

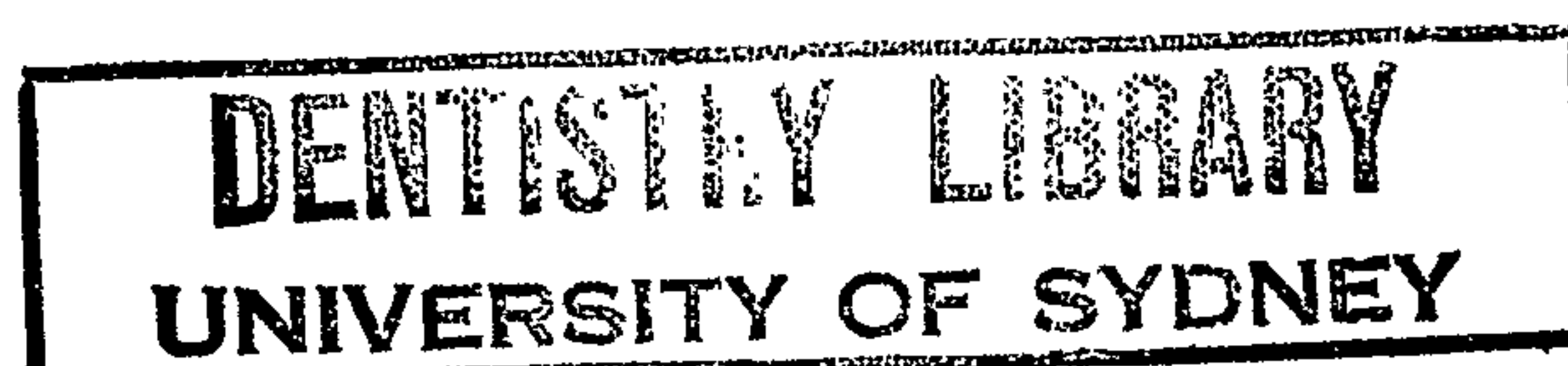
**An Investigation of the Dimensional
Accuracy and Stability of
Dental Composite Inlays**

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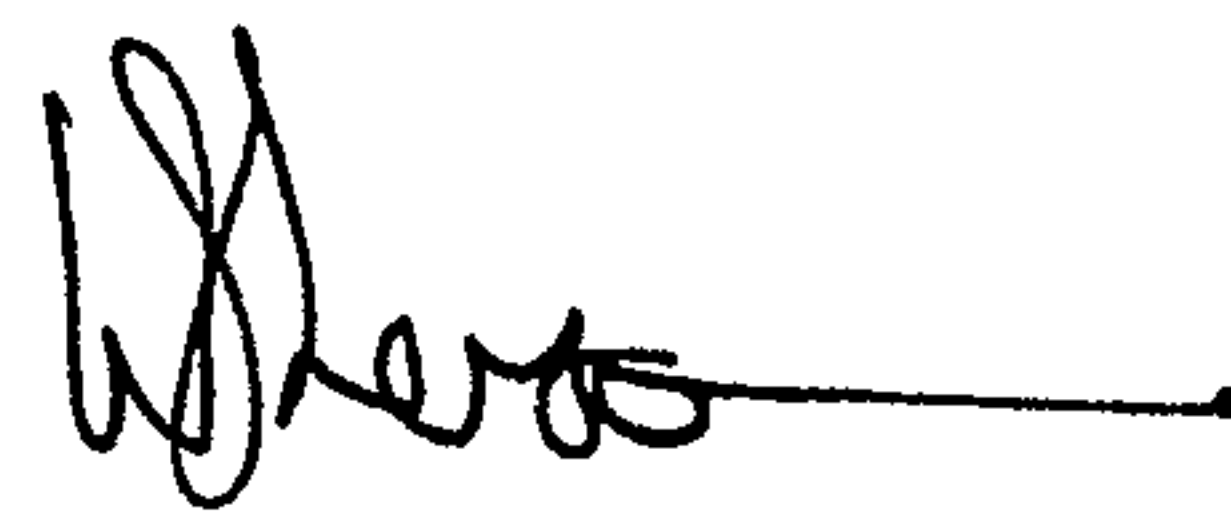
Thesis submitted in fulfilment of the requirements
for the degree **Master of Science (Dentistry)**.

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This is to certify that the work presented in this thesis was carried out by the candidate in the Faculty of Dentistry, University of Sydney, and has not been submitted to any other university or institution for a higher degree.

A handwritten signature in black ink, appearing to read 'Wayne Sherson', followed by a horizontal line.

Wayne Sherson, 1992.

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CHAPTER ONE - LITERATURE REVIEW

1.1 Introduction

Gold alloy inlays have long been fabricated by the precision casting method in dentistry. The foundation of the precision casting method is the concept of "compensation" whereby the shrinkage of gold alloys is compensated by other factors in the fabrication process, mainly, expansion of the investment material, in order to achieve a high degree of accuracy. It is the intention of this review to examine this precision casting method to apply and develop its basic principles to the fabrication of recently introduced dental composite inlay materials. In view of this, the review examines the traditional inlay fabrication in Part I and new composite inlay materials in Part II.

PART I - INLAY MAKING IN DENTISTRY

1.2 Inlay Materials

The first materials used for metal inlay fabrication were pure gold and then gold alloys (Taylor 1932) which are the longest serving materials in dentistry. Recently, the marked increase in the price of gold has led to the development of base metal alloys (Bertolotti 1989). However, both noble and base metal alloys lack any aesthetic qualities.

The first dental porcelain inlays, said to have been first

produced in the U.S.A. in 1890, were functional but lacking aesthetics (Jones 1985). Around 1900 the use of porcelain accelerated with the release of both low and medium fusing porcelains (Jenkins 1902; Jones 1985). However, failure to recognise the limitations, particularly in relation to the strength and brittleness of the material, led to a demise in its popularity (Le Gro 1921). As well, static fatigue, which is believed to be a stress dependent chemical reaction between water vapour and the surface faults in the porcelain, allows spontaneous crack propagation (McLean 1988) which may lead to failure of the restoration. The development of alumina reinforced low and medium fusing porcelains (McLean and Hughes 1965) and more recent high alumina content porcelains (Witkowski and Wohlwend 1986) with at least double the strength of ordinary glass porcelain (Johnston and O'Brien 1980; Witkowski and Wohlwend 1986) has seen a resurgence of porcelain inlay fabrication.

Another new class of material, castable glass-ceramics, has recently been developed. This type of material was first introduced to dentistry in the late 1960's as a technique of producing denture teeth by casting (MacCulloch 1968) but early research on their use as a restorative material found the material too weak (Hench et al. 1971). It was not until 1984 that a castable glass-ceramic material, Dicor, was commercially available for crown fabrication, although experimental restorations had been fabricated for several years (Adair and Hoekstra 1982; Adair et al. 1982). This material was later recommended for the fabrication of inlays (Hoekstra 1986) and a pilot clinical study (Cavel et al. 1988) reported promising results but there appears to be little published research to support this. Dicor is a tetrasilicic mica glass which is similar to the machinable glass-ceramics (Grossman 1972; Barret et al. 1980; Adair and Grossman 1984).

The castings are heat treated or cerammed to allow crystal growth within the glass. The resultant glass-ceramic has excellent aesthetic properties (Adair and Grossman 1982) but lacks strength in thin section which is compounded by the presence of porosity (Vrijhoef et al. 1988). Another mica forming glass-ceramic (Sumii et al. 1981; Sumii 1983, 1985) and a hydroxyapatite based material, Cerapearl (Hobo and Iwata 1985a, 1985b, 1985c; Hobo 1988), have been reported. However, there has been little published research on these materials.

A composite inlay material was introduced in the early 1980's (James 1983) taking advantage of the good aesthetic properties of traditional composite filling materials but with improved physical properties (Wendt 1987a, 1987b). Early clinical studies of composite inlays have reported encouraging results (Bishop 1989; Wendt and Leinfelder 1990). The new inlays are reviewed in Part II.

1.3 Dimensional Accuracy

The development of reversible hydrocolloid impression material during the 1920's, and its subsequent application to gold inlay fabrication (Sears 1937) allowed accurate reproduction of the cavity in which the wax pattern could be fabricated. The reversible hydrocolloid was improved to the stage that during the 1950's it was perhaps the only impression material being widely used for cast restorations (Asgar 1971).

From 1955 onwards a new generation of rubber base materials were developed and gained widespread use. The first of these were polysulphide rubber materials (Pearson 1955; Skinner and Cooper 1955) and this was followed by silicone based

impression materials (Skinner 1958) which are known today as the condensation cured silicone impression materials. More recently polyether impression materials (Espe 1964) and in the early 1970's, addition cured silicone impression materials have gained popularity because of their more stable dimensional properties (Sawyer et al. 1973; Sawyer and Stine 1974). These elastomers are polymers with a high coefficient of thermal expansion, $136-156 \times 10^{-6}/^{\circ}\text{C}$ (Phillips 1990a). An impression taken at 37°C will be cooled to room temperature before pouring of the die and a significant dimensional change will take place (Peppit 1984; Phillips 1990a). It appears that the effect of this on the dimensional accuracy of inlays has not been investigated even for gold casting.

Dental stones used for the indirect fabrication of restorations have setting expansion values close to zero, varying from +0.06 to +0.33 percent at 24 hours (Hiraswa and Nagamitsu 1976). Gold and glass-ceramic inlays are fabricated using the lost wax method involving removal of the wax pattern from the die. In contrast, composite and porcelain inlays are processed on the die, a stone die for the former and a refractory die for the latter.

Accuracy of porcelain restorations is achieved by layered application of the material which minimises the effects of firing shrinkage of the porcelain. However, because of the multiple heating cycles firing shrinkage will cause distortion and slumping of the marginal porcelain detracting from the accuracy of the restorations (Phillips 1990b). Studies on the accuracy of porcelain crowns will provide some information on the accuracy of porcelain inlays and it appears that an ideal cement space can be achieved (Sato et al. 1986; Woolsley et al. 1987). However, there appears to be little published research on the accuracy of porcelain inlays.

The main concern regarding accuracy of gold inlays has been the shrinkage of the gold alloy from its solidification to room temperature (Section 1.4). Today a high degree of accuracy is generally achieved with cast gold restorations (McAloon 1989). The initial cast dimensions of glass-ceramic inlays can be controlled in a similar fashion to cast gold restorations. However, further dimensional changes can occur during the ceramming procedure. This dimensional change is due to the development of crystals and the relief of stresses stored in the cast structure. The extent of this dimensional change is in the region of 1.6 percent (Wohlwend et al. 1989), although its effect on the accuracy of restorations has not been fully studied.

1.4 Assessment of Dimensional Accuracy

The introduction of the lost wax method into dentistry in 1907 and 1908, by Jameson, Taggart and Solbrig (Ward 1962; Hagman 1977) was perhaps the start of fabricating accurate cast restorations in modern dentistry. The independent efforts of these researchers and other early investigators (Price 1908, 1911; Lane 1909; Shell 1923; Van Horn 1925; Coleman 1926, 1928; Souder 1927; Rice 1929; Weinstein 1929) can be considered as the basis of inlay fabrication as we know it today.

Taggart (1907) found that the irregular expansion of investment material when heated to casting temperature proved a serious obstacle in the production of accurate castings and sought to overcome this by casting into a cold mould. He also noted that although different metals undergo different amounts of shrinkage on cooling, the castings produced from different

metals were of similar fit in the cavity. He attributed this to either the maintenance of pressure on the castings during cooling or the expansion of investment equalising the metal contractions. However, he did not attempt to measure the dimensional discrepancy or consider other factors affecting the accuracy of the restorations.

Coleman (1926, 1928) and Souder (1927), by direct measurement of cast specimens, determined the shrinkage of a gold alloy to be 1.25 percent and this remained the basis of compensation and development of investment material for many years (McAloon 1989). In light of more recent research (Finger and Jorgensen 1980) the shrinkage value is thought to be between 1.68 and 1.8 percent which is similar to that obtained by Price (1908, 1911) as well as Coleman's original measurement in 1926 using a comparator method.

Coleman (1926, 1928) and Souder (1927) were perhaps the first investigators to consider the compound effect of the factors affecting the dimensional accuracy and thus were able to present a formula to "compensate" for the gold shrinkage in attempting to fabricate accurate inlays. This involved the control of mould dimension through setting and thermal expansions of the investment material which is still the basis of the compensation for gold alloy shrinkage. One of the key features used in assessing the "compensation" of this shrinkage was the adoption of a metal die. Coleman (1926, 1928) whilst working at the National Standards Laboratory in the U.S.A. developed a die which was used by other researchers (Souder 1927; Rice 1929) and later became the standard die for the American Dental Association specification No. 2 for casting investment of dental gold alloys (American Dental Association 1962). It consisted of a mould representing an MOD inlay cavity machined from a cylindrical steel rod 12.7 mm in

diameter. It is unfortunate that the MOD die became a standard method since the accuracy of MOD inlays is difficult to assess. An oversized inlay will not fit the isthmus but will seat in the mesiodistal dimension whilst an undersized inlay will seat along the isthmus but will not fit in the mesiodistal direction.

Many designs have been developed by independent researchers for the assessment of small gold castings so that the discrepancy between a casting and the die could be measured using a measuring microscope. Taylor (1931) developed both Class I and MOD inlay dies. The dimensions of the Class I die were approximately 6.4 mm at the top, 6.2 mm at the bottom and 5 mm in height. The very small taper, one in twenty five, presented difficulties in making and assessing the fit of castings.

Fusayama (1959a, 1959b) developed a series of dies for measuring the accuracy and distortion of castings. Although one of the dies was representative of a Class I inlay, most of the research was based on Class III or MOD dies. His method was unique in that the system allowed the measurement of both undersized and oversized restorations as well as the degree and direction of any distortion occurring. However, the thin dies (2 mm) were not representative of clinical restorations.

A more realistic die system was designed by Sumii (1971) for the measurement of wax patterns. These were machined from stainless steel and were representative of both MOD and Class I inlays. The dimensions of the Class I cavity were 7.6 mm at the top, 7.1 mm at the bottom and 5 mm in height providing a taper of one in ten. The MOD cavity was machined from a cylindrical rod 9 mm in diameter. The dies were designed to measure both oversized and undersized patterns by

means of measuring the protrusion at either side of the die. Matsumoto (1974) used the same MOD die and a full crown die of similar dimensions for the measurement of gold castings. He also connected two dies to assess the accuracy of bridgework. Similar metal dies, 9.8 mm in diameter, have been used for the assessment of gold crown accuracy by Morey (1985) and McAloon (1989). Their measurements were unique and probably more accurate as they compared the discrepancy between wax pattern and casting.

PART II - DENTAL COMPOSITE INLAYS

1.5 Materials and Processing

The first composite inlay material, Isosit, reported by James (1983) is similar to a crown and bridge resin, Pyroplast SR, which appeared in the late 1960's; they were unique in employing a high processing temperature, 120°C (Isosit) and 135-140°C (Pyroplast) in place of the conventional cold curing. To make heat curing possible the liquid contained large amounts of cross linking agents composed of ethylene glycol dimethacrylate which had a high boiling point, 162°C at 2mmHg (Hiraswa and Atsuta 1968) which is higher at atmospheric pressure and this increased the curing time. The incorporation of the dimethacrylate and the high processing temperatures resulted in a highly cross linked structure with improved physical properties compared to the conventional methacrylates (Atsuta et al. 1969, 1971, 1972). However, the new material had a poor resistance against water sorption because of the hydrophilic bond of the ethylene glycol units (Atsuta et al. 1969). An alternative polymer, urethane dimethacrylate,

appeared in the mid 1970's in both direct filling resins, Durafill and Isopast (Miyazaki et al. 1981), and a crown and bridge resin, SR Isosit-N (Atsuta and Nakabayashi 1979). The structure is analogous to Bis-GMA with the bisphenol-A groups being replaced by aromatic isocyanate groups (Bayne 1989) making it more flexible and requiring little or no dilution with cross linking agents such as TEGDMA (Dulik et al. 1981). SR Isosit-N was also unique in that it was cured by simultaneous application of heat and pressure, silica reinforced and contained prepolymerised particles (Hirasawa et al. 1979). The physical properties of this material were comparable and often superior to the methacrylate and Bis GMA based crown and bridge resins (Hirasawa et al. 1979).

Most new composite inlay materials utilise light for primary curing. This reflects the development on composite filling materials from chemical to light curing. However, the manufacturers recommend the utilisation of heat for post curing. Composite inlay materials available today can be classified according to the method of curing, composition of monomer and the type and size of filler particles and they are shown in Table 1.1.

Isosit is unique in that it is the only material which is cured by heat. Furthermore, a simultaneous application of pressure, which is more correctly termed hydrothermal processing, is employed. This allows for an indirect fabrication technique only.

In contrast, the other materials are light cured and either indirect or direct fabrication technique may be used (Hussey 1988; Biederman 1989; Burke et al. 1991). The manufacturers recommend post curing with either heat (Clearfil, EOS), light (P-50) or a combination of both (Brilliant, Kulzer).

Table 1.1 : Composite Inlay Materials

NAME	MANUFACTURER	PRIMARY CURING METHOD	FILLER	MONOMER
Isosit Inlay/ Onlay	Ivoclar	heat	microfil	UDMA
Clearfil CR Inlay	Kuraray	light	hybrid	Bis GMA
Brilliant	Coltene	light	hybrid	Bis GMA
Kulzer Inlay/ Onlay	Kulzer	light	hybrid	Bis GMA
EOS	Ivoclar	light	microfil	UDMA
P-50	3M	light	hybrid	Bis GMA

In composite filling resins the amount of residual monomer can be as high as 25 to 56 percent after storage at 37°C for 24 hours (Ruyter and Svendsen 1978; Chung and Greener 1990). This is significantly reduced by increasing the curing temperature (Wu 1983). This in turn improves the physical properties (Wendt 1987a, 1987b; Bausch et al. 1981) and wear resistance (Lutz et al. 1984). As well, the duration of the polymerisation reaction decreases and a high degree of polymerisation is achieved in a relatively short time (Wu 1983) but the degree of polymerisation shrinkage may also increase. As the application of heat to a restoration in the mouth is difficult and possibly damaging to the tooth and pulpal tissues, one of the most important characteristics of composite inlay fabrication is the utilisation of heat which is not possible with composite filling materials, thereby minimising the limitations associated with incomplete polymerisation (Bryant 1987).

1.6 Polymerisation Shrinkage

The apparent major factor affecting the dimensional accuracy of composite inlays is polymerisation shrinkage which is analogous to the metal shrinkage in gold inlay casting. Relevant values are available for composite filling materials and the shrinkage has generally been measured by the use of a dilatometer (Smith and Schroonover 1953; Macchi and Craig 1969; de Gee and Davidson 1981). This involves placing the specimen in a liquid filled container, analogous to a thermometer. The change in height of the column of liquid can be recorded to indicate the dimensional change of the specimen per unit volume. This also provides linear shrinkage, approximately one third of the volumetric change.

Poly(methyl methacrylate) direct filling resins were subject to a high degree of polymerisation shrinkage, between 6 and 9 percent by volume (Smith and Schroover 1953). The addition of inorganic fillers significantly reduces these values. The Bis-GMA resin developed by Bowen (1962, 1965a, 1965b) which contained 70 percent vitreous silica by mass (approximately 55 percent by volume), exhibited a volumetric shrinkage of 2.7 percent (linearly 0.9 percent). Comparable values have long been the target for compensation in gold inlay fabrication (Section 1.4).

Lee et al. (1969) used a mercury dilatometer to measure the volumetric shrinkage of four chemically cured composite filling materials and Macchi and Craig (1969) used a similar technique to measure the shrinkage of high filler content composites. The filler content of these materials were mostly similar (70-73 percent by mass) to that of Bowen's experimental resin (Peterson et al. 1966; Macchi and Craig 1969). The volumetric shrinkages 1.5-2.1 percent (Lee et al. 1969) and 1.2-1.3 percent (Macchi and Craig 1969), were considerably lower than that given by Bowen (1963), although no comment was made on this particular point. There appear to be no published data on the linear polymerisation shrinkage of composite inlay materials.

Reducing the particle size allows an increase in the filler content which in turn reduces the polymerisation shrinkage (Munksgaard et al. 1987). However, this was counteracted by the associated increase in viscosity which reduces the flow of resin during the initial stages of polymerisation (Asmussen 1975) as well as detracting from the handling properties of the filling material (Dulik et al. 1981)).

The early composite resins had filler particles in the range of 10 to 100 μm which according to current standards would be classified as macrofills. Further reductions in particle size to 1 to 10 μm produced conventional filled composites and filler particles of less than 1 μm (usually 0.01 to 0.1 μm) are known as microfills. Hybrid composites incorporate a range of particle sizes to accommodate the good finishing properties of the microfills whilst maintaining the better handling properties of the conventional filler composites. A further development is the incorporation of prepolymerised microfills which result in an increase in the apparent particle size to maintain a high filler loading and to improve the handling properties (Ehrnford 1981).

The Bis GMA monomer has an extremely high viscosity (Dulik et al. 1981) and this will determine the viscosity of the composite (Ferracane et al. 1981) which in turn affects the mixing, handling, bonding and repair characteristics (Braden 1977, Dulik et al. 1981). The resin matrix viscosity has been reduced by dilution of the Bis-GMA with other difunctional monomers with a much lower viscosity such as triethylene glycol dimethacrylate (TEGDMA) (Cowperthwaite et al. 1979, 1981, Dulik et al. 1981).

The type and concentration of the dilutant affects the degree of polymerisation shrinkage because the dilutant molecules are larger, increasing the volumetric shrinkage (Asmussen 1975). It will also affect some of the physical properties such as thermal expansion (Dulik et al. 1981), water sorption (Cowperthwaite et al. 1979) and the handling and storage characteristics (Dulik et al. 1981) and may also influence adhesion and duration of polymerisation (Asmussen 1975). There have also been attempts to modify the structure of the monomer such as the removal of the pendant hydroxyl groups as well as

replacing the bisphenol-A portion of the molecule with aromatic isocyanate groups to form urethane dimethacrylate (Section 1.5). Generally these changes have had very little effect on the degree of polymerisation shrinkage of the composite resin (Bayne 1989).

The use of monomers that expand on polymerisation (Endo and Bailey 1974) has failed to produce composite restoratives with significantly improved properties. Similarly, the use of fluorocarbon based polymers, which are hydrophobic and attempt to reduce the problems associated with water sorption, increased both the polymerisation shrinkage and the coefficient of thermal expansion (Douglas et al. 1979; Douglas and Craig 1982).

Under ordinary conditions the polymerisation shrinkage of composite filling materials will result in the formation of a gap between the filling and the cavity wall (Branstrom et al. 1984; Davidson et al. 1984; Munksgaard et al. 1985) providing a site for bacterial invasion and subsequent disease action (Branstrom and Vojiovic 1976). If the polymerisation shrinkage is unrestricted, the resultant gap should reflect the linear shrinkage of the composite filling material. However, in a cavity the polymerisation shrinkage will be restricted by many factors and the resultant gap is not so readily predictable (Asmussen 1975; Munksgaard et al. 1987).

Wall to wall polymerisation shrinkage is the term used to describe the effective shrinkage that occurs. The method of measuring wall to wall polymerisation shrinkage has involved placing a composite filling in a cylindrical cavity cut in enamel and dentine (Asmussen and Jorgensen 1972) or in dentine (Munksgaard et al. 1987), curing the material and then measuring the gap formation through a microscope. The values

obtained ranged from 0.1-0.8 percent (Asmussen and Jorgensen 1972; Munksgaard et al. 1987). In an attempt to explain these values, Jorgensen (Asmussen 1975) proposed a model for wall to wall shrinkage in which the gap formation was dependent on the adhesion forces between the material and the cavity wall, the volumetric contraction of the composite and the viscosity of the material.

In the initial stages of the polymerisation reaction the forces generated are small (Bowen 1967), presumably smaller than the adhesive forces, and the viscosity of the material is low. Therefore the flow of the material will compensate for the shrinkage. As the material polymerises, the viscosity will increase (Jacobsen et al. 1977) restricting the flow of the material and the forces generated in the material will increase (Bowen 1967). When the force from shrinkage becomes larger than the forces of adhesion, gap formation occurs.

Although the filler content is important in reducing volumetric shrinkage, it may not be as significant in wall to wall shrinkage as no direct relationship between filler content and wall to wall shrinkage was shown in composite filling materials with low dilutant content (Asmussen 1975). Asmussen attributed this to the increased viscosity of the material associated with the increased filler content impeding the flow of the material in the early stages of polymerisation. However, other researchers have shown a positive correlation between filler content and wall to wall polymerisation in composite filling materials with a higher dilutant content (Munksgaard et al. 1987) and commented that the size and shape of the filler particles and its effect on viscosity were important considerations. Munksgaard et al. (1987) partially attributed the discrepancy in results between these two studies to the effect of the dilutant monomer

content on the viscosity of the materials as Asmussen (1975) found a positive correlation between the gap width and the dilutant monomer content.

Processing the material outside the mouth will minimise these effects as the resultant gap between the inlay and the cavity wall may be filled with a luting agent. Application of heat will provide an opportunity of completing the polymerisation, although this will increase the degree of shrinkage (Section 1.7).

1.7 Dimensional Accuracy

The historical steel die method (Section 1.4) has been applied to measure the accuracy of both composite filling and inlay resins.

Bowen et al. (1982), using a die developed by Taylor (1931), obtained reproducible measurements for polymerisation shrinkage and hygroscopic expansion in composite filling materials. Suzuki et al. (1990) used the die developed by Fusayama and found that the U-shaped composite inlay restorations did not seat completely on the die. This gave an average linear shrinkage of 0.63 percent after the initial light-curing of the material (Clearfil CR Inlay). This was further increased to 0.71 percent after 15 minutes of heating at 100°C. Despite this the external dimension of the restorations were very close to that of the die both before and after heating. However, they did not discuss the large discrepancy between the internal and external dimensions of the restorations. In a subsequent paper (Kobayashi et al. 1991), they investigated methods of compensating the

dimensional change using the same die.

The direct technique of inlay fabrication may create difficulties in shaping and finishing the interproximal regions (Martin 1990) and in removing the restoration from the cavity (Sheth et al. 1989; Candio 1988). Subsequent heating may induce distortion (Candio 1988; Schneider 1989) and the accuracy of these procedures has not been fully investigated.

The indirect method will overcome many of these problems. However, in the case of Isosit the stone die is subjected to both heat (120°C) and pressure (6×10^5 Pa). This may provide ideal conditions for the dehydration of dental stone which results in a significant loss in the strength of the die (Hasratininsih 1987). Consequently a second die is probably needed for fitting and finishing of the restoration. The extent and effect of die breakdown on the dimensional accuracy of the restorations has not been studied. As well, the indirect technique is more costly and time consuming (Christensen 1989; Schneider 1989).

1.8 Dimensional Stability

Dimensional stability is not generally discussed for the metal and ceramic inlays because their thermal expansion coefficients; gold alloys, $14 \times 10^{-6}/^{\circ}\text{C}$ (Craig 1985), porcelain, $8 \times 10^{-6}/^{\circ}\text{C}$ (Grossman 1983) and glass-ceramics, $7 \times 10^{-6}/^{\circ}\text{C}$ (Grossman 1983) approximate natural tooth structure, $11 \times 10^{-6}/^{\circ}\text{C}$ (Souder and Peters 1920). These materials are not adversely affected by water sorption and they are more durable than polymers. In contrast, there have been extensive studies on the effects of water sorption and

the abrasion resistance of composite filling materials.

Any restorative material placed in the mouth will be subject to temperature change of 4-55°C (Nelsen et al. 1952). Therefore it is ideal that the restorative material has a coefficient of thermal expansion similar to that of tooth structure so that it does not induce any undue force in the cement or tooth structure. In fact the original attempts of adding inorganic fillers to dental polymers were not to reduce the polymerisation shrinkage but rather to reduce the coefficient of thermal expansion to a value comparable with tooth structure (Rose et al. 1955; Bowen 1956).

Coefficients of thermal expansion of dental unfilled resins have been reported to be in the range of $92-127 \times 10^{-6}/^{\circ}\text{C}$ (Rose et al. 1955; Bowen 1956; Stanford 1971). This was greatly reduced by addition of inorganic fillers to the range $27-41 \times 10^{-6}/^{\circ}\text{C}$ (24-88°C), and some even approached a range similar to that of tooth structure, although the latter were not viable restorative materials (Macchi and Craig 1969).

Therefore, the thermal expansivity of composite filling material appears to be still three to four times that of tooth structure. More importantly, Peterson et al. (1966) found that the reported reduction of the coefficient of thermal expansion for a high filler content composite (Addent) was not reflected in the leakage patterns of restorations in vitro. Asmussen (1974a, 1974b) found that thermocycling composite filling materials between 37 and 50°C had little effect on the width of the marginal gap. There appears to be no published research on the coefficient of thermal expansion of composite inlay materials and the effect of temperature variation on their dimensional change.

Unlike gold alloys, porcelain and glass-ceramics, composite filling materials will undergo substantial dimensional changes due to the absorption of oral fluids (Hirasawa 1965, Hirasawa et al. 1968). In fact there is a direct correlation between the mass of absorbed water and the degree of expansion (Hirasawa et al. 1983). Generally there is a variety of data on water sorption in the literature that is difficult to compare because of variations in storage times and the way in which the uptake is expressed (Paffenbarger and Rupp 1974). The water sorption of a selection of composite inlay resins ranged from 6.5 to 23.5 mg/mm³ (Takahashi et al. 1989). Both the resultant dimensional change and the effect of water sorption on the physical properties of the materials were not studied.

Bowen et al. (1982) used the die method (Section 1.7) to directly measure the linear hygroscopic expansion of composite filling resins. This ranged from 0.07 to 0.56 percent for a group of commercial and experimental composite filling materials. They estimated that the time required to achieve maximum expansion for most of these materials was greater than one year. Von Kollmannsperger and Venz (1980) suggested that hygroscopic expansion may compensate for the polymerisation shrinkage of composite filling materials. Hirasawa et al. (1983) found that the hygroscopic expansion did not compensate the polymerisation shrinkage after seven days immersion. In contrast, Bowen et al. (1982) found that it was possible to compensate polymerisation shrinkage with hygroscopic expansion with some of their experimental composites after a much longer immersion time.

Water sorption appears to occur by diffusion through the polymer matrix (Braden et al. 1976; Braden and Clarke 1984) and is dependent on many factors including the type of polymer

and concentration of copolymers and plasticisers (Kalachandra and Turner 1987a, 1987b; Ohtani 1987) and the size, shape and content of filler particles (Soderholm 1983; Braden and Clarke 1984; Fan et al. 1985). Poly(methyl)methacrylate based composites accommodate additional water at the filler matrix interface (Kalachandra 1989). Therefore, differences in the filler matrix bond will influence water uptake (Beech and Brown 1972). Water sorption plays a major role in the failure between filler and matrix (Soderholm 1985; McKinney 1985; Draughan et al. 1985) as well as leaching out filler elements (Soderholm 1983) and this induces filler failures and filler matrix debonding (Soderholm and Calvert 1983; Soderholm 1984). Water sorption also reduces the strength of matrix material (Calais and Soderholm 1988) which reduces the tensile strength of the composite (Soderholm 1985; Soderholm and Roberts 1990) and this may explain the relatively high frequency of bulk fractures in composite restorations (Moffa et al. 1984).

Hydrolysis is an important deteriorative process in modern polymer composites as many commercial materials incorporate hydrolysable linkages (Hino and Arai 1989; Fuji and Arai 1990). Hosoda et al. (1987a, 1987b) looked at the structural changes occurring at the surface of composite filling materials under alkaline conditions. After seven days immersion in NaOH at 60°C they observed swelling of the matrix, crack formation and small gaps between filler and matrix, presumably the result of hydrolysis.

These structural changes should be reflected by changes in tensile strength, hardness and wear resistance which measure the material's ability to resist plastic flow (Boyer 1987). Changes in these properties are often parallel and as hardness testing is simple, standardised and relatively non destructive it is the most commonly used indicator of wear performance

(Boyer 1987). McKinney and Wu (1985) showed a significant reduction in Knoop hardness and enhanced in vitro wear rates of composite filling resins after seven days immersion at 37°C in various food simulating liquids and water. However, another study (Harrison and Draughan 1976) has shown little or no relationship between hardness and abrasive wear of composite filling resins. Similarly, studies of composite inlay resins have reported considerably different Knoop hardness numbers for the same material; 50 (Inoue et al. 1989), 65 (Hosoda et al. 1989), 68 (Takahashi et al. 1989) for Isosit, 68 (Inoue et al. 1989), 100 (Hosoda et al. 1989), 102 (Takahashi et al. 1989) for Brilliant and 104 (Inoue et al. 1989), 131 (Hosoda et al. 1989), 167 (Takahashi et al. 1989) for Clearfil.

One explanation for these apparent discrepancies is the difficulty in measuring the residual indentation because of the translucency of the material, distortion due to elastic recovery and sensitivity to surface structure and surface preparation (Boyer 1987). Many of these limitations will be overcome if hardness is assessed by continuously recording indentation depth as a function of load which will provide information on the complex relationship between the plasticity and elasticity of the material (Loubet et al. 1986).

CHAPTER TWO - PURPOSE AND SCOPE OF THE INVESTIGATION

The dental precision casting technique controls the accuracy of the restorations by compensating the shrinkage of molten gold alloys (Section 1.3). The accuracy of these restorations has been assessed by a method in which a tapered metal die is used (Section 1.4). However, assessment of dimensional accuracy has neglected to include the impression taking step and this must be investigated. The dimensional stability of gold and ceramic inlays is of little concern because they absorb a minimal amount of water and have coefficients of thermal expansion similar to tooth structure (Section 1.7).

Composite inlay making has several advantages over the conventional composite filling technique. These are the possibility of compensating the polymerisation shrinkage during the fabrication process as well as the improvement of physical properties due to more complete polymerisation (Sections 1.2). However, unlike gold and ceramic inlays these materials are expected to be subject to considerable dimensional changes after cementation, although their thermal expansivity is unknown as this type of material is relatively new.

In this study the magnitude of dimensional changes occurring during impression taking and fabrication of composite inlays is first examined in order to establish their dimensional accuracy. The traditional metal die method (Section 1.4) is utilised for this purpose. This method is then expanded for the assessment of dimensional stability which is influenced by the change in temperature and the absorption of water (Section 1.8). The relative magnitude and importance of both dimensional accuracy

and dimensional stability can then be discussed in comparison to gold and ceramic restorations.

Water sorption may be detrimental to the structural integrity of composite inlay materials which may lead to long term dimensional instability associated with breakdown of these restorations (Section 1.8). Conflicting experimental data has limited the ability of conventional hardness testing to detect these early structural changes (Section 1.8). Therefore, an attempt is finally made to develop a new method of testing hardness to provide a reproducible laboratory technique to evaluate early structural changes and this is applied to inlay samples soaked in water.

CHAPTER THREE - MATERIALS AND METHODS

PART I - MATERIALS

3.1 Materials

The three composite inlay materials used in the present study were either hydrothermal or light curing type materials.

The hydrothermal curing type was a urethane dimethacrylate based resin, SR ISOSIT Inlay/Onlay¹. The material (hereafter, Isosit), was a two component system, comprising of a paste and a two part liquid. The composition of Isosit, as provided by the manufacturer, is shown below.

Composition of Isosit Inlay/Onlay (Ivoclar 1988b)

Paste		mass
Urethane Dimethacrylate		19 - 20 %
Aliphatic Dimethacrylate		4 %
Lanthanidefluoride (x-ray contrast)		20 - 21 %
Silanised Silicium Dioxide		55 %
Catalysts, Stabilizers, Pigments		2 %
N Liquid		
Diethyl Ketone		90 %
Dibenzoyl Peroxide		10 %
Cross Linking Paste		
Diurethane Dimethacrylate		84 %
1,4 Butanediol Dimethacrylate		4 %
Fumed Silica		12 %

1 Ivoclar, Schaan, Liechtenstein.

The liquid was prepared by mixing two components, N-Liquid and cross linking paste. The mixture (N-Fluid) was prepared daily as required by mixing 4 drops of the liquid with 1 gradation of the paste in a Dappen's dish which was then left covered for 10 minutes before use. The N-Fluid was stirred prior to each application.

The light curing materials studied were Brilliant¹ and Clearfil Composite Resin Inlay² (hereafter, Clearfil). Both were a one component paste system dispensed in a syringe. The composition of Brilliant, as provided by the manufacturer, is shown below.

Composition of Brilliant (Source: Coltene)

Components	mass
Bis GMA	9 %
Ethylmethacrylate	9 %
TEGDMA	5 %
Barium aluminum borosilicate (filler)	77 %

The composition of Clearfil was not provided by the manufacturer, except that it was a highly filled (86.5 percent) hybrid composite. The manufacturers of these light curing materials recommended post curing with either simultaneous light and heat (Brilliant) or heat (Clearfil).

The materials were all stored in a refrigerator at $4\pm 2^{\circ}\text{C}$ and were left under ambient conditions, $20\pm 1^{\circ}\text{C}$ and 50 ± 5 % relative humidity for 30 minutes prior to mixing.

1 Coltene AG, Altstatten, Switzerland.

2 Kuraray, Tokyo, Japan.

3.2 Measurement of Filler Content

The filler content of the three materials was determined by means of simultaneous differential thermal analysis-thermogravimetry (DTA-TG)¹.

Paste dispensed from the container or syringe was placed in a platinum pan and heated to 1000°C at a rate of 10°C/minute. The amount of material used for each analysis was 20±1 mg. In the case of the two component system (Isosit), paste samples were analysed with and without N-fluid. The samples with N-fluid were only heated to 300°C in order to determine the polymerisation temperature.

Mass decreases at various temperatures were obtained from the TG curve and peak temperature was determined for endothermic and exothermic reactions appearing in the DTA curve. Three samples were analysed for each material. Ignited-alumina was used as reference material and the thermobalance was calibrated with a standard 20±0.1 mg mass.

3.3 Processing Conditions

An autoclave (Ivomat) supplied for the processing of Isosit is shown in Figure 3.1. The recommended curing conditions were 120°C, 6×10⁵ Pa for 10 minutes. Distilled water (500 mL) was placed in the pressure chamber for each processing.

1 Thermoflex, Rigaku Denki Ltd, Tokyo, Japan.

Brilliant specimens were cured by a light probe¹ for 3 minutes and then exposed to simultaneous light/heat in an oven² heated by a halogen lamp (Figure 3.2) for approximately 8 minutes.

A light box³ was used for the curing of Clearfil. The irradiation time (3 minutes) was determined by running a preliminary experiment based on the manufacturer's recommendation of "around 3 minutes, dependent on the light box used". This was followed by a heating in an oven maintained at 100°C for 15 minutes.

The heating conditions of the three materials were monitored by measuring the temperature during heat curing. A thermocouple (Type K) was connected to a chart recorder⁴ via a linear thermocouple module⁵. Calibration of the module and recorder was carried out by supplying various emfs through the module using a multiple function indicator/calibrator⁶ with a resolution of 0.001 mV or 0.1°C.

In the case of Isosit the thermocouple was inserted through the lid of the processing chamber (Figure 3.1) and two other processing temperatures (100 and 110°C) were also monitored for the recommended processing time (10 minutes). The pressure within the Ivomat was also studied by inserting a pressure gauge through the lid of the processing chamber (Figure 3.1). In the case of Brilliant and Clearfil the thermocouple was inserted into the DI 500 and the oven. At least three measurements were made for each processing method.

1 Heliomat, Vivadent, Schaan, Liechtenstein.

2 DI 500, Coltene AG, Altstätten, Switzerland.

3 Astron Dental, Wheeling, U.S.A.

4 Yew type, Yokogawa Electric Works, Tokyo, Japan.

5 Action Instruments, San Diego, U.S.A.

6 Micromite II, Thermoelectric Co., Saddle Brook, U.S.A.

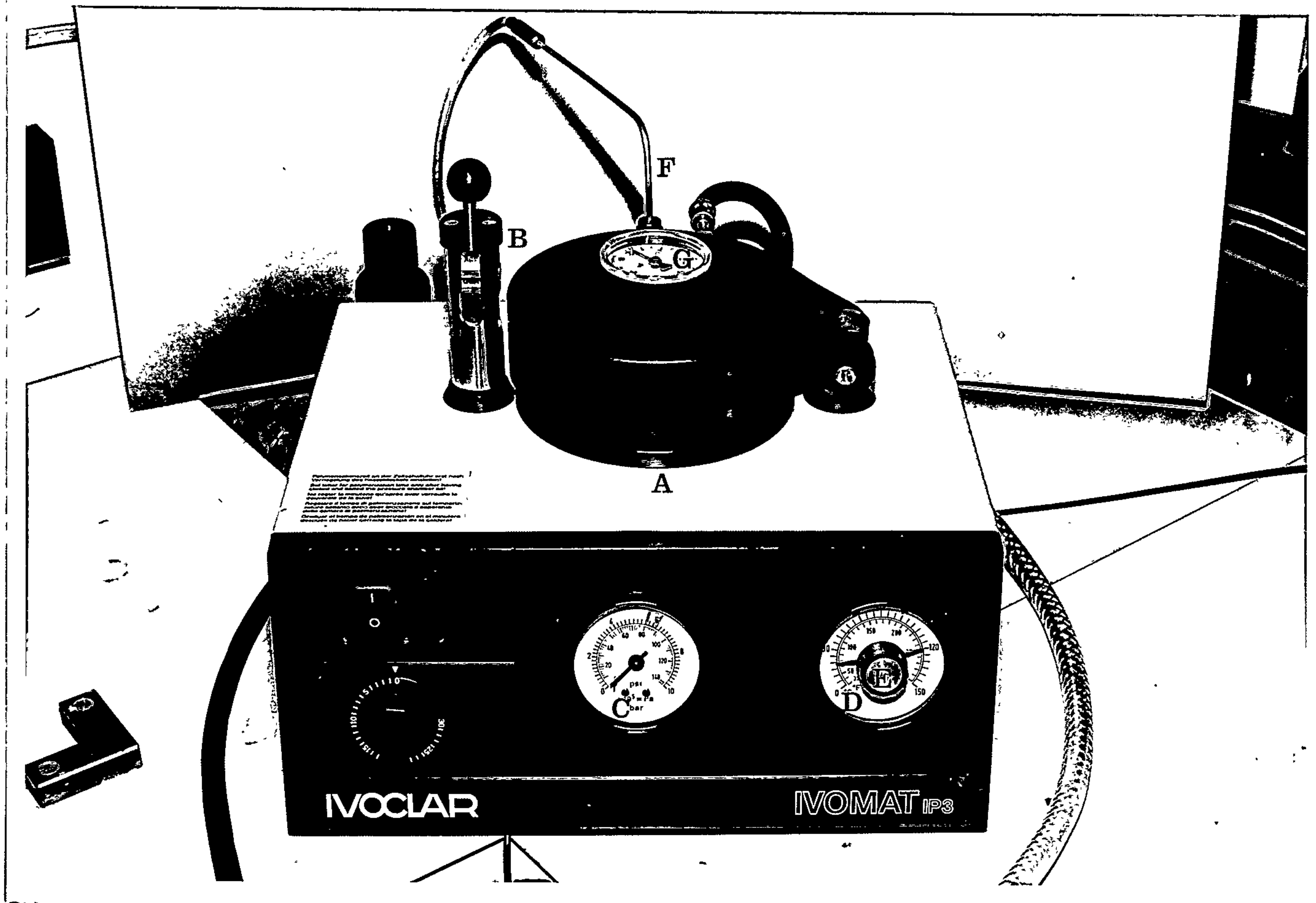


Figure 3.1 : IVOMAT

The Ivomat supplied for the processing of Isosit consisted of a processing chamber (A) pressurised by compressed air and heated by an internal element. A clamping lid (B) sealed the chamber. The temperature and pressure in the chamber is shown on gauges (C) and (D) respectively. The desired processing temperature was selected by adjusting the thermostat (E). A thermocouple (F) and a pressure gauge (G) were inserted through the clamping lid of the processing chamber to record temperature and pressure.

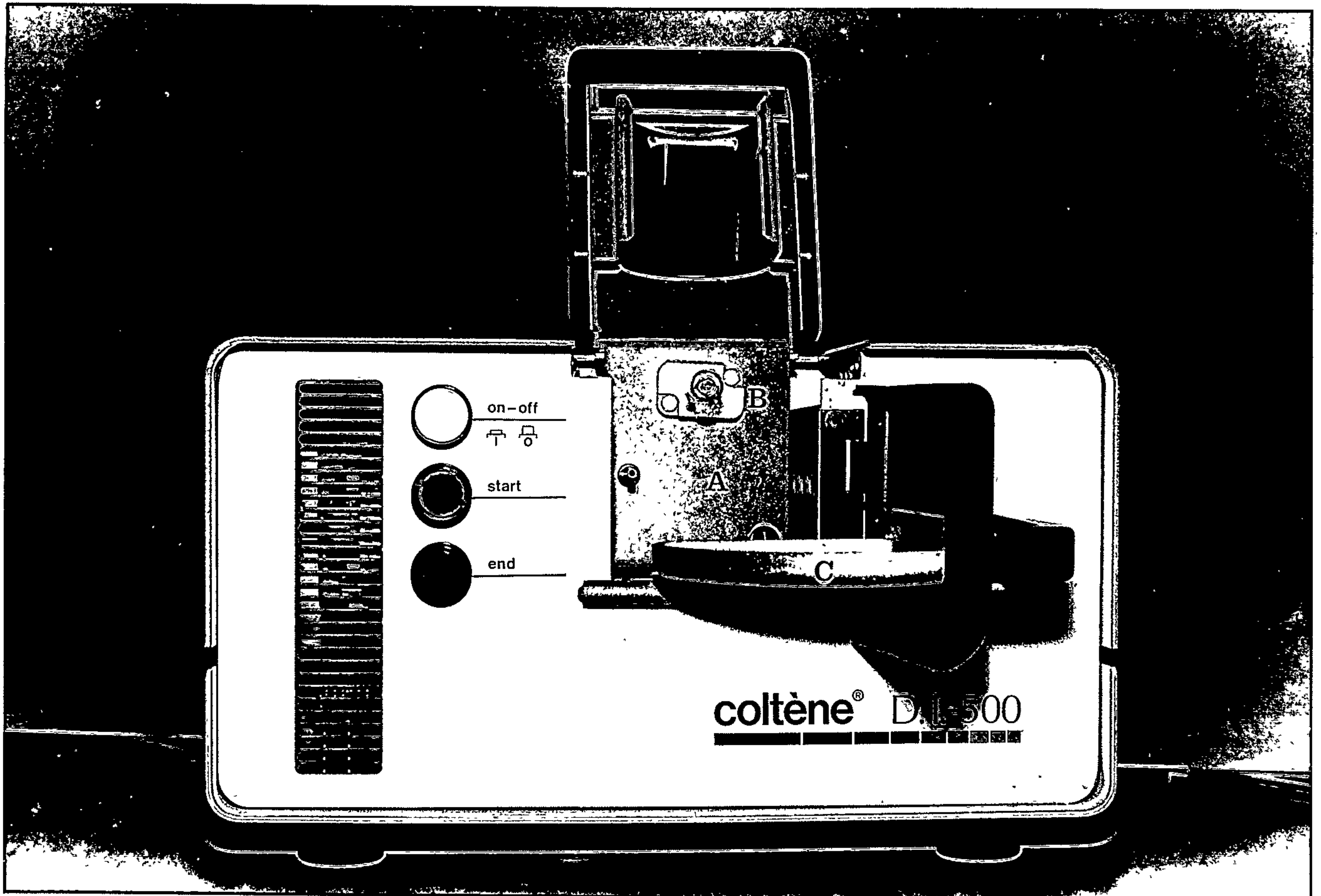


Figure 3.2 : DI 500

The DI 500 supplied for processing Brilliant consists of a heating chamber (A) containing a halogen lamp (B). The specimen tray (C) is attached to the door.

PART II - DIMENSIONAL ACCURACY

3.4 Metal Die Assembly and Stone Die

A stainless steel die representing a Class I inlay cavity was designed for measurement of dimensional accuracy and stability of inlays. It had diameters of 7.2 mm and 8.0 mm and was 4 mm high, providing a taper of 1 in 5 (Figure 3.3). Four points (90° intervals) were scribed on the outer circumference of the die.

To simulate a practical inlay fabrication process the metal die assembly was kept in an incubator, whose temperature and relative humidity were controlled at $36\pm1^{\circ}\text{C}$ and 100%, for at least 30 minutes and impressions of the die assembly were taken using an addition cured silicone impression material, Hydrophillic Exaflex (regular viscosity)¹. Equal masses (10 g) of catalyst and base and retarder (3 drops) were mixed with a stainless steel spatula on a glass slab for 30 seconds.

A perforated brass custom impression tray was coated with adhesive and left to dry for at least 5 minutes. The impression mix was loaded into both a syringe and the tray. The die was injected with impression material and the tray was secured to a loading rod with a pin (Figure 3.4). Temperature changes occurring in the impression were recorded with a thermocouple (Type K) inserted into the impression (Figure 3.5). A 1 kg force was applied to the loading rod to seat the impression. Impressions were left for the time recommended by the manufacturer (6 minutes) from the start of mixing before

¹ Type I, medium viscosity, GC, Tokyo, Japan.

unseating. The lever was then attached to the jig and the impression was unseated from the die with a short sharp movement and cooled to ambient temperature.

A dental stone¹, which had been homogenised in a mixer² for 10 minutes and then stored in a sealed container at ambient conditions, was mixed using the water/powder ratio (0.26) recommended by the manufacturer. The mix was prepared following the specifications of Australian standard 1616; powder (60 g) was sifted into distilled water (15.6 mL) in a rubber mixing bowl over a 15 second period. The powder was left for 15 seconds prior to mixing by hand with a plastic spatula for 30 seconds at a rate of 3 turns per second.

The stone mix was left at ambient conditions for 60 minutes from the start of mixing and then removed from the impression. The excess stone on the base of the die was trimmed on a model trimmer. The die was weighed and then left at ambient conditions until a constant mass was achieved. Following removal of the stone die from the impression, a second die was poured in the same manner as this was recommended for the fabrication of Isosit inlays.

Isosit specimens were prepared in the stone die. The internal surface of the die was coated with the N-Fluid mixture using a small brush and then a roll of paste was placed in the die in one increment and pressed into place using a stainless steel instrument. The excess was removed and then the surface was coated with the N-Fluid mixture.

Separately, specimens were made from dies prepared from an

1 Plastone, Low Expansion Type, GC, Tokyo, Japan.

2 Rotomixer, Forster Equipment Company, Wheelstone, England.

impression taken at room temperature. In this case the impressions were left on the metal die for 10 minutes from the start of mixing. These specimens served as control to investigate the effect of dimensional changes occurring in the impression subjected to the temperature change from 36 to 20°C.

The effect of temperature and pressure used during processing of Isosit on the die stone was also studied by measuring the compressive strength of cylindrical specimens of Plastone.

Stone mixes were prepared in the same manner described previously and vibrated into a cylindrical Teflon mould, 15 mm in diameter and 25 mm in height, using a table vibrator. At the loss of gloss from the mix, a glass plate was applied to the top of the mould to remove excess material and provide a flat surface on the specimen. This was left in place for 50 minutes from the start of mixing and then the mould opened. At 60 minutes from the start of mixing the specimens were weighed. Six specimens were prepared from each mix.

Strengths of dry and wet stone specimens were compared with those of specimens immersed in water (500 mL) in the Ivomat and subjected to various temperatures at 6×10^5 Pa for 10 minutes.

3.5 Specimens for the Measurement of Polymerisation Shrinkage

Specimens for this measurement were fabricated in the metal die using the three inlay materials. Isosit specimens were prepared in the same manner as described in Section 3.4. Brilliant and Clearfil pastes were dispensed into the die in one increment and then pressed into place using a stainless steel instrument.

The die was secured in a clamp (Figure 3.3) which located the die in a loading device (Figure 3.6). A Teflon insert was placed on the die and a static load of 500 g was applied for 60 seconds.

The specimens were processed according to the manufacturer's recommendations (Section 3.3). Isosit specimens were processed in the die with the Teflon insert in place. This was removed to facilitate light curing when Brilliant and Clearfil specimens were prepared (Section 3.3). After initial light curing the Brilliant and Clearfil specimens were removed from the clamp and base and the bottoms of the specimens were irradiated. Brilliant specimens were removed from the die prior to the subsequent light/heat curing. The base and clamp were reassembled prior to the post heat curing of Clearfil specimens. At least six specimens were prepared for each material.

Immediately after processing, specimens were trimmed by hand using wet or dry sandpaper (grit 200) to remove the flash which had formed at the top. They were then weighed, placed in an open glass tube and kept in a desiccator containing silica gel at ambient temperature. Changes in mass were recorded at 24 hour intervals for 5 days.

3.6 Measurement of Dimensional Accuracy

A preliminary experiment was conducted on Isosit specimens to establish a seating load for reproducible measurement of dimensional accuracy. Six specimens, which were processed under various processing conditions (Section 3.3) and kept in a desiccator, were subjected to eight loading conditions

shown below.

LOAD	300 g	500 g	1 kg	2 kg
TIME	30s 60s	30s 60s	30s 60s	30s 60s

The metal die used in the preparation of specimens was placed in a recess of a loading device (Figure 3.7) centred beneath a loading pin. Specimens were weighed and then placed in the die so that a mark on the specimen surface aligned with a mark on the die surface. This ensured that repeated measurements were taken at the same points on each specimen. The load was applied to the specimens within 90 seconds of their removal from the desiccator.

The specimens were loaded for 30 seconds starting with the lightest load and progressing to the heavier loads. At least 10 minutes recovery time in the desiccator was given before the next successive loadings and the same measurement was repeated once for each specimen. The specimens were kept in the desiccator for at least 2 days before 60 second loading experiments were undertaken in the same manner as for the 30 second loading.

The specimens, seated in the stainless steel die, were all protruding from the bottom of the die due to shrinkage which occurred during polymerisation. The seated specimens were placed on the stage of a travelling microscope¹ and the protrusion of the specimen from the die was measured at 90° intervals (Figure 3.8). A formula for the calculation of the percentage linear polymerisation shrinkage or diametral accuracy of the specimen is derived from the geometric relationship between the convergence of the die and the protrusion of the specimen. The derivation of this formula is shown in Appendix A-1 and A-2. In simplified terms this

¹ Beck Instruments, London, England.

formula is;

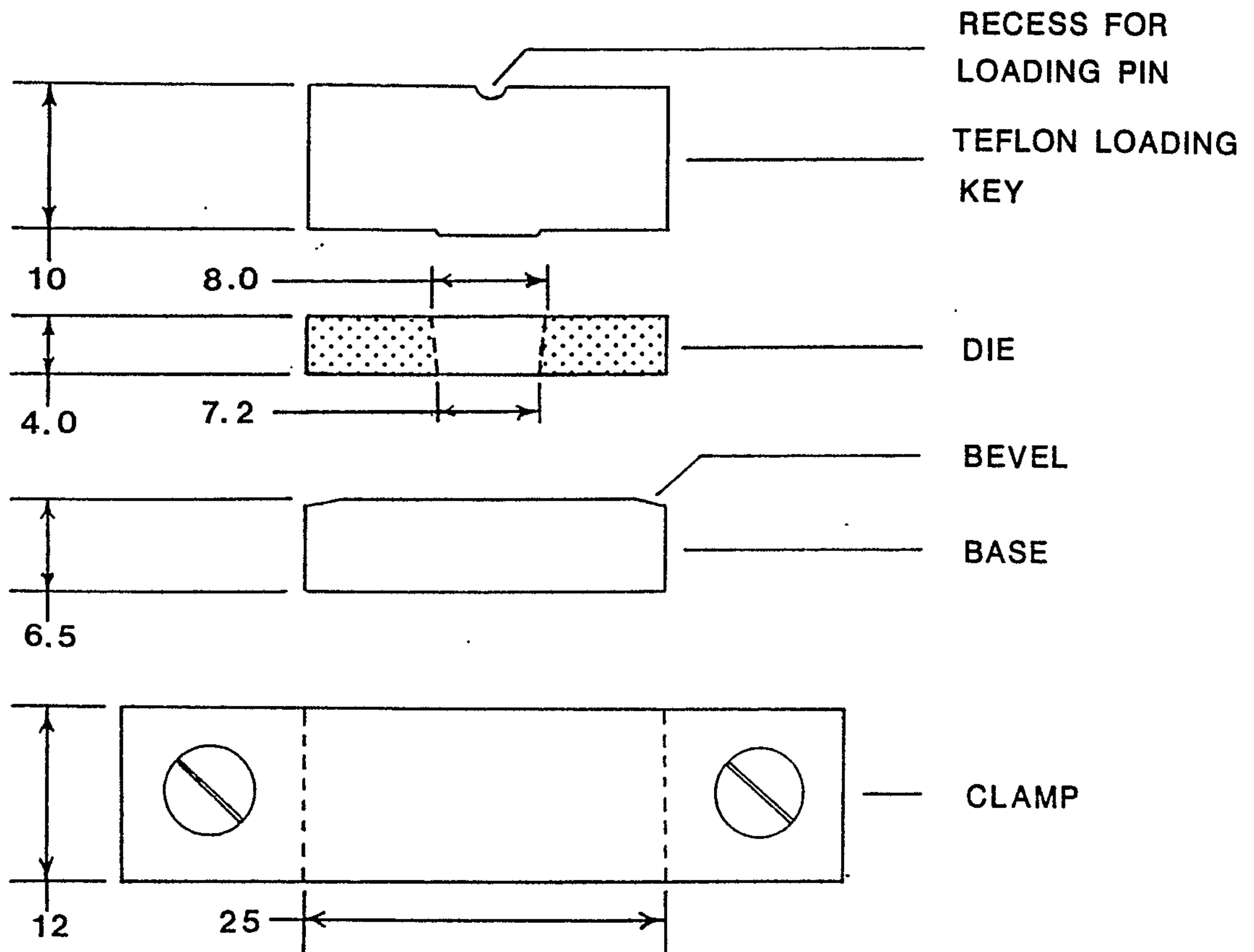
$$\begin{array}{lcl} \% \text{ LINEAR} & \text{PROTRUSION} & 100 \\ \text{DIMENSIONAL} & = \text{-----} \times \text{-----} & \\ \text{CHANGE} & \text{CONVERGENCE} & \text{CAVITY DIAMETER AT THE BASE} \end{array}$$

The results of the preliminary experiments determined the most appropriate load to be 2 kg for 60 seconds (Appendix B) and this condition was used for the rest of the present study. For the metal die kept at mouth temperature and humidity (Section 3.4), an adjustment was made to account for the dimensional change of the stainless steel die using the coefficient of thermal expansion.

A method which employed a transducer¹ was also investigated for the measurement of protrusion. For this six Isosit specimens processed according to the manufacturer's recommendations were used and they were seated in the metal die under a load of 2 kg for 60 seconds. The die was placed on the stage of the transducer assembly (Figure 3.9). A recording pin which incorporated the transducer was lowered on the die and a record of the die thickness was first taken. This was followed by a recording of the height of the specimen protruding upwards. The measurement was repeated once providing two measurements of protrusion for each specimen. Calibration of the transducer was undertaken using standard gauge blocks² of 1 mm and 4 mm. Analysis of the preliminary experiments showed no significant difference between the methods and the transducer method exhibited several advantages over the microscope method. Therefore, the microscope method was replaced by the transducer method for the Brilliant and Clearfil specimens.

1 Linear variable differential transformer, LVDT, 200DC-D, Schaevitz, Pennsauken, New Jersey, U.S.A.

2 Mitsutoyo, Tokyo, Japan.



ELEVATION (DISSEMBLED)

FIGURE 3.3 : DIE ASSEMBLY

The die and a stainless steel base were held by a brass clamp which prevented flash formation at the bottom of the inlay. The base had a bevelled edge to allow its separation from the upper member of the die after an inlay was processed.

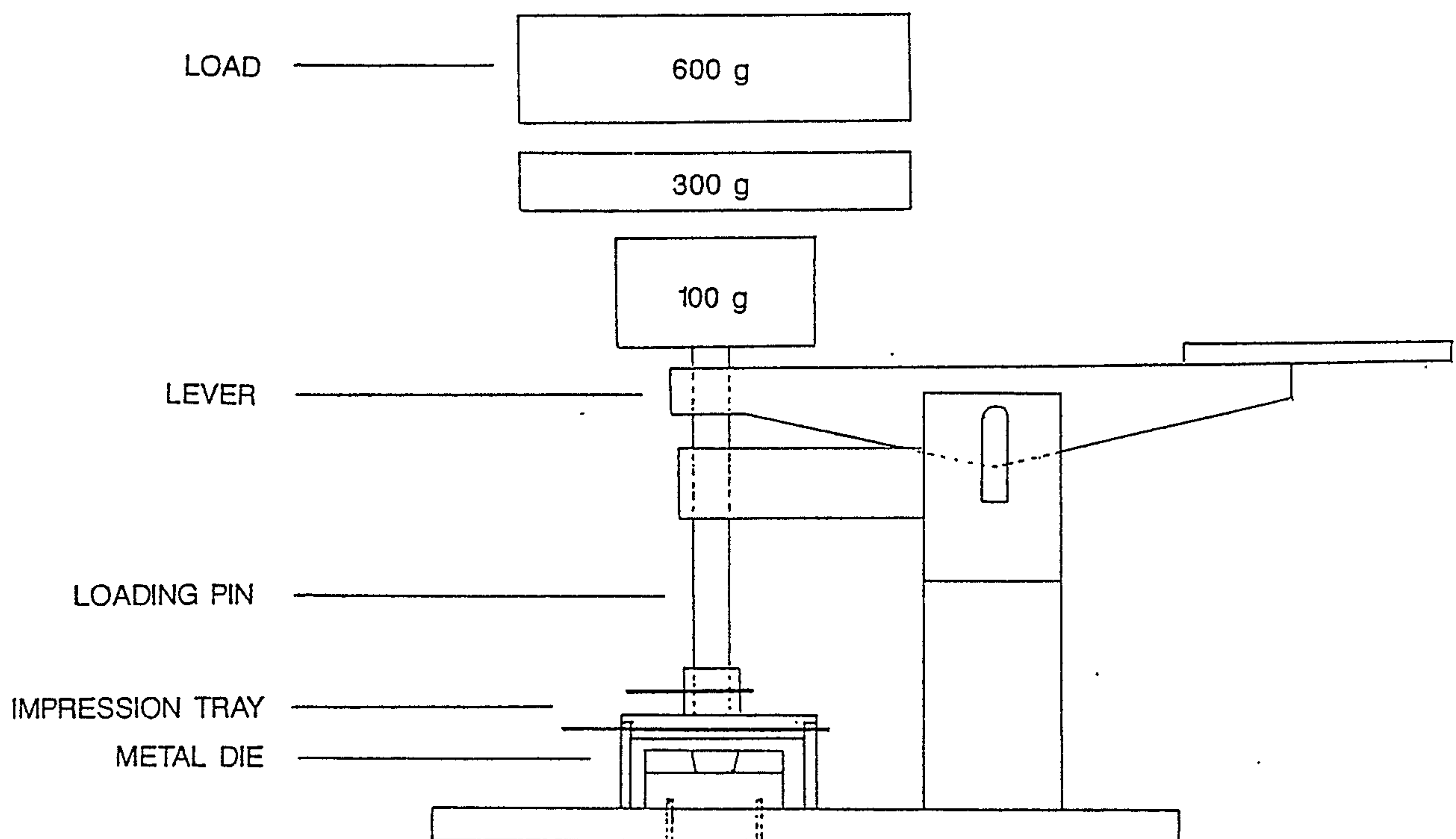


Figure 3.4 : IMPRESSION TRAY AND LOADING DEVICE

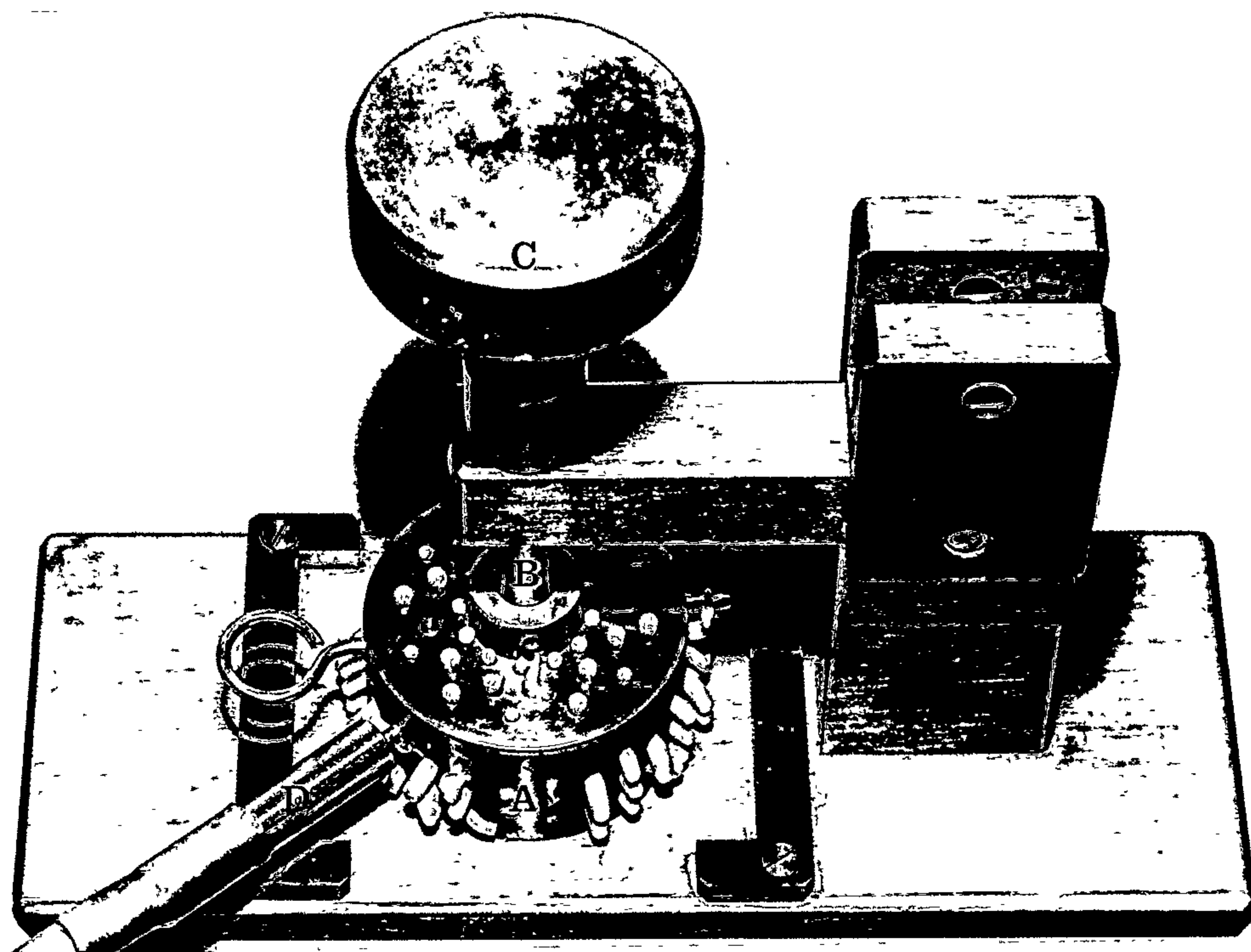


Figure 3.5 : IMPRESSION WITH THERMOCOUPLE IN PLACE

(A) impression tray (B) loading rod (C) load (D) thermocouple

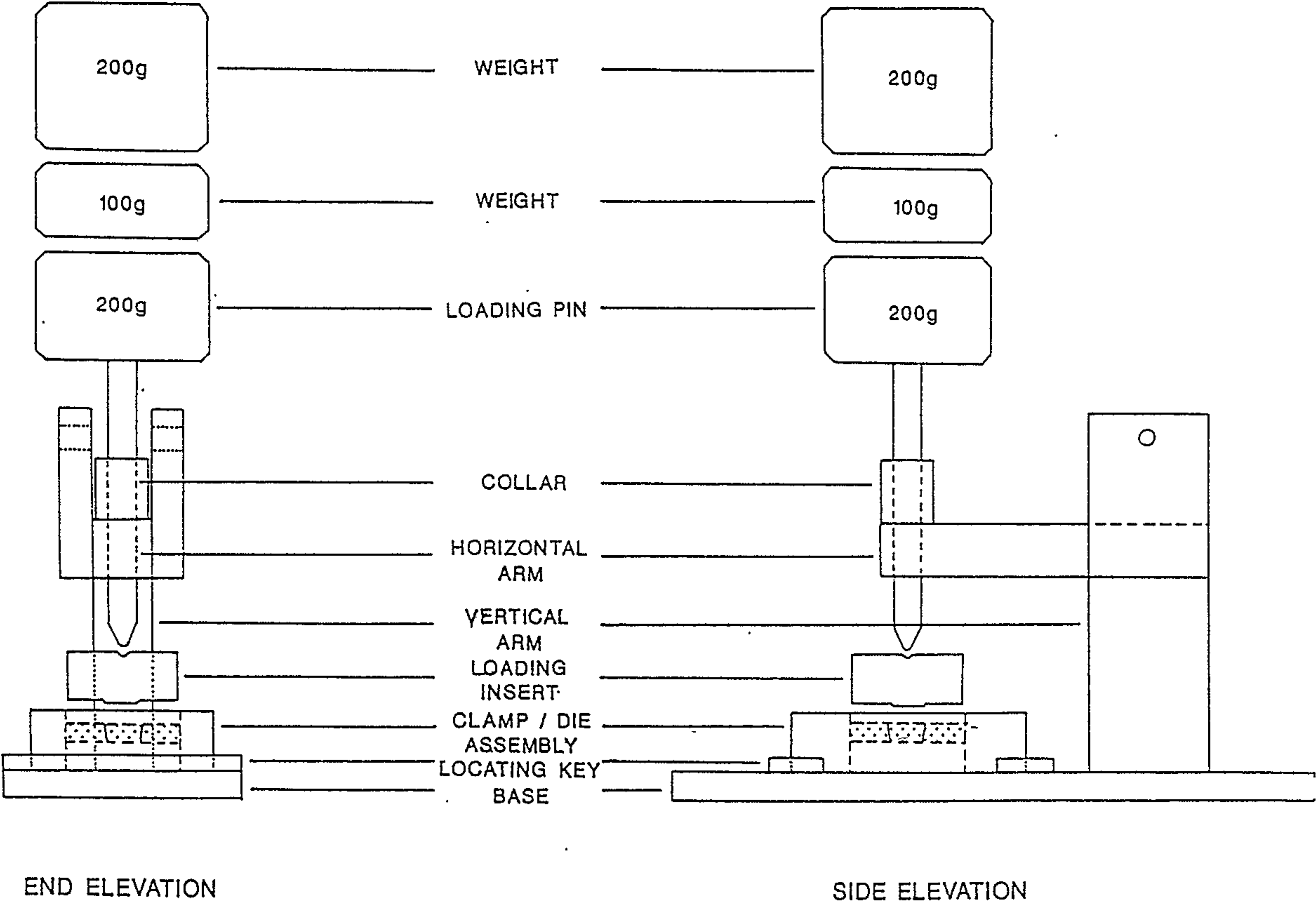


FIGURE 3.6 : LOADING DEVICE FOR SPECIMEN PREPARATION

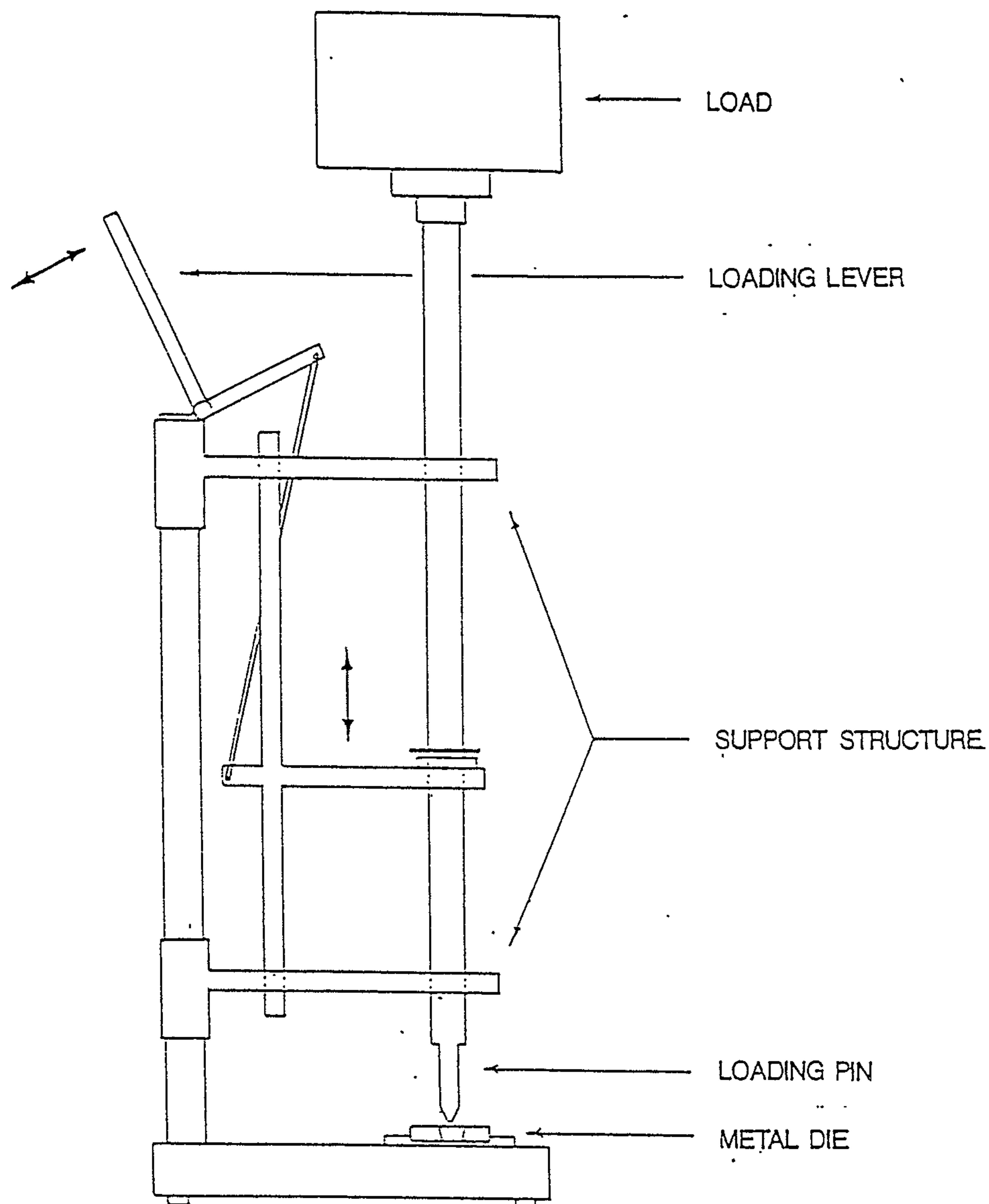


FIGURE 3.7 : SHRINKAGE MEASUREMENT LOADING DEVICE

The loading device consisted of a loading pin supporting a bucket containing lead shot. The load was applied to the specimens by a lever mechanism and varied by placing or removing lead shot.

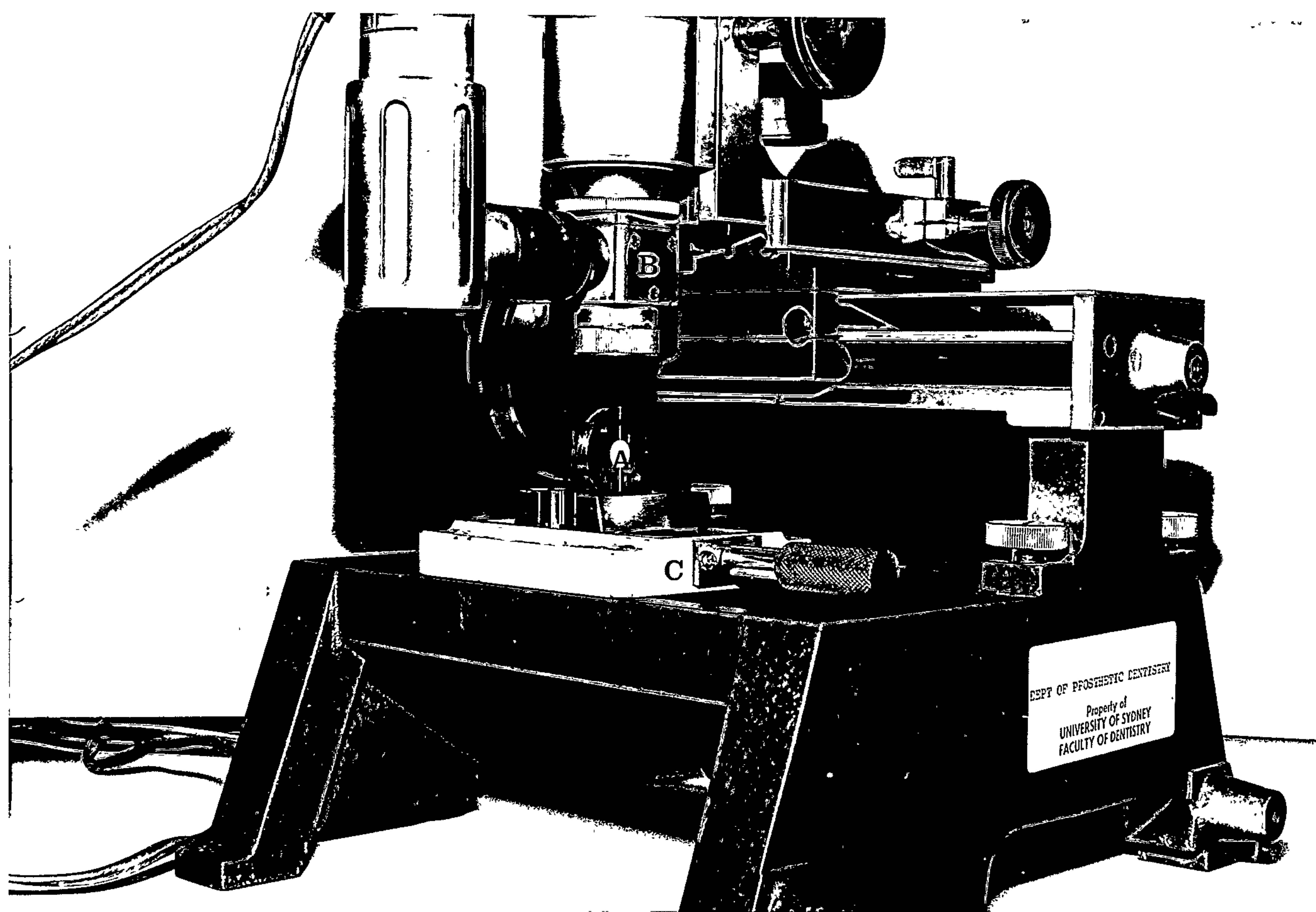


Figure 3.8 : MICROSCOPE MEASUREMENT OF PROTRUSION

The specimen protruding from the stainless steel die (A) was positioned under the travelling microscope (B) by a clamp (C). The clamp was opened and the die rotated through 90° intervals so that four measurements of protrusion could be made.

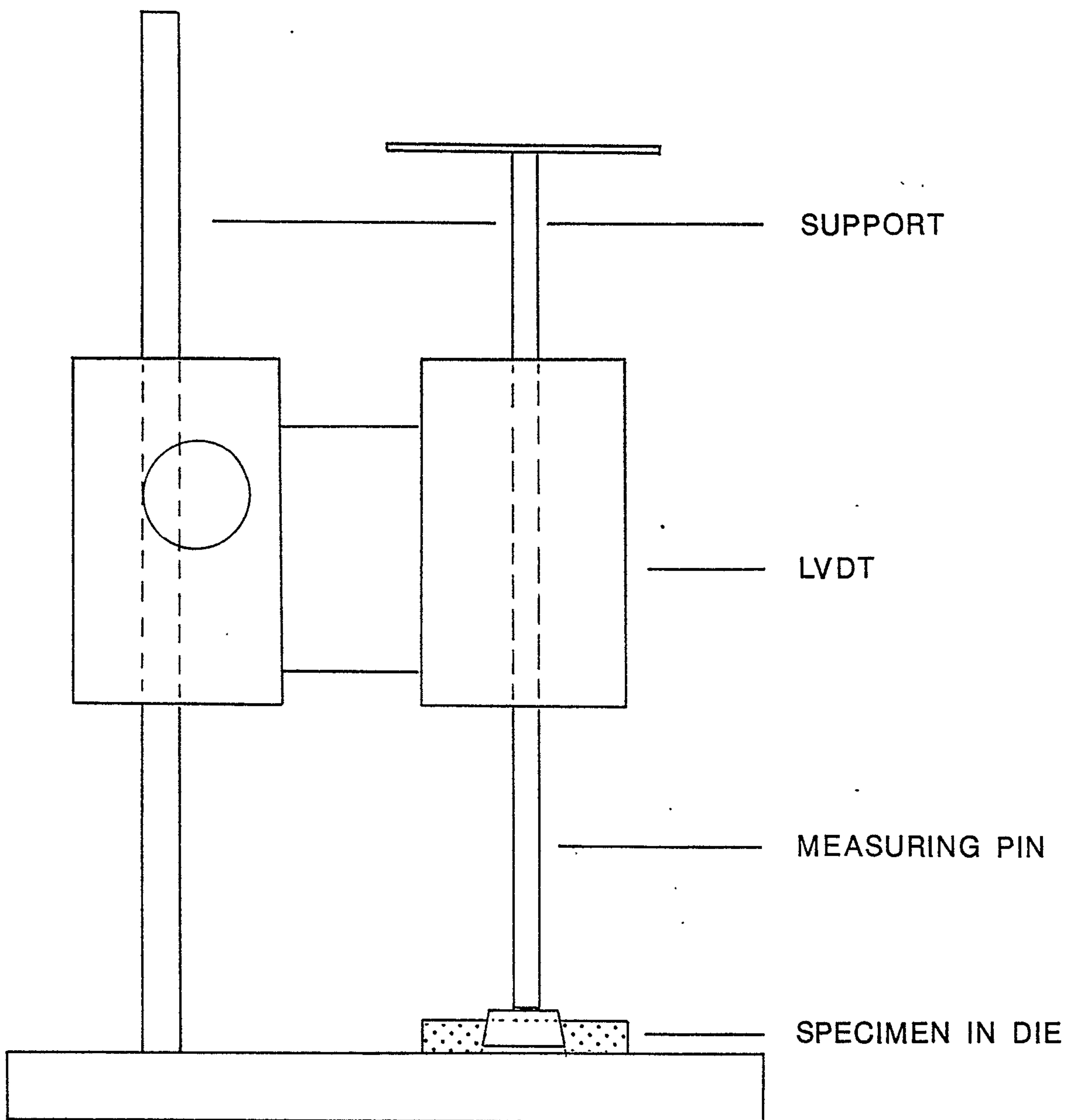


Figure 3.9 : TRANSDUCER MEASUREMENT OF PROTRUSION

The specimen seated in the die was placed on a stage beneath the measuring pin. The linear variable differential transformer (LVDT) was connected to a pen chart recorder.

PART III - DIMENSIONAL STABILITY

3.7 Effects of Water Sorption

Following the measurement of dimensional accuracy (Section 3.6), six specimens of each material were partially immersed in distilled water at 37°C. This was to eliminate the possibility of air entrapment by complete immersion. After 24 hours the specimens were removed, held against filter paper for 30 seconds to remove excess water, weighed¹ with an accuracy of 0.1 mg and then completely immersed in distilled water at 37°C. After seven days, 30 days and six months of immersion the specimens were weighed and then seated in the die. Protrusion of the specimen from the die was measured with the transducer method (Section 3.6) to assess the dimensional stability. The specimens were weighed again and immersed in water for at least another 10 minutes before repeating the measurements. This provided twelve measurements of protrusion for inlays of each material at each particular time.

3.8 Measurement of Coefficient of Thermal Expansion

The effect of thermal expansion on the dimensional stability of the inlays was assessed by measuring the coefficient of thermal expansion of the three materials. For this cylindrical specimens were prepared in a vented Teflon mould (Figure 3.10). The internal surface of the mould was coated with the liquid (N-Fluid) using a small brush. A roll of paste was

1. AE 163, Mettler, Greifensee, Switzerland.

placed in one increment using a stainless steel instrument and the surface was coated with the liquid. Brilliant or Clearfil paste was also placed into the mould in one increment and then pressed into place using a stainless steel instrument. A Teflon plug was inserted into the end of the mould and a static load of 500 g was applied to the specimens for 60 seconds before curing. Each curing was carried out within 5 minutes of spatulation of the paste.

Isosit specimens were cured with the plugs in place in the Ivomat. The base of the mould and the plugs were removed for both Brilliant and Clearfil specimens to facilitate light curing. Clearfil specimens were then placed in the oven. With Brilliant specimens the mould was opened and the central section of the specimen was cured for a further 60 seconds before it was removed from the mould and then cured in the DI 500.

After processing, the Isosit and Clearfil specimens were removed from the mould. The flash present on all specimens was trimmed with a scalpel and the ends were machined in a centre lathe¹ to ensure the samples were of accurate length with parallel ends. To remove oils originating from the machining process the specimens were washed in water containing detergent for 3 minutes using an ultrasonic cleaner and rinsed with distilled water. They were then dried to a constant mass prior to testing.

The test specimen and a cylindrical alumina reference specimen, each 12 ± 0.1 mm in length by 5 ± 0.1 mm in diameter, were placed on the detecting rods of a differential dilatometer, Thermomechanical Analyser (TMA)². A measuring load

1 Emco, Hallein, Austria.

2 Rigaku Denki Ltd, Tokyo, Japan.

of 10 g was applied to the samples by a counterweight on each detecting rod (Figure 3.11). At least three specimens of each material were heated at a rate of 5°C/minute up to a maximum temperature of 310°C. Isoit specimens processed at 100°C for 10 and 20 minutes were also included and at least three specimens were measured for each condition.

3.9 Hardness Testing Device

The hardness testing device developed is shown in Figure 3.12. It consisted of a Vickers diamond indenter mounted on a loading pin which was driven by a motor¹ at a speed of approximately 1 µm/second. A displacement transducer², mounted between the support arm and the loading pin, was calibrated by a micrometer³, with an accuracy of 0.02 µm, attached to the loading pin. Specimens were placed on a load cell⁴ mounted in a brass stage which was calibrated using weights of known mass. A representative calibration of the transducer and load cell is shown in Figure 3.13. This arrangement allowed continuous measurement of indentation depth and indenting forces which were recorded on an X-Y recorder⁵.

The new method developed was verified by measuring the

-
- 1 1.8° stepper motor, Model No. 332-082, R.S. Components Ltd. Perth, Western Australia.
 - 2 Linear variable differential transformer (LVDT), 200DC-D, Schaevitz, Pennsauken, New Jersey, U.S.A.
 - 3 Model No. 842135, Mitutoyo, Tokyo, Japan.
 - 4 LM-A, Kyowa Electronic Instruments, Tokyo, Japan.
 - 5 Type 3036, Yokogawa Hokushin Electric, Tokyo, Japan.

hardness of a standard metallic sample¹ of known hardness numbers, 769-780 for a load of 300 g. Three indentations were made on the sample with a maximum force of 450 ± 50 g.

Three specimens were prepared of each inlay material in the same manner as for polymerisation shrinkage measurement (Section 3.5). Immediately after processing the top surface of the specimens was trimmed and smoothed using wet or dry sandpaper (grit 200) and placed in distilled water for two hours at ambient conditions. Excess water was removed from the specimens in the manner described previously (Section 3.7) and they were weighed prior to testing. Specimens were placed on the load cell and a downward force was applied to the loading pin. The force was increased to a maximum of 800 ± 50 g and then the direction of the motor was reversed to measure the force-depth relationship during unloading. Recording ceased when the force on the indenter returned to zero. The diagonal length of the indentation which generally provides the Vickers hardness number was calculated from the depth recorded using the general formula derived from the geometry of the indenter tip (Figure 3.14).

Six hardness measurements were made on each of the sanded top and moulded bottom surfaces of the specimens with a maximum force of approximately 800 g. The depth of penetration at 500 and 800 gf upon loading and that at 20 gf upon unloading were read from the recording. Testing sites were spaced equally around the diameter of each specimen which was aligned to the front of the testing machine by a pencil mark.

The hardness measurements were repeated on the bottom surface of the specimens after seven days immersion in

1 Shimadzu, Tokyo, Japan.

distilled water at 37°C. Testing sites were chosen so as not to coincide with a previous indentation. The hardness of the three specimens that were immersed in distilled water for six months for the measurement of dimensional accuracy (Section 3.7) were also measured. Six evenly spaced indentations were made on the bottom surface of each specimen.

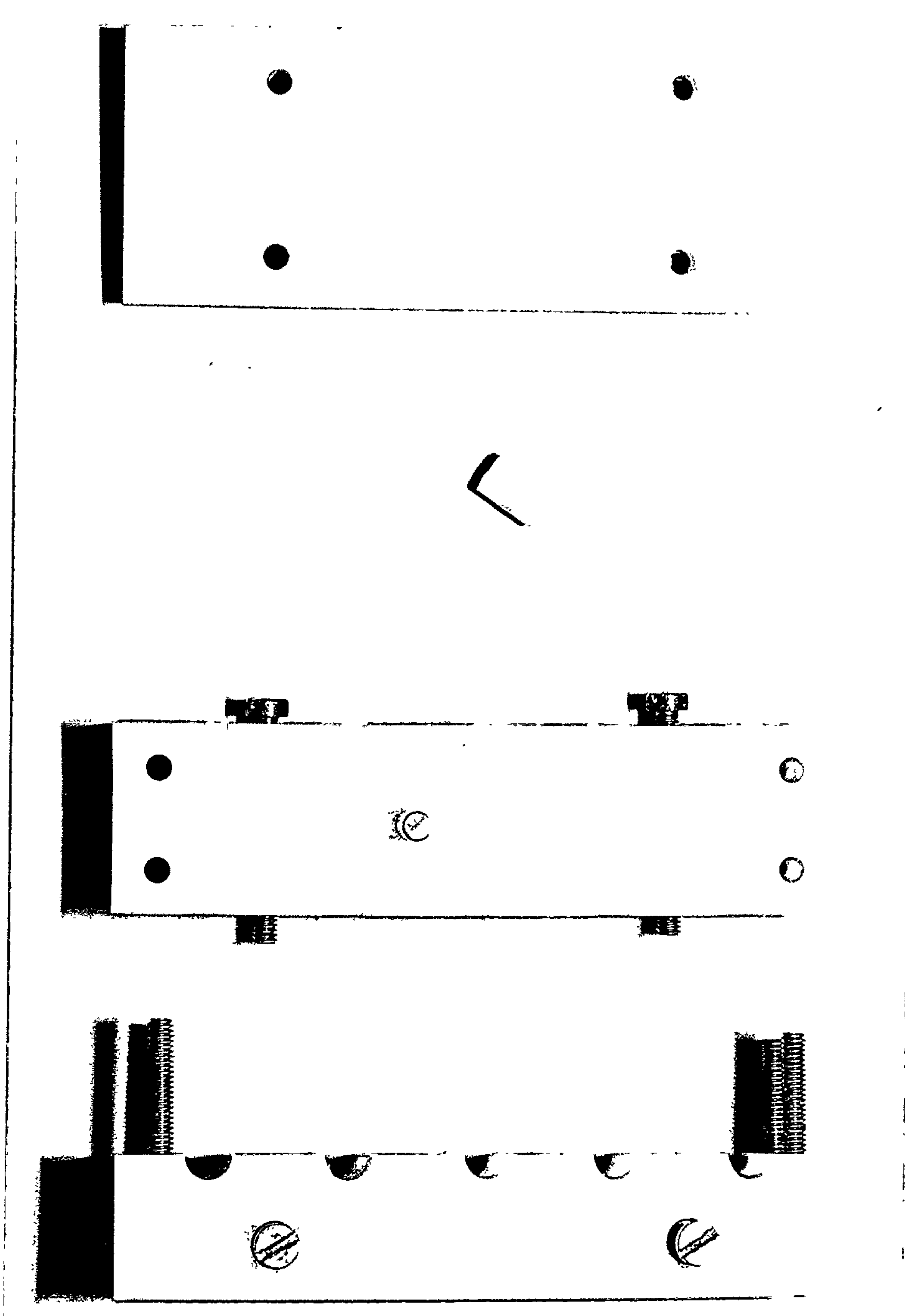


Figure 3.10 THE TEFLON MOULD FOR TMA SPECIMENS

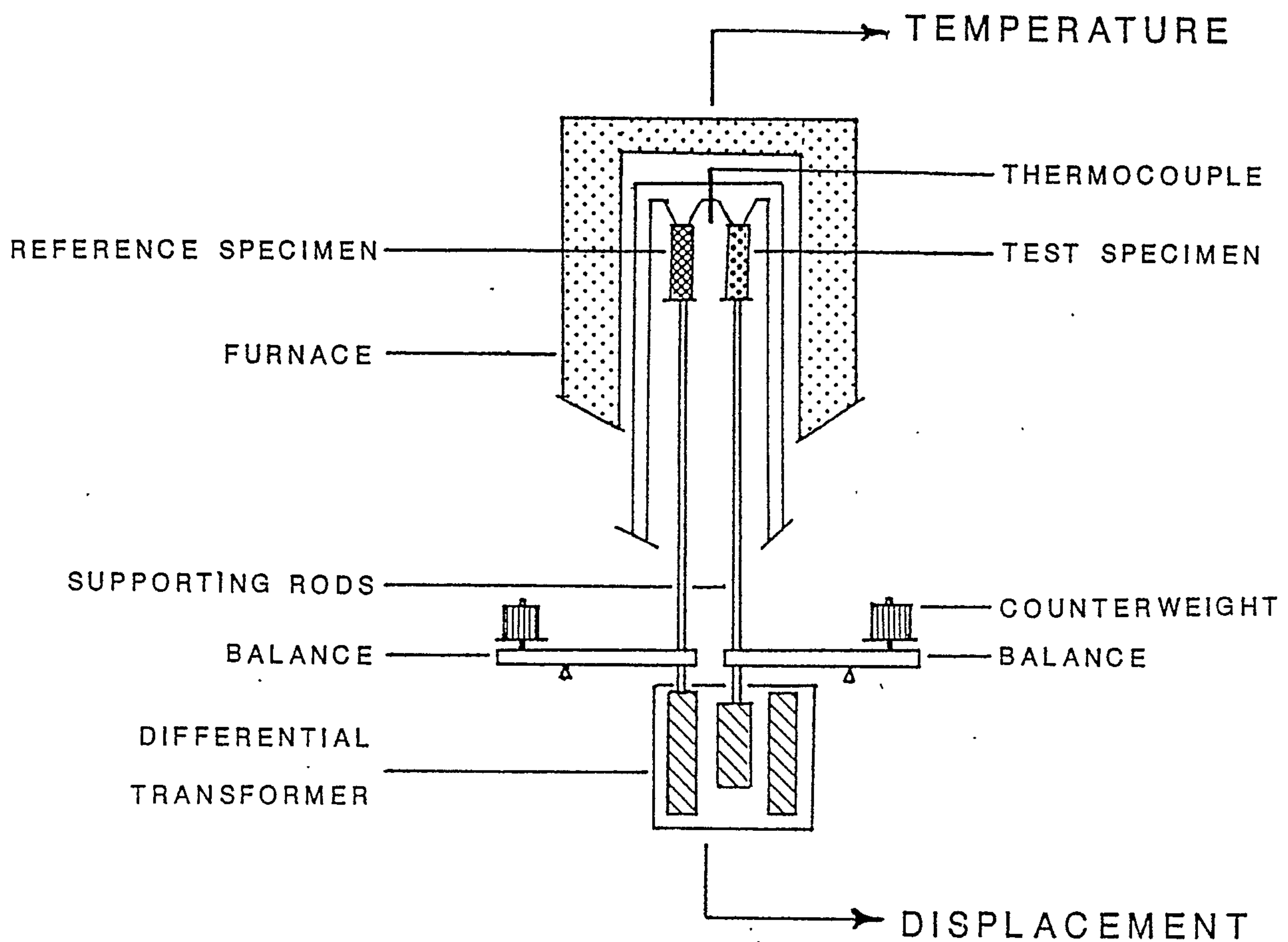


Figure 3.11 : DIFFERENTIAL DILATOMETER

The samples are placed on stages supported by the detecting rods contained within the supporting tube. The detecting rods are connected to the differential transformer, the rod supporting the reference sample to the coil and the rod supporting the reference sample to the core. As the samples are heated, the expansion will push down the respective detecting rods changing the relative positions of the core and coil of the differential transformer. This difference is transmitted to the recorder as an electric signal and presented as plots of expansion and temperature versus time.

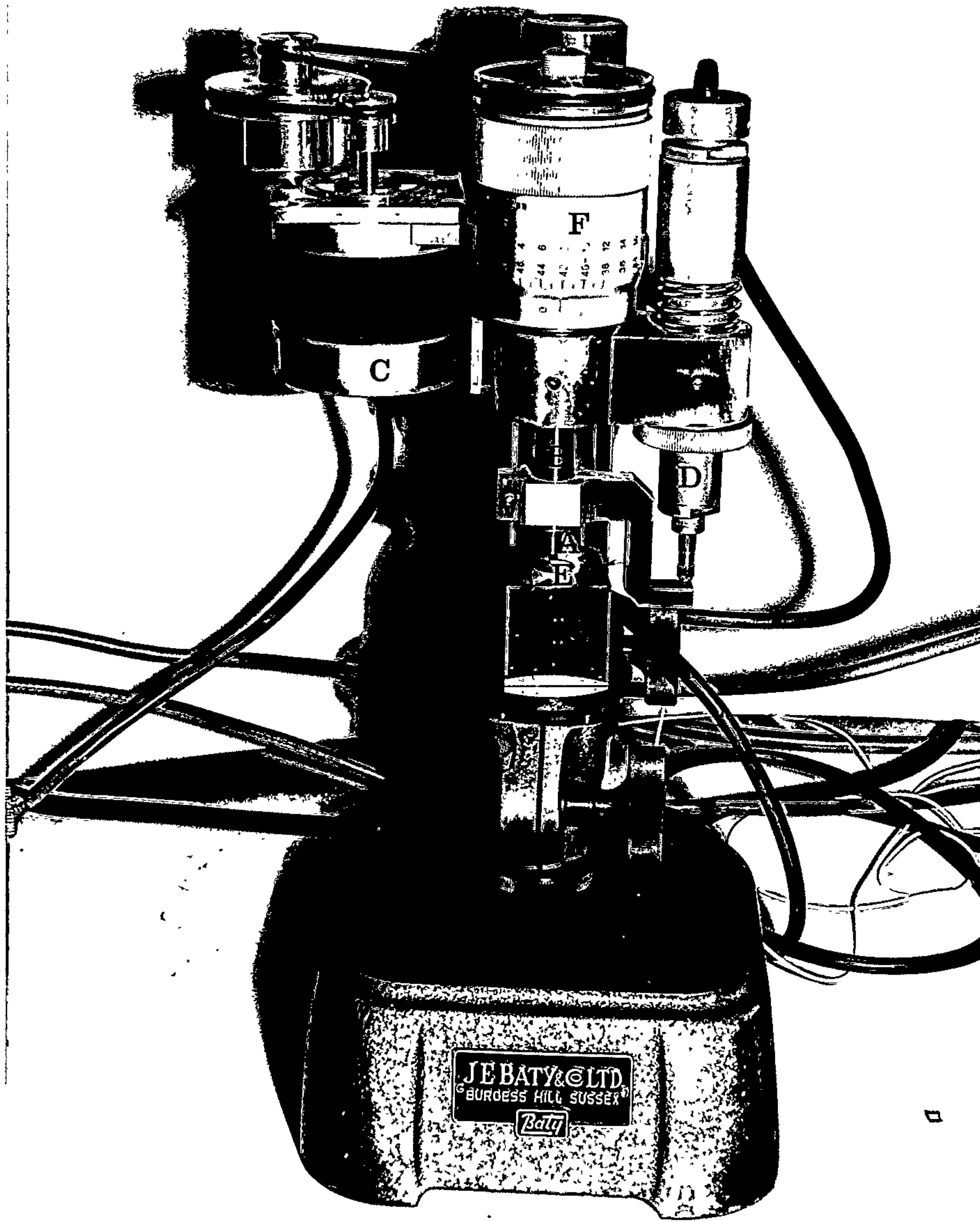


Figure 3.12 : HARDNESS TESTING MACHINE

(A) Vickers indenter (B) loading rod (C) motor
(D) displacement transducer (E) load cell
(F) micrometer.

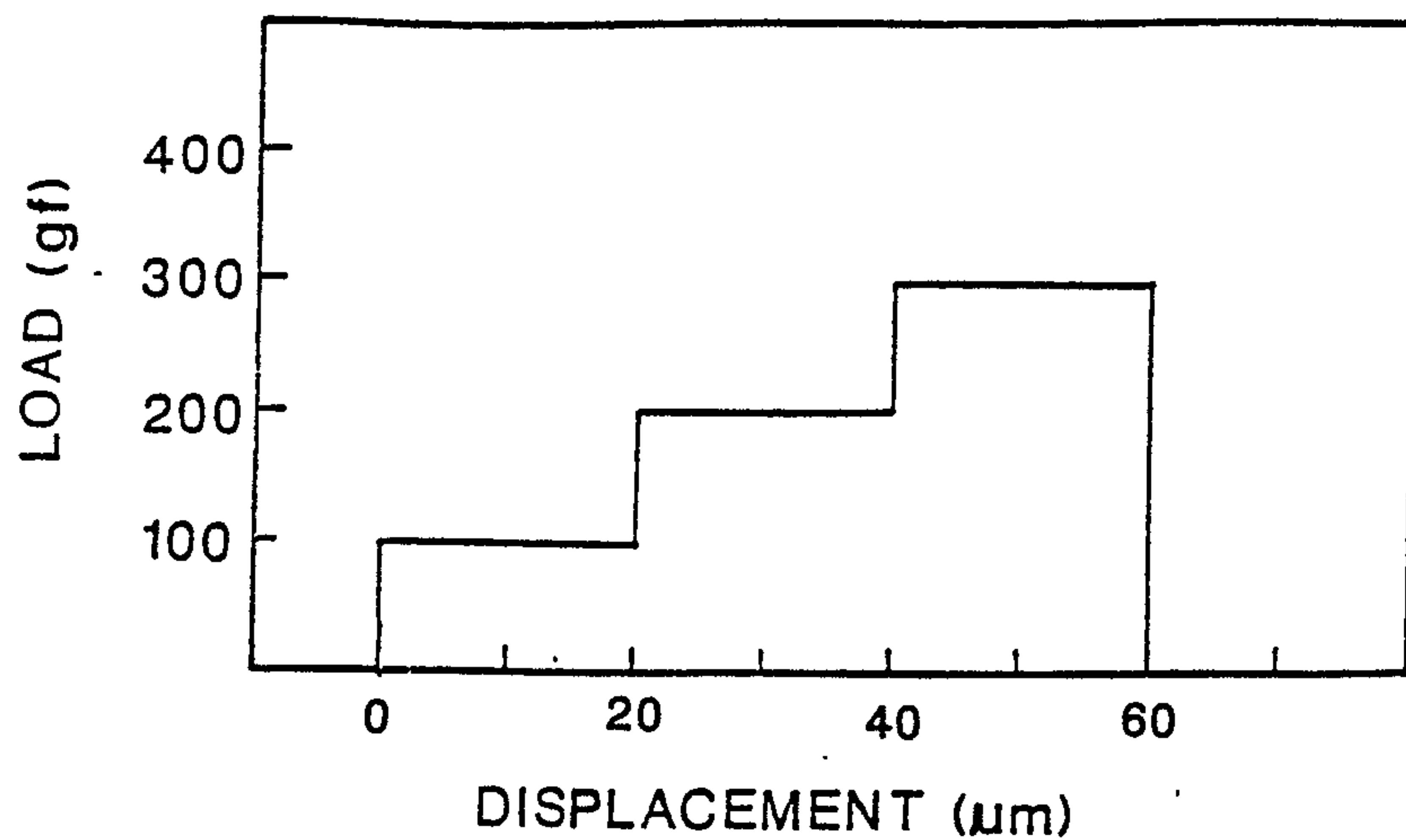
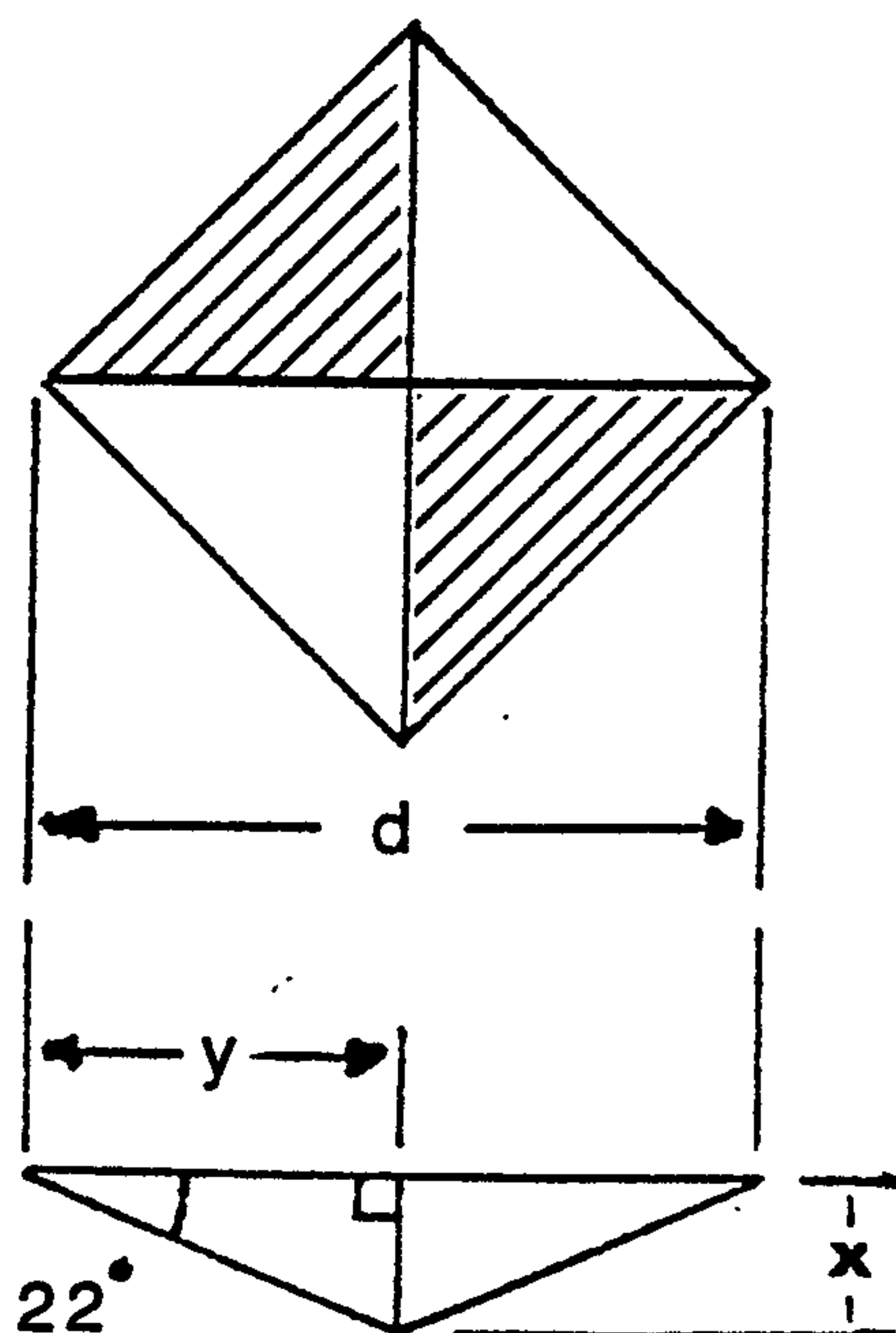


Figure 3.13 : CALIBRATION RECORDING OF THE TESTING MACHINE



$$H_v = \frac{1854.4 \times \text{load (gf)}}{d^2 (\mu\text{m})}$$

$$y = \frac{x}{\tan 22}$$

$$d = 2y = \frac{2x}{\tan 22}$$

$$H_v = \frac{1854.4 \times \text{load (gf)}}{\left(\frac{2x}{\tan 22}\right)^2}$$

Figure 3.14 : CALCULATION OF VICKERS HARDNESS NUMBER FROM THE GEOMETRY OF THE INDENTER TIP

CHAPTER FOUR - RESULTS

PART I - MATERIALS

4.1 Filler Content

TG results from the three inlay materials are summarised in Table 4.1. Representative DTA-TG curves are also shown in Figures 4.1 to 4.4. Reproducible TG curves were obtained in each material, the variation being less than 0.5 percent at any temperature.

In all materials only a small mass decrease of 1-2 percent occurred between 100 and 200°C and exothermic peaks were noted in the DTA curves of Isosit and Clearfil. Above this temperature the DTA curves of all materials became markedly complex showing a combination of both exothermic and endothermic peaks. A rapid mass decrease occurred between 300 and 400°C in Isosit and at a higher temperature, 400 to 500°C for Brilliant and Clearfil, giving the appearance that the depolymerisation reaction occurred as a two phase process. The TG curves of both Brilliant and Clearfil reached a constant at about 600°C, which provided values for filler content of 75 (Brilliant) and 86 (Clearfil) percent by mass. The TG curve of Isosit (Figure 4.1) did not reach a constant value by 1000°C. However, the mass decrease after 600°C was relatively small (Table 4.1). This provided a value for filler content of 69 percent by mass.

The DTA curve of Isosit with N-Fluid (Figure 4.4) showed a large exothermic peak at about 95°C indicating the breakdown of

the initiator Dibenzoyl Peroxide.

4.2 Processing Conditions

Representative heating curves obtained from the Ivomat are shown for the three temperature settings in Figure 4.5. For the recommended processing conditions (120°C, 10 minutes), the total processing time was 17 minutes. This was because the timer started operating after the selected temperature was reached. During the 10 minute processing time the temperature never significantly dropped below 120°C but it did reach 7.5°C above the recommended temperature. Similarly, the temperature remained about 5°C above the selected temperature at both the 110 and 100°C settings. For the ten minute setting the total processing times were 16 minutes and 14 minutes for the 110 and 100°C settings, respectively.

Representative processing temperatures of the three materials are summarised in Figure 4.6. Although a maximum temperature of about 90°C was reached momentarily in the DI 500, a lower temperature of about 75°C prevailed for the majority of the 8.5 minute processing cycle. The temperature of the dry oven used to heat the Clearfil remained fairly constant at 115°C.

A representative recording of the pressure measured in the Ivomat chamber is shown in Figure 4.7. The pressure rapidly reached 6×10^5 Pa and remained substantially constant ($\pm 2 \times 10^4$ Pa) during the processing time.

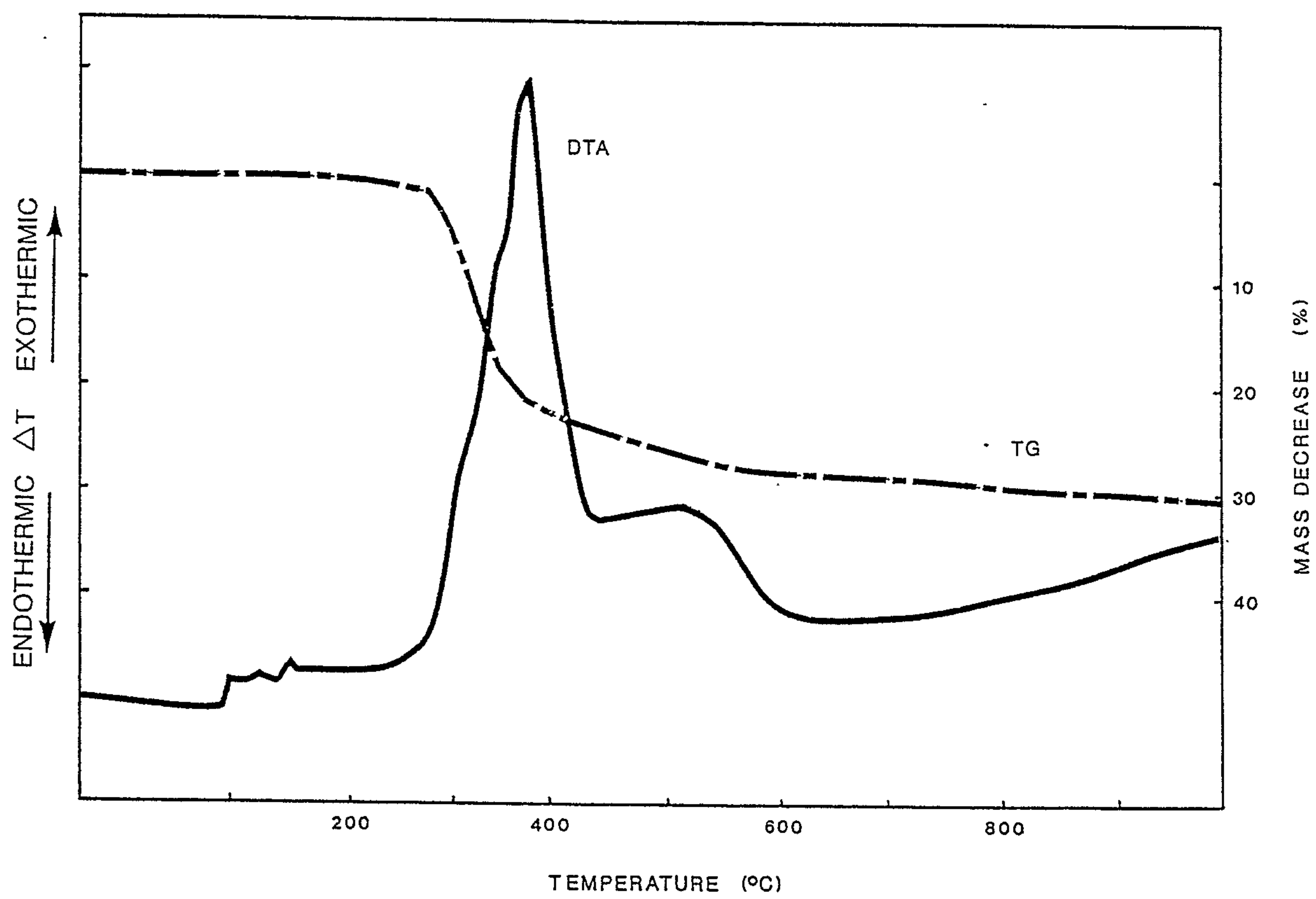


Figure 4.1 : DTA-TG curves for Isosit

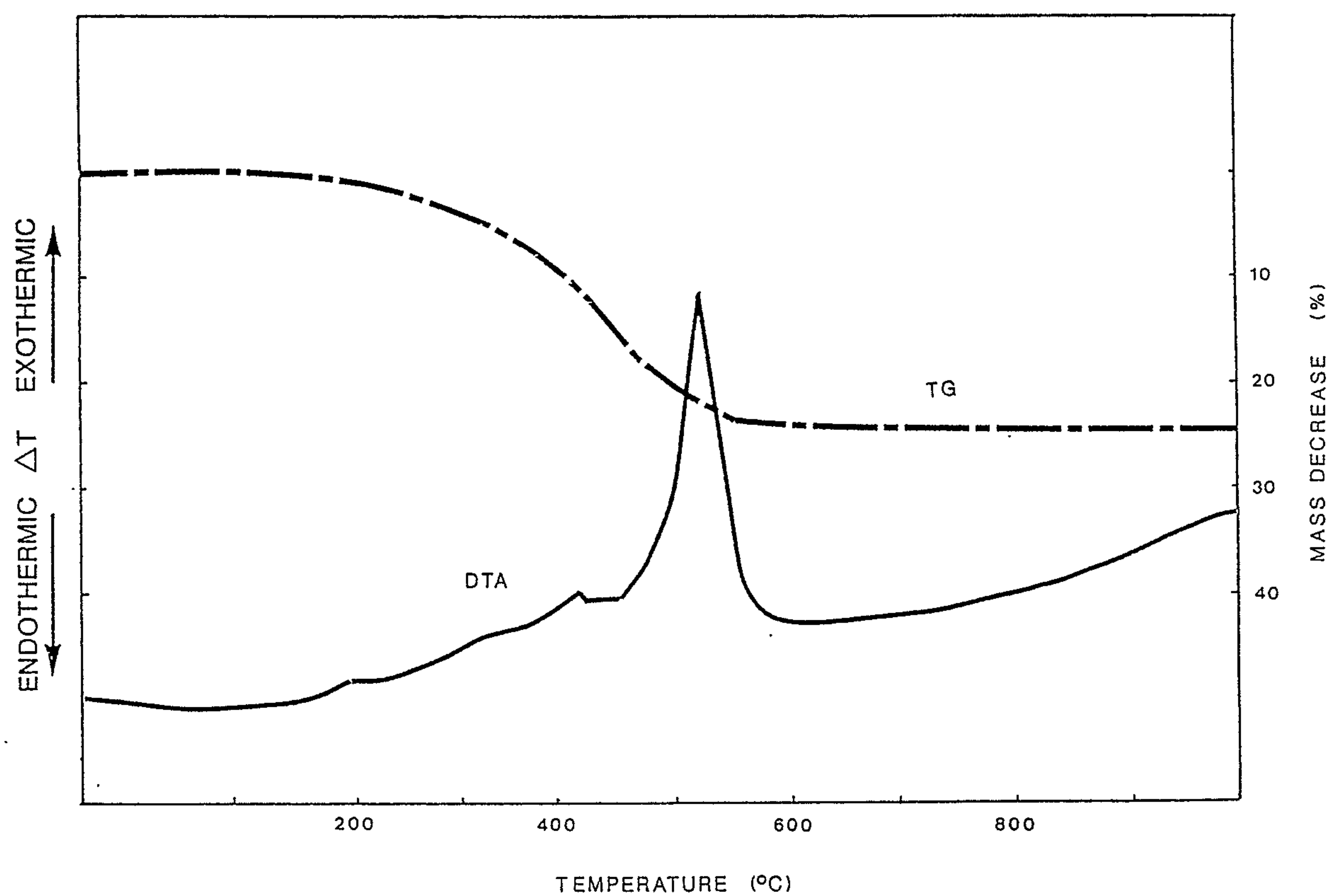


Figure 4.2 : DTA-TG Curves for Brilliant

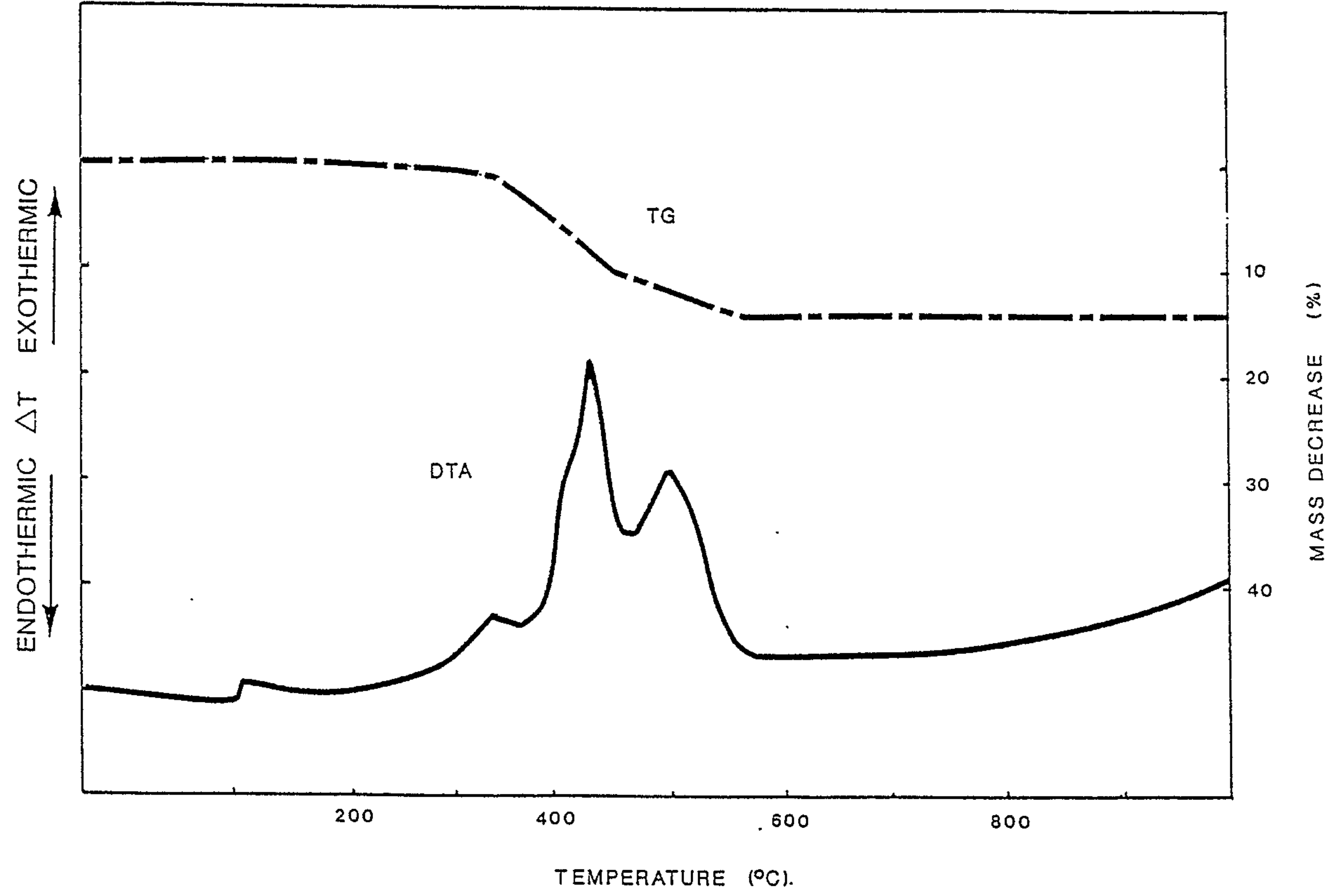


Figure 4.3 : DTA-TG curves for Clearfil

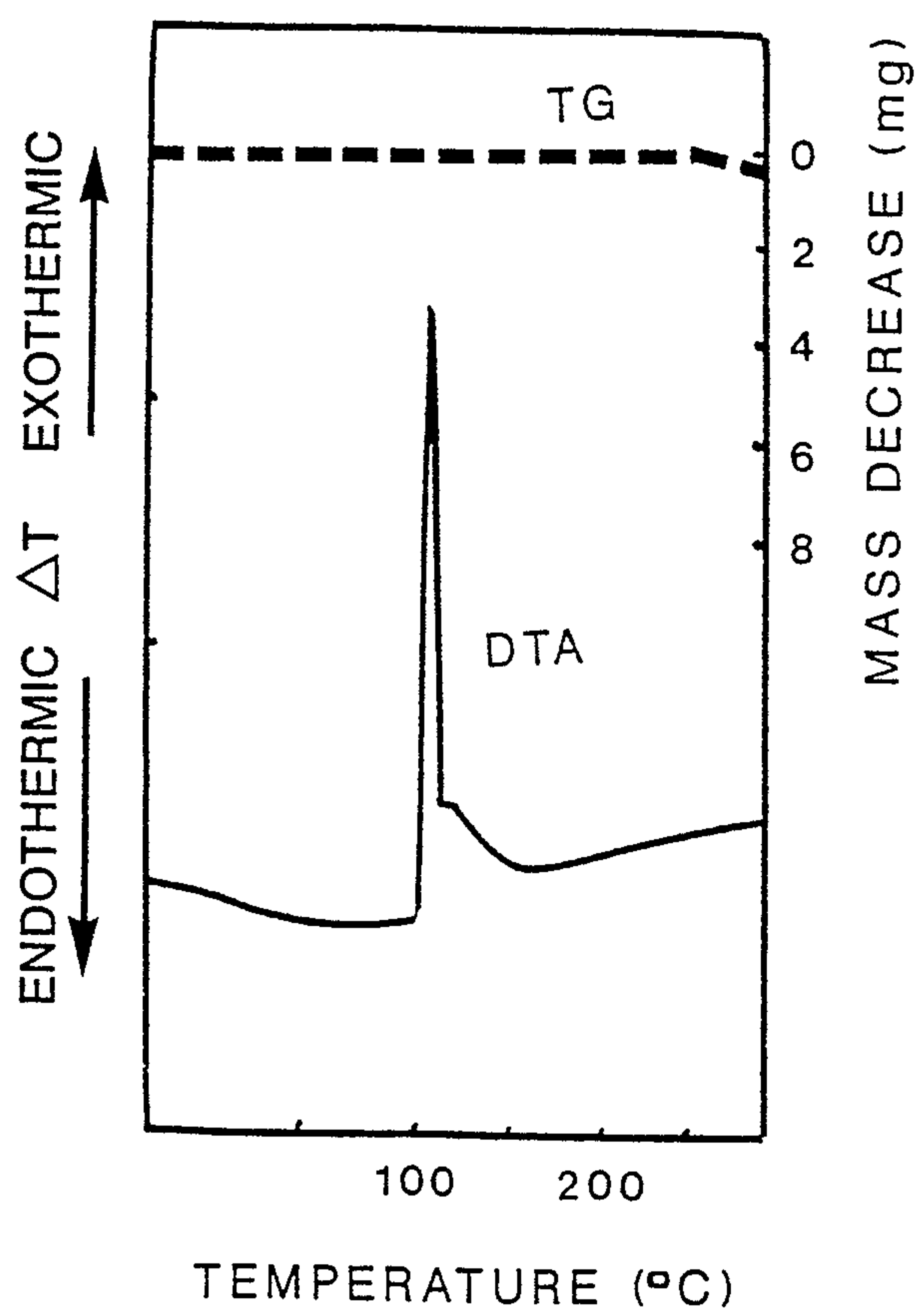


Figure 4.4 : DTA-TG curve for Isosit with N-Fluid

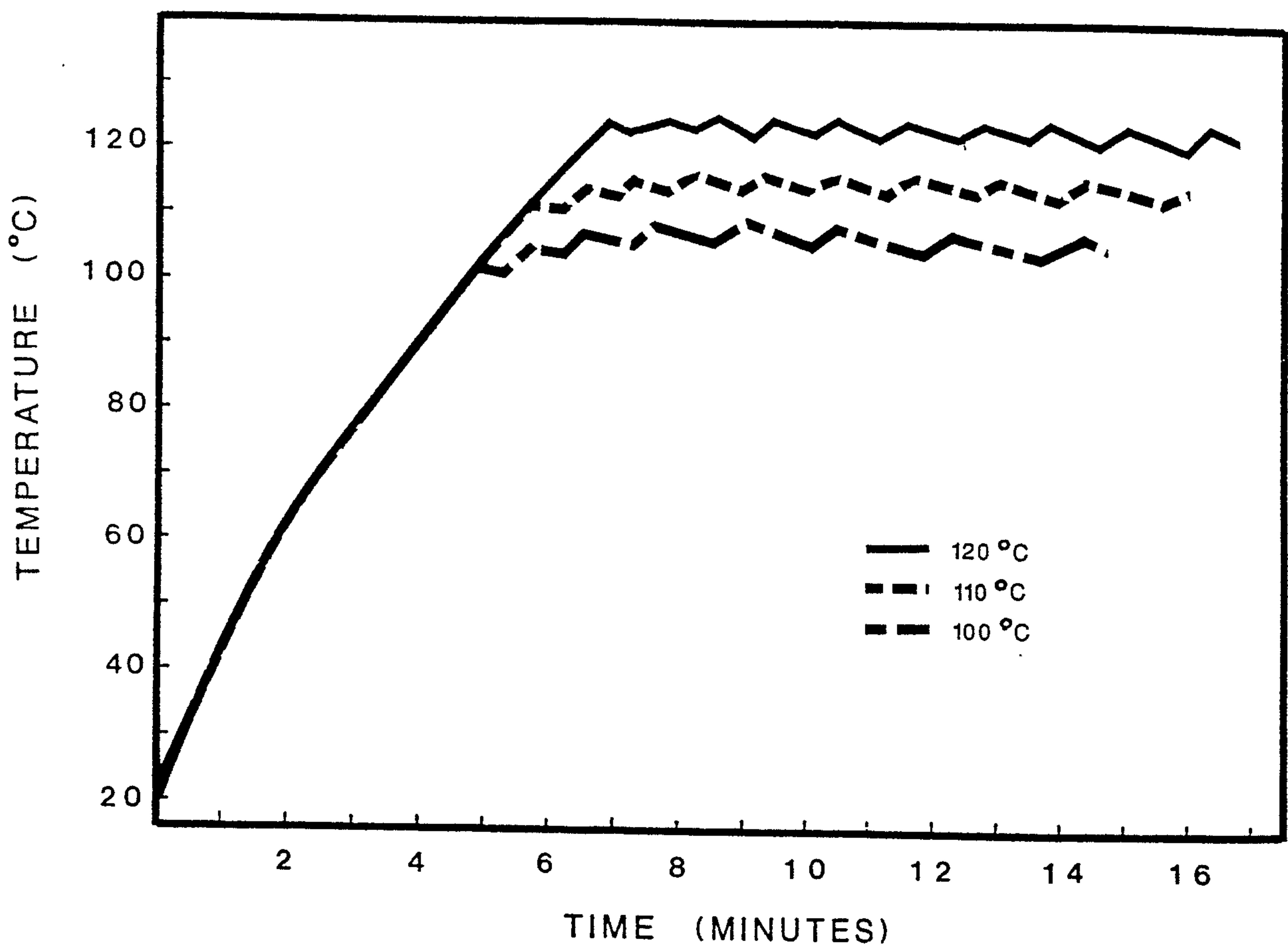


Figure 4.5 : Representative temperature recordings of 500 mL of water in the Ivomat.

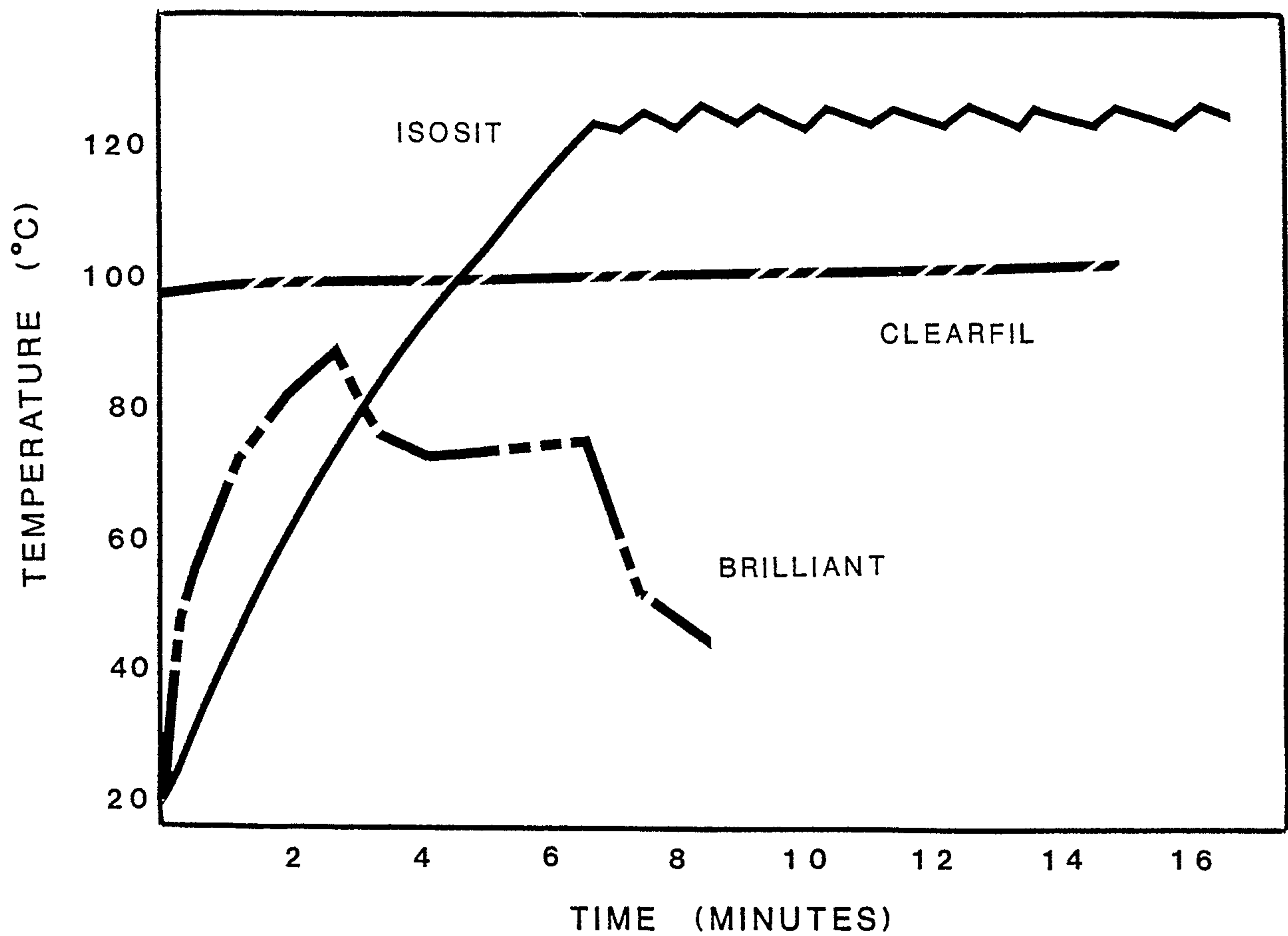


Figure 4.6 : Representative processing temperatures of Isosit (Ivomat), Brilliant (DI 500) and Clearfil (dry oven).

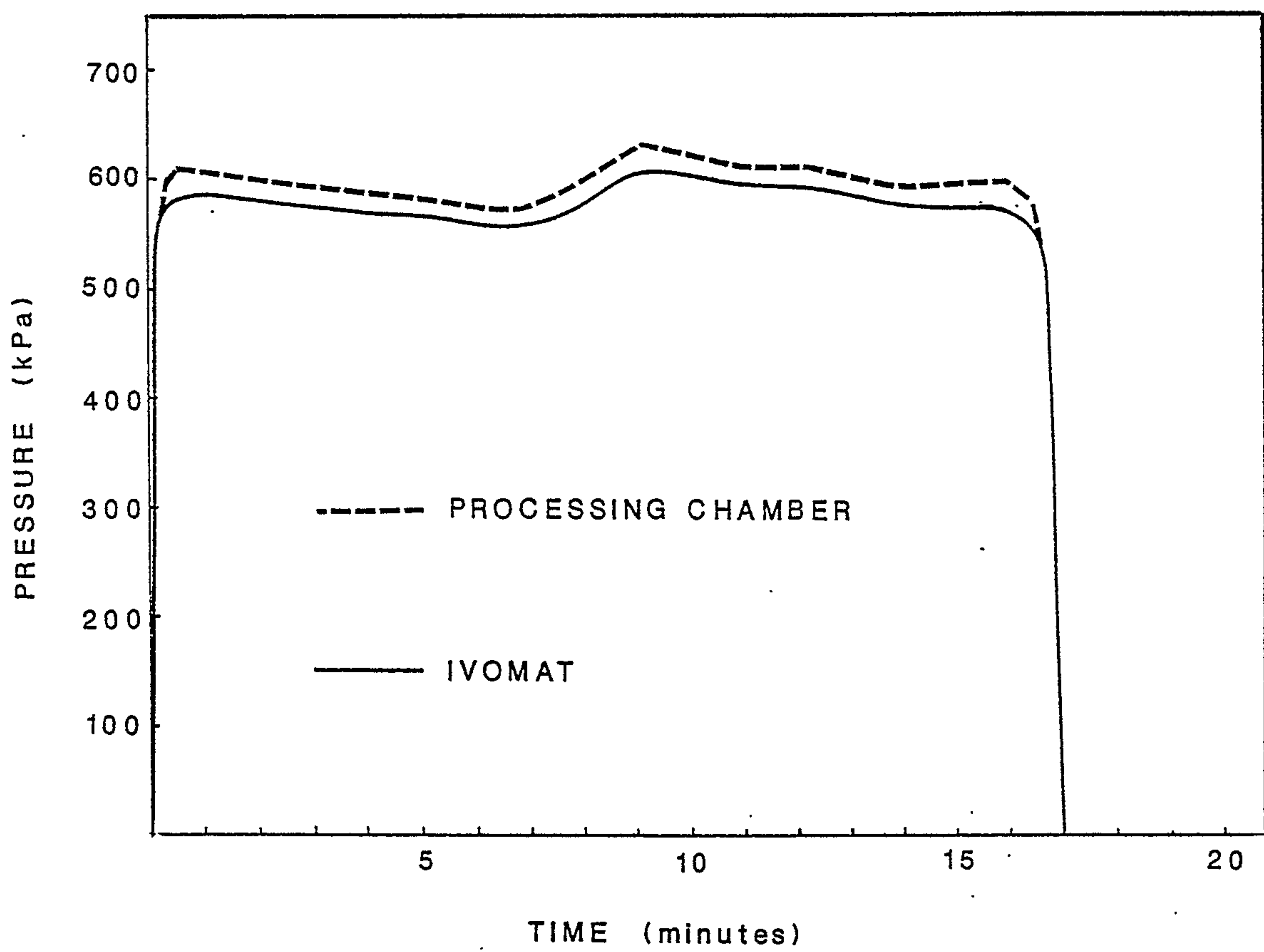


Figure 4.7 : Representative processing pressure Isosit.

**Table 4.1 : Percentage mass decrease of Isosit (IS)
Brilliant (BR) and Clearfil (CL)**

PART II - DIMENSIONAL ACCURACY

4.3 Impression Taking and Die Fabrication

A representative recording of the temperature change which occurred in the impression is shown in Figure 4.8. The temperature of the impression material loaded into the tray reached 37°C within the recommended setting time of six minutes. On removal from the oven the impression took approximately 60 minutes to cool to room temperature. A further temperature rise was seen in the impression due to the setting reaction of the die stone.

Axial protrusion measurements made of Isosit specimens for the impressions taken at ambient temperature and for the impressions taken at mouth conditions are shown in Tables 4.2 and 4.3. The mean axial protrusion of specimens from the impression taken at mouth conditions (0.430 mm) was significantly greater ($p < 0.05$) than the mean protrusion of specimens from the impression taken at ambient temperature (0.301 mm).

The axial protrusion measurements of Isosit specimens from the second pour model are shown in Table 4.4. The mean axial protrusion of specimens from the second pour model (0.320 mm) was not significantly different ($p > 0.05$) from specimens from the first pour model (0.301 mm).

The results of the compressive strengths of die stone are shown in Figure 4.9. The mean dry and wet strengths of the control specimens were 75 and 34 MPa respectively.

Dry and wet specimens immersed in 500 mL of water in the pressure chamber of the Ivomat at 20°C for 10 minutes showed strengths similar to the wet specimens. Similarly, dry and wet specimens immersed in 500 mL of water in the pressure chamber of the Ivomat at 100°C for 10 minutes showed strengths similar to wet strength. However, the higher temperatures (110 and 120°C) significantly ($p < 0.05$) reduced the strengths to 15 and 3 MPa respectively.

4.4 Polymerisation Shrinkage

The axial protrusion of Isosit specimens cured at 120°C for 10 minutes and measured using the microscope is shown in Table 4.5. There was no significant difference ($p < 0.05$) between the mean axial protrusion of Isosit specimens formed in the steel die (0.321 mm) and those formed in stone models made from impressions taken at ambient temperature (Section 4.3).

The mean difference between the two microscope measurements of Isosit specimens at each point is shown in Table 4.6. The mean difference (16 μm) was less than 5 percent of the mean protrusion.

The axial protrusion of a random selection of Isosit specimens cured at either 120°C for 10 minutes or 100°C for 10 or 20 minutes and measured using both the microscope and transducer is shown in Table 4.7. There was no significant difference ($p < 0.001$) between the two methods of measurement