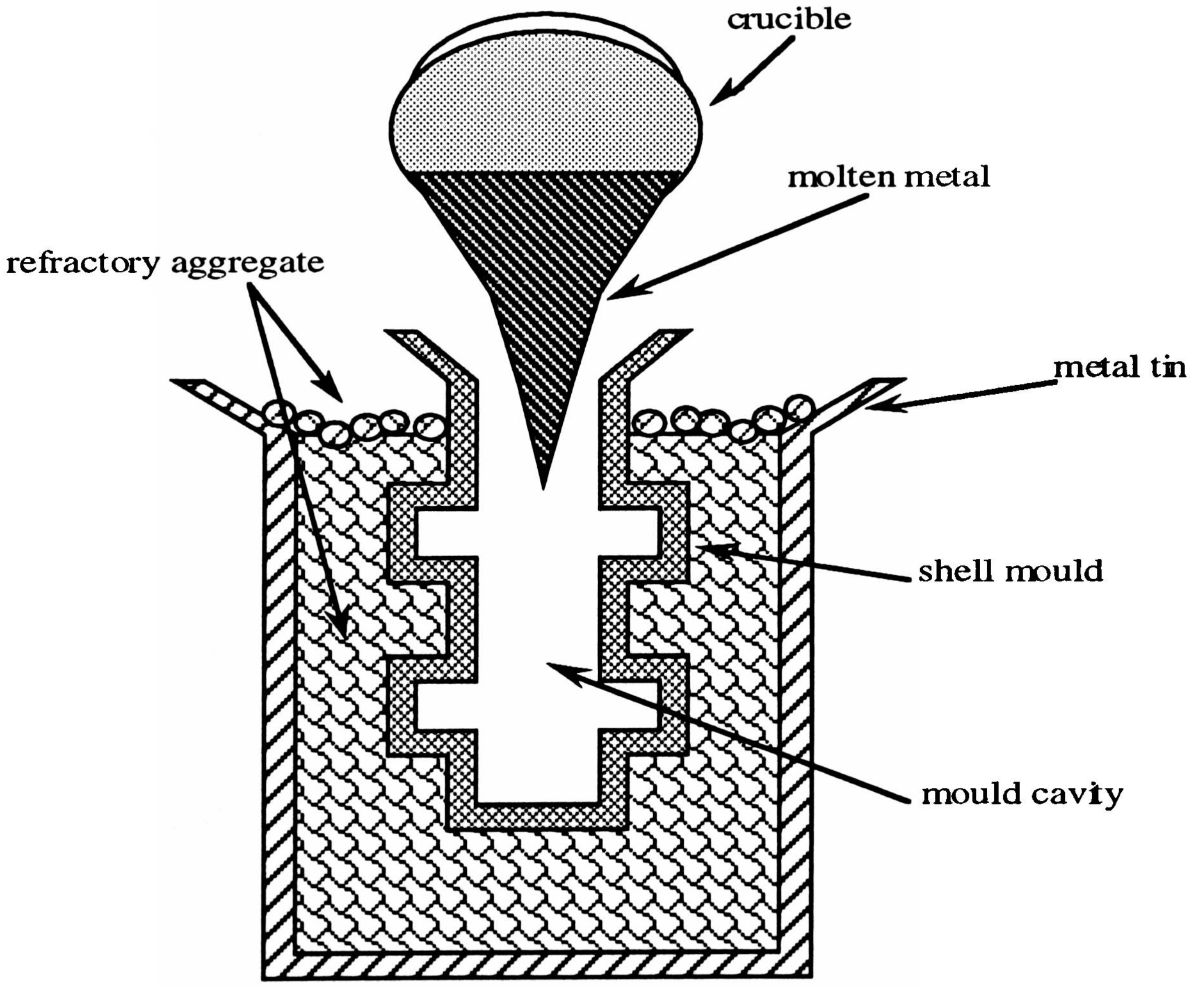


Figure 68: Cross-sectional view of a refractory backed shell mould

The entire assembly was then placed into a furnace and preheated to 1000°C (+/- 50°C) for approximately 60 minutes prior to casting. Meanwhile, the metal was melted, by electrical induction, inside a refractory lined crucible and the slag produced on the melt surface removed by skimming with a metal rod. Deoxidation was carried out by adding a mixture of elements to the melt that have a greater affinity for oxygen than the alloy. The metal was brought up to the pouring temperature and the melt surface was again cleaned by skimming. This final clean-off removed the deoxidant materials.

The mould was removed from the preheat furnace and the thermocouples attached by compensating cable to the data collector. The data analysis system consisted of an Asystant General Purpose Interface Bus connected to an IBM computer using Asystant software. The metal was poured into the mould and the software initiated. As the casting cooled the response of the thermocouples was recorded as a function of time.

3.10.2: 60 Kg Mould

One large mould with a 60 Kg metal capacity, was manufactured using standard slurry formulations, robot dipping procedures and stucco sequences to duplicate a typical air-cast shell. Pt / Pt 13% Rh thermocouples were incorporated into the structure between the coats so that a thermal profile across the mould section could be determined.

3.10.2.1: Preparation of Mould

A wax pattern used was designed and assembled as shown in Figure 69. Again care was taken that no holes, sharp edges or unattached joints were left in the wax. The wax pattern chosen was a typical large component, which possessed flat areas where the thermocouples could be attached, which again simulated the area in which bulging normally occurs in larger moulds. The wax was degreased using acetone to ensure that all residual silica from the wax injection process was removed.

Primary and secondary slurries were prepared and a mould built up in the same manner as for the 5 kg moulds (see Section 3.10.1.1). Again thermocouples were embedded within the mould section but, as the number of slurry coats was increased, the positioning was altered slightly (Figure 70). This mould was robot dipped, with the primary coat air dried for 3 hours and the secondary coats ammonia gelled before air drying for three hours also. After completion the mould was dewaxed in a steam autoclave and fired at 1,000°C (+/- 50°C) for 60 minutes.

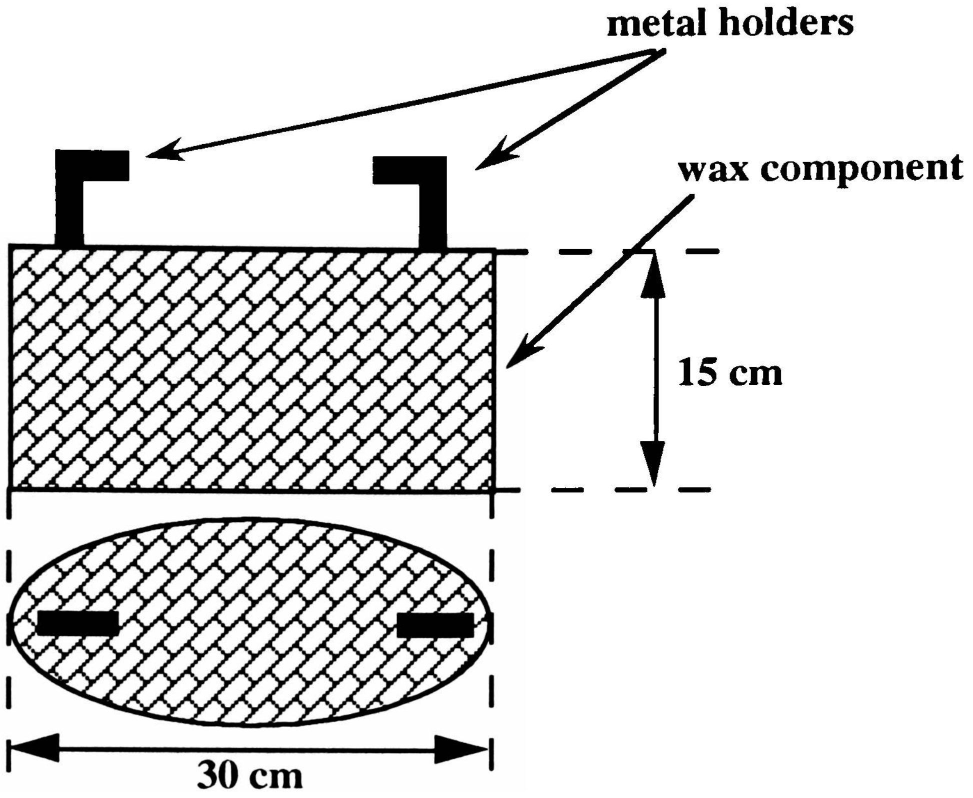


Figure 69: Schematic of wax assembly for 60 kg mould

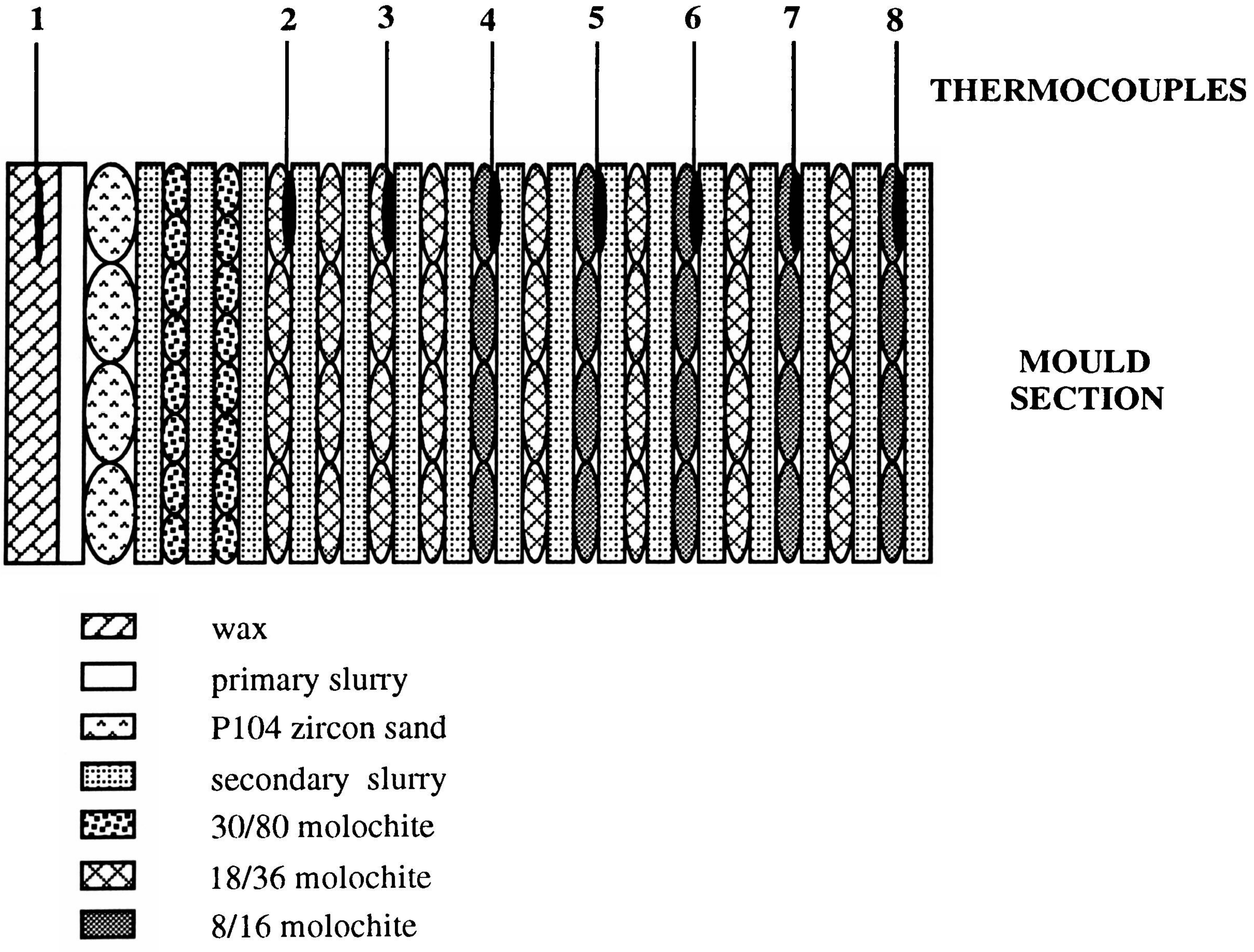


Figure 70: Slurry / stucco/ thermocouple schedule (60 kg mould)

3.10.2.2: Casting Procedure

The metal used for casting of the 60 kg mould was a low alloy steel and the specification can be seen in Table 14. The casting parameters and procedure was identical to that of martensitic stainless steel (Section 3.10.1.2) except that 0.3% by weight of aluminium powder was used as a deoxidant. Again the temperature response of the thermocouples was monitored as a function of time.

Table 14: Compositional specification for 60 kg low alloy steel.

Element (wt%)		C	Si	Mn	P	S	Mo	Ni
Low alloy 2	MIN	0.24	0.2	1.2	-	-	0.3	0.3
	MAX	0.32	0.6	1.7	0.025	0.025	0.5	0.7

3.11: Mould Strength Tests (Room Temperature)

It is useful to know the strength of a mould system in order to predict the ability of the system to withstand stresses induced during manufacture, dewaxing and casting. Two basic strength tests were carried out at room temperature; the flexural or transverse strength test and the 'bursting disc' test. Although the results obtained are useful, it must be remembered that the strength of a system at low temperatures often bears little resemblance to that at higher temperatures. This must be taken into account when using results as a basis for the prediction of overall mould behaviour.

3.11.1: Bursting Disc Test

This is a very basic test carried out upon 10 cm disc shaped mould samples. The method was designed and built at Deritend Precision Castings to be used as a simple comparative test for quality control of ceramic slurries. However, the test is prone to inaccuracies and, as such, the results obtained cannot be quoted as absolute values. If the same test procedure is followed for every sample, then the results provide a useful comparison between the strengths of various mould systems and also between green and fired samples. The test can also be used to determine the optimum firing temperature of the new binder systems.

3.11.1.1 Preparation of Samples

The discs were prepared by using a metal pattern coated in wax to facilitate removal of the ceramic (Figure 71). The pattern is dipped and stuccoed to form a shell then dried. Since some of the shell systems under investigation could not be autoclaved because of sensitivity to the presence of water vapour, sample removal was carried out by hand. This was achieved by grinding away the ceramic around the edges of the disc to produce two disc samples from every metal pattern. The wax between the pattern and sample was softened using a small gas burner and the discs removed by a gentle sliding action. Excess wax was removed with a scalpel and the samples were tested green or fired in a furnace prior to testing.

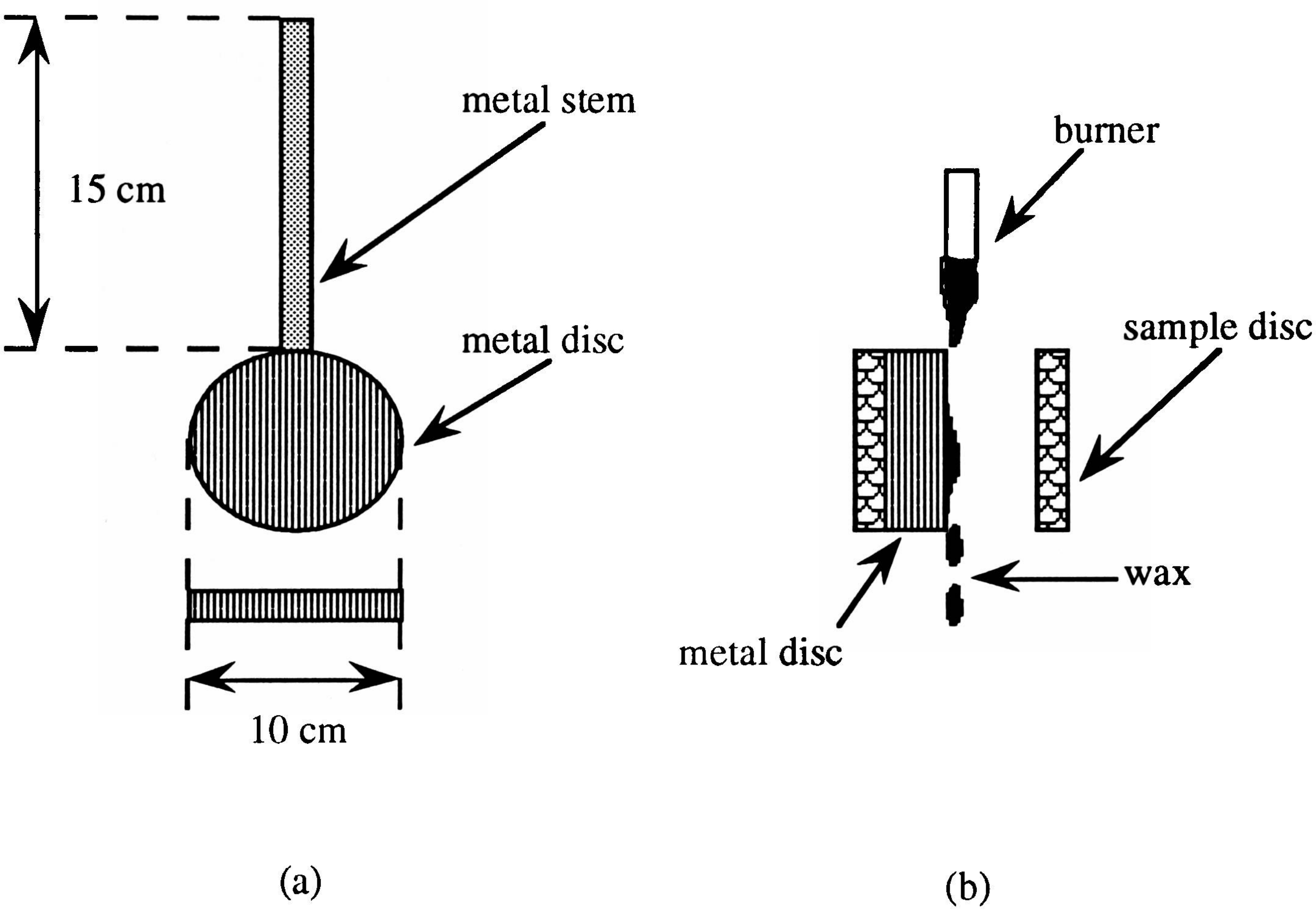


Figure 71: The bursting disc test (a) basic pattern (b) removal of test pieces

3.11.1.2 Test Procedure

The basic test apparatus is shown in Figure 72. With the plunger fully removed from the barrel (pressure = 0), the lid is removed and the sample placed on top of the rubber diaphragm. The lid is replaced tightly, effectively clamping the disc in place.

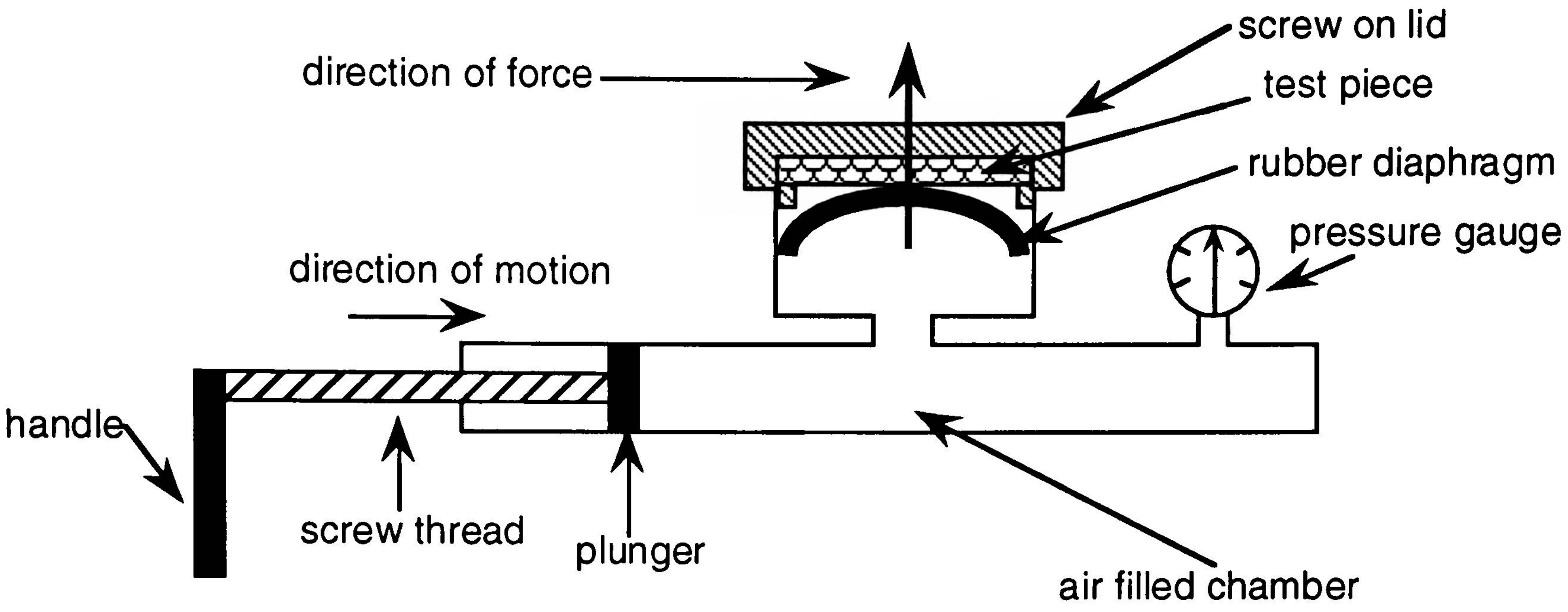


Figure 72: Test apparatus for the bursting disc test

The plunger is placed into the barrel and turned slowly, This forces the plunger into the barrel and increases the air pressure underneath the diaphragm. In this way, the diaphragm exerts pressure onto the specimen, and this pressure within the chamber is registered by a gauge. Continued force is applied to the specimen until the disc 'bursts' and therefore fails. The reading on the dial (pounds per square inch) is recorded This is the point at which inaccuracies can occur since some discs fail dramatically and others only crack slightly on the top surface and it is sometimes difficult to decide upon the exact moment of failure. To reduce this error, the point of disc failure was deemed to be the pressure at which cracks first appeared in the surface.

3.11.2: Flexural or Transverse Strength Test

The flexural strength test is one of eight principal methods of determining the strength of clays and other ceramic materials (see Section 2.6.3). The test has, to a greater extent, superseded compression or tensile tests due to the greatly reduced cost of equipment. The test piece is supported upon two triangular 'knife-edges' and a pressure is applied by a third knife-edge placed centrally on top of the test piece. The procedure is well documented in the literature(38) and many tables of typical refractory strengths have been published(130).

3.11.2.1: Preparation of Samples

Mould samples were prepared in the same manner as for the bursting disc test (Section 3.11.1.1) except that the test pieces were rectangular in shape (Figure 73).

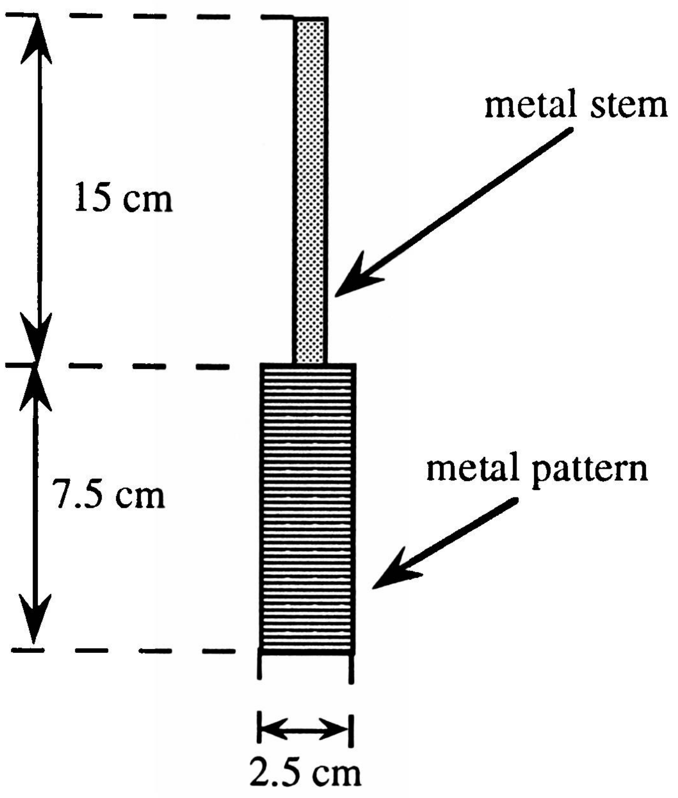


Figure 73: Metal pattern for producing flexural strength test pieces.

3.11.2.2: Test Procedure

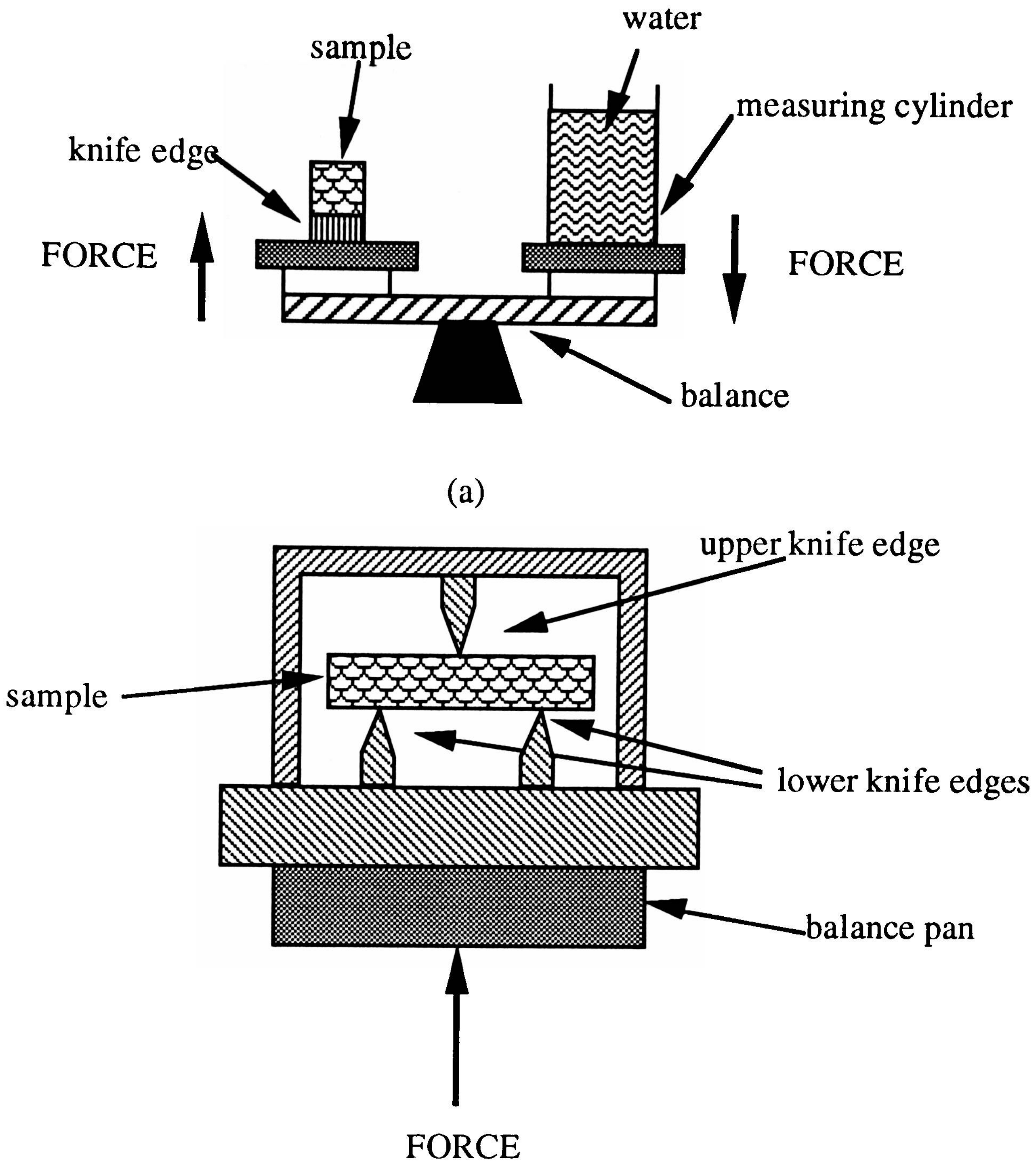
The test apparatus can be seen in Figure 74. The dimensions of the sample to be tested were measured before the sample was placed upon the supporting knife edges with the primary coat facing upwards. The loading knife edge is gently brought into place and water is added slowly to the measuring cylinder which tips the balance and forces the knife edges together. Further water is added until fracture of the test piece occurs. The failure load (M) is calculated from the volume of water using Equation 19, remembering to add the weight of the measuring cylinder itself. From this value for the failure load, the modulus of rupture can be calculated using Equation 20.

$$M = \rho \times V$$ **Equation 19**

M = mass (g)
ρ = density (g/cm^3)
V = volume (cm^3)

$$F = \frac{3\,W\,L}{2\,B\,T^2}$$ **Equation 20**

F = Modulus of Rupture (Kg/mm^2)
W = Failure Load (Kg)
L = Span between load points (mm)
B = width of sample (mm)
T = thickness of sample (mm)



(b)

Figure 74: Apparatus for flexural strength test (a) loading section (b) sample holder

3.12: Creep Tests in Three Point Bending Mode

Creep is the name given to the slow deformation of solid materials over extended periods of time whilst subject to mechanical loading(88). Creep of silica binders, initiated by high temperatures and high loads, is considered to be the the mechanism by which ceramic shell bulging, and hence casting failure occurs in large investment casting moulds. In order to investigate this theory, several creep tests at high temperature were carried out using traditional mould samples. The creep results were also used as an initial comparison between the structural performance of traditional silica bound shell samples and experimental shell systems using non-silica based binders. All tests were carried out at the National Engineering Laboratories (Scotland).

3.12.1: Principles

When a beam is loaded in the three point bend mode, one half of the beam experiences compression and the other half tension. The stress experienced across the mould wall during casting is considered to be analogous, with the primary coat experiencing maximum compression and the final secondary coat maximum tension (as long as the sample is loaded into the holder with the primary coat facing upwards. Creep tests were therefore carried out in the three point bending mode as this was considered to be more representative of the actual stress conditions encountered. A more detailed description of the theory of bending and the creep testing of ceramic materials can be found in Section 3.12.

The sample is loaded into the sample holder and a specified load is applied via the knife edges. Knife edges are use to minimise any localised stress concentrations formed at the points of contact. If the material exhibits a standard linear elastic response(132), a fixed deflection will occur as can be seen in Figure 75(a). However, if the material creeps, the deflection will continue to increase, even though the load remains the same (Figure 75(b)). This deflection is measured as a function of time by a displacement transducer and displayed as a standard deflection/time curve. If the sample holder is placed into a furnace, temperature can be added as a separate variable and the creep of a sample at fixed load whilst undergoing a variable thermal cycle can be obtained.

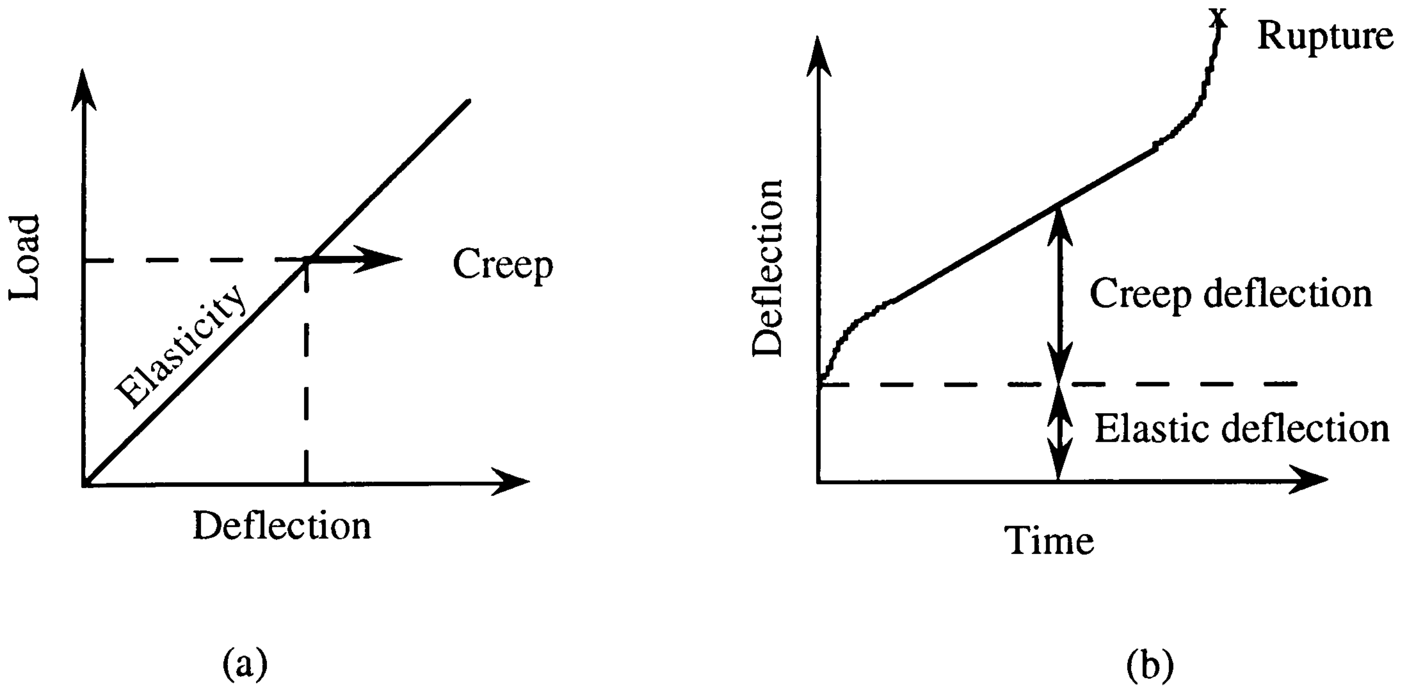


Figure 75: (a) Load deflection diagram for a material exhibiting a linear elastic response in tension
(b) Standard creep curve

3.12.2: Sample Preparation

A rectangular perspex former was coated with a layer of wax and a mould shell was built up with slurry and refractory stucco according as required (slurry formulations, drying times and stucco schedules are tabulated in Appendices 7 to 9). Individual test pieces (approximately 100 mm by 14 mm) were cut directly from the former using a diamond-impregnated cutting wheel. This eliminated the need for autoclaving, as some of the non-silica binder systems are sensitive to the infiltration of excess water. Test pieces were then fired, using temperatures and durations previously determined by thermal expansion tests (see Section 13).

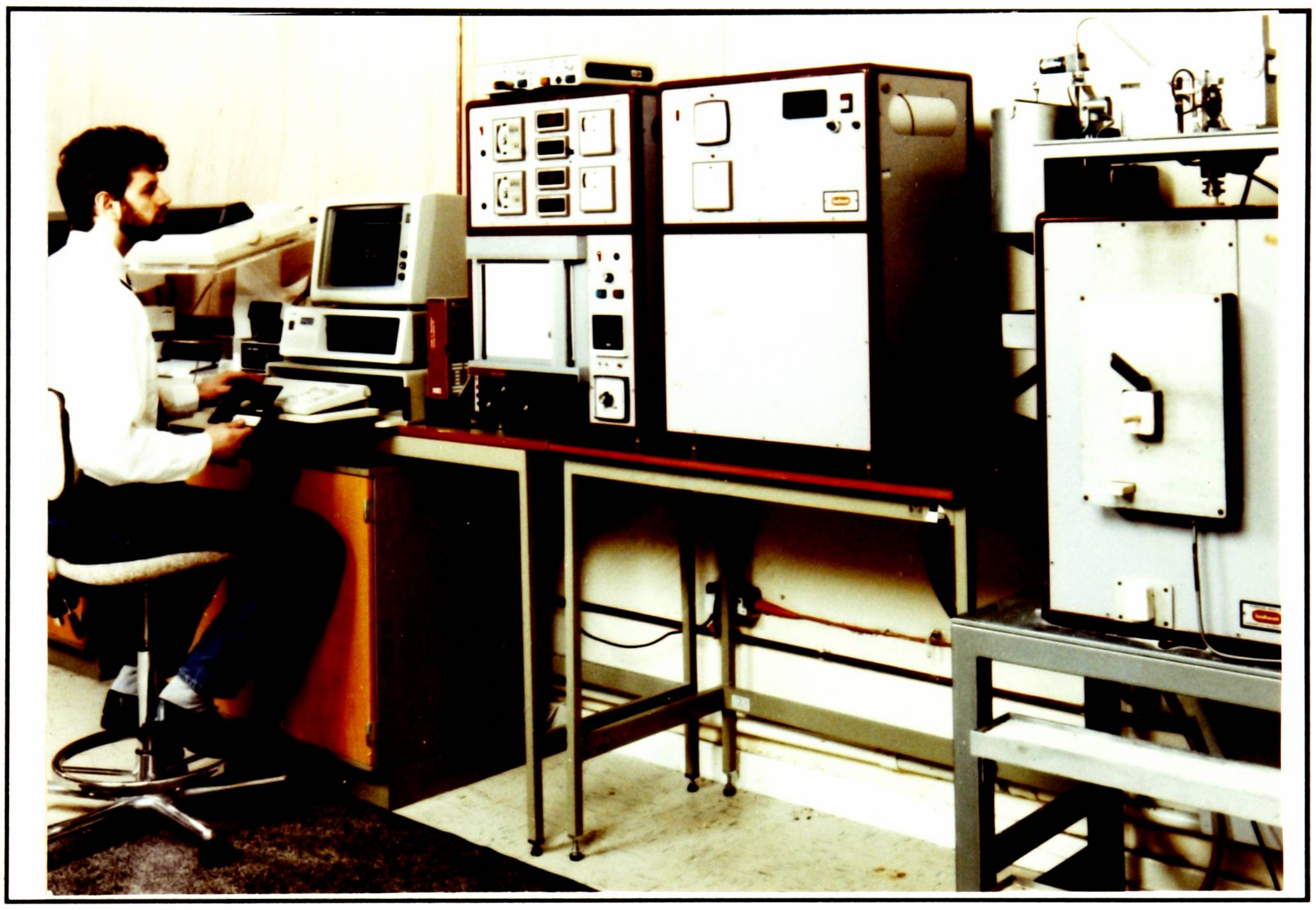


Figure 76: Creep testing apparatus (courtesy of N.E.L)

3.12.3: Testing Procedure

The thermal profile to which all of the samples were subjected during creep testing was determined by monitoring of the temperature within the primary coats (as a function of time) of a 60 kg mould during metal casting (see Section 3.10.2). The test pieces were measured and located on the test fixture (Figure 76) in an electric resistance furnace at room temperature. The test fixture consisted of two support bearing edges, each with a radius of 2.5 mm, placed 76 mm apart, and a central load application edge with a radius of 2.5 mm (Figure 77). The furnace was heated to 1,000°C at a nominal rate of 450°C per hour. The samples were held at this temperature for 60 minutes, to simulate pre-heating of the mould prior to casting, and then heated to 1420°C at a nominal rate of 450°C per hour. As soon as the test piece reached 1420°C, the load was applied manually using weights. After 50 minutes the test piece was allowed to furnace cool whilst still under load. Any deflection which occurred in the test piece was measured by a 0.5 mm resolution displacement transducer and displayed on a calibrated chart recorder.

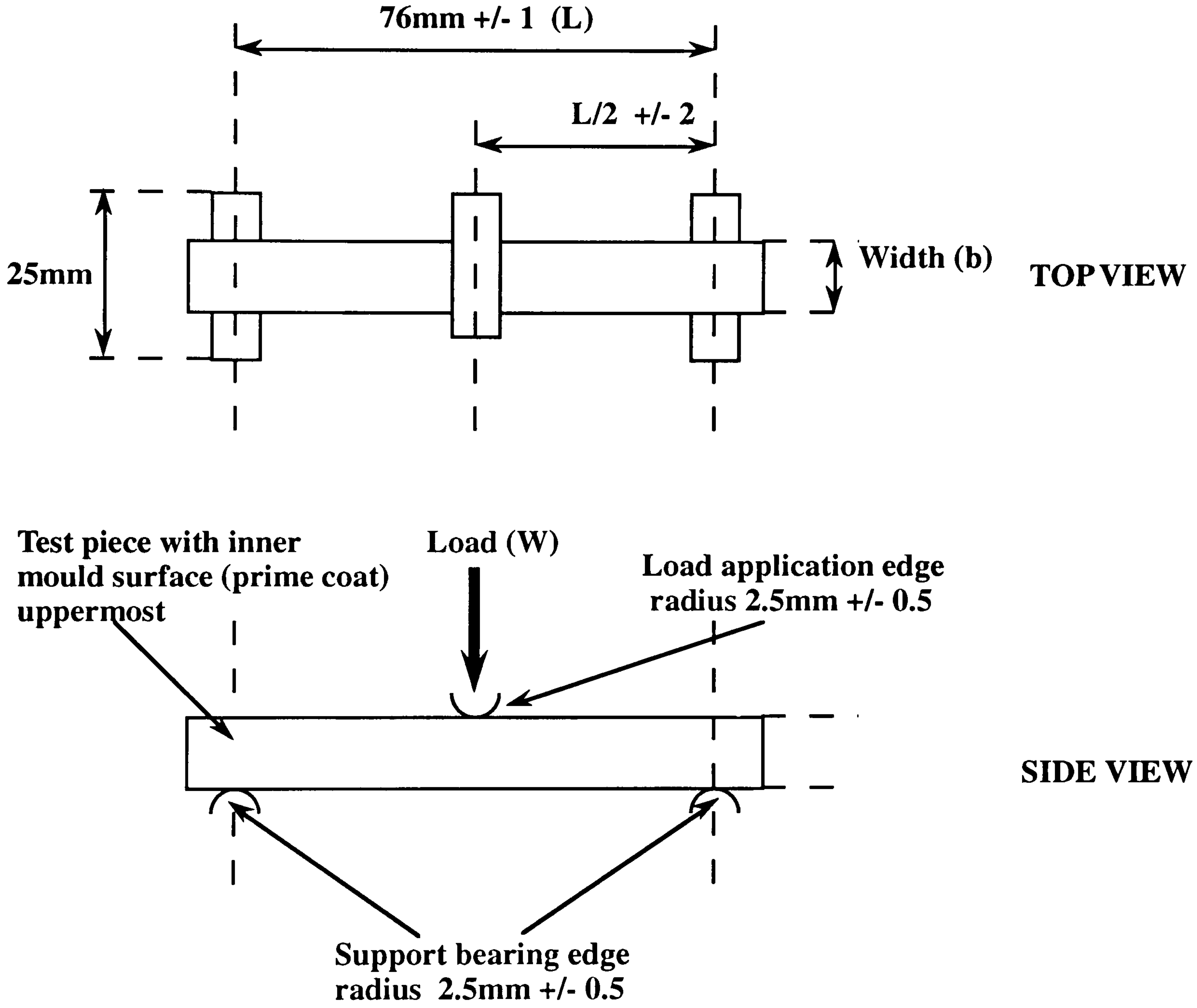


Figure 77: Creep testing apparatus (courtesy of N.E.L)

3.13: Thermal Expansion/Contraction Tests

In its green state (unfired) an investment casting mould possesses very little mechanical strength. During firing, the fundamental structure of many of the ceramic constituents is altered, a result of which is the development of sufficient strength to withstand the rigours of metal casting. The amount of strength developed is directly dependent upon the firing temperature and the duration, which for silica based systems has been thoroughly investigated(133). However, optimum firing parameters for non-silica based systems have not been categorised, and as such thermal expansion/contraction tests were carried out to determine the firing conditions for these systems. All tests were carried out at the National Engineering Laboratories (Scotland).

3.13.1: Principles

On application of heat a mould sample undergoes several fundamental structural changes. Initially a small amount of expansion will take place due to heating of the air contained within the pores. Individual refractory components also expand, the degree of which is determined by thermal expansion properties. However, as the binder itself begins to sinter the structure will contract as the fired ceramic bonds develop between individual particles. Contraction will continue to take place until the binder is completely sintered and a stable structure is reached. The temperature at which this contraction takes place is considered to be the optimum firing temperature of the mould system. Each binder system has a different sintering temperature. Thermal expansion tests were used to determine the optimum firing temperatures and durations for several silica and non-silica based mould systems.

3.13.2: Sample Preparation

Samples were prepared using the same formers and procedures as for creep testing (Section 3.12.2). For consistency samples for both types of test were dipped at the same time. Test pieces, approximately 10 mm wide and 50-55 mm in length, were again cut directly from the former using a diamond-edged cutting wheel. The end-faces were then ground down until they were flat and parallel.

3.13.3: Test Procedure

The test fixture consists of two rods to hold the specimen and one rod placed centrally between these two to hold the control thermocouple and also to provide additional support for the test piece (Figure.41). Both sample holding rods are made from 99% purity recrystallised alumina.

Of the two rods which hold the test-piece, one is fixed and the other pivots about a hinge. Any expansion in the test piece forces this rod outwards and the movement is detected by a displacement transducer. The output of the transducer (ΔV) and the temperature thermocouple (ΔT) is fed into a data acquisition devise (usually a computer) and results are obtained graphically (Figure 79). Thermal expansion as a percentage is plotted against temperature and the point at which the trace begins to flatten is the temperature at which sintering is occurring in the sample.

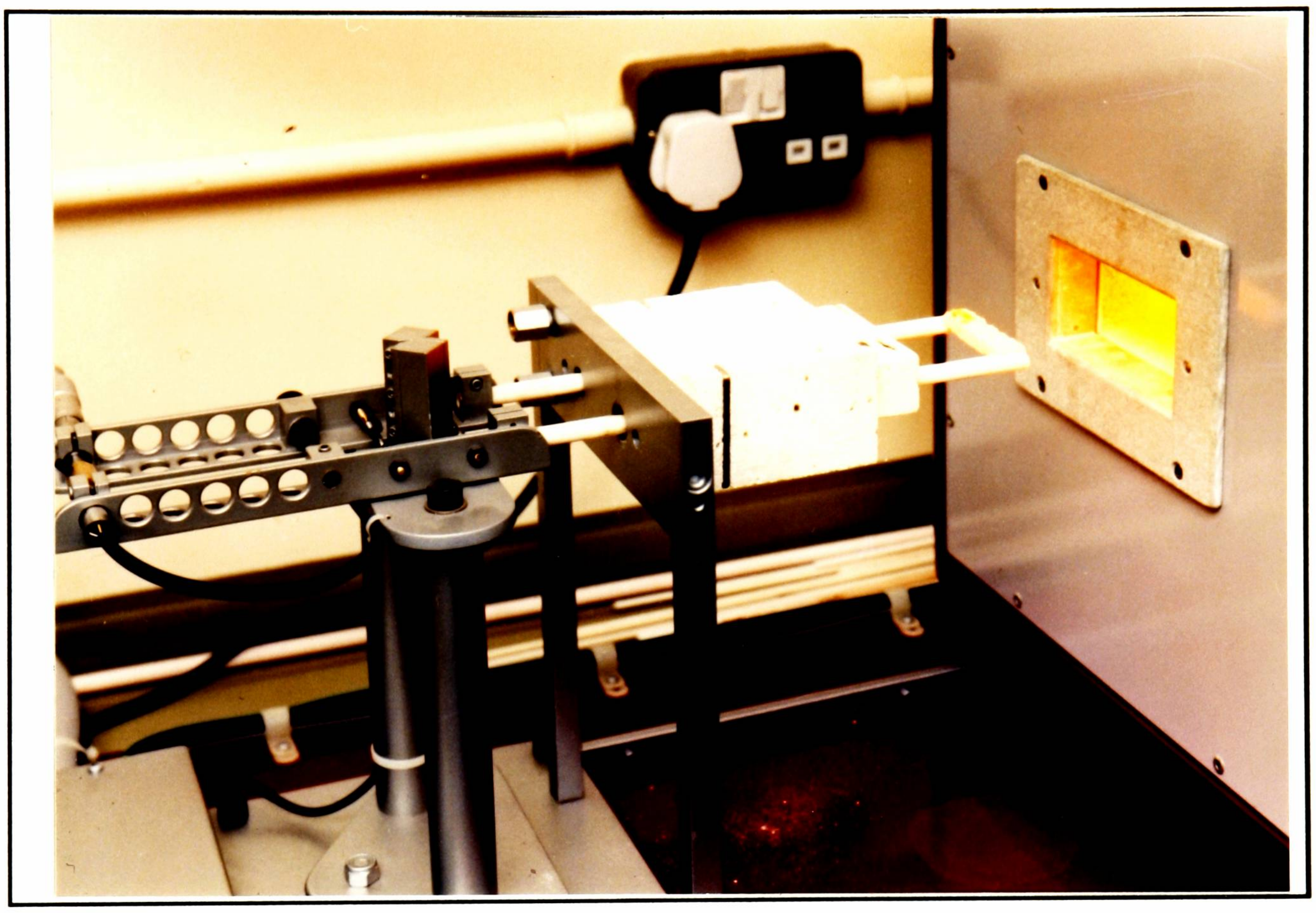


Figure 78: Thermal expansion testing apparatus (courtesy of N.E.L)

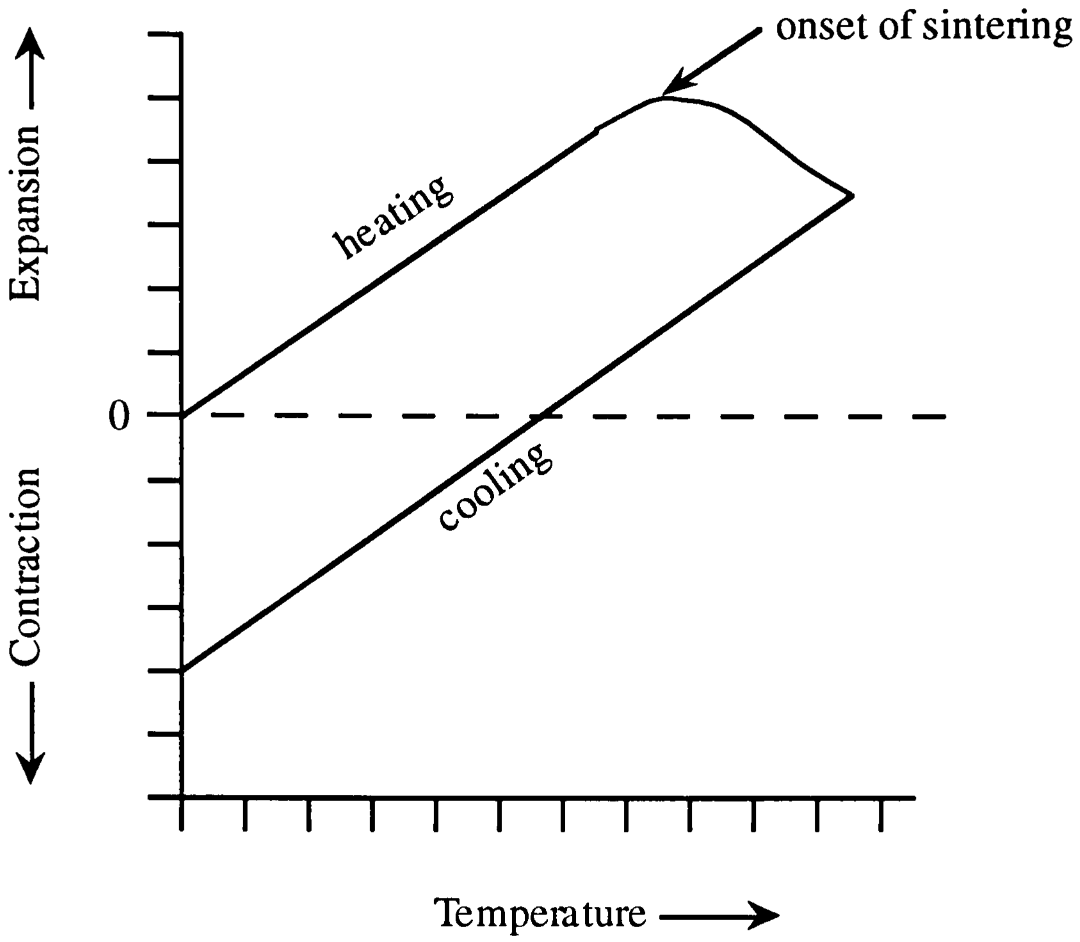


Figure 79: Standard thermal expansion trace for an investment mould sample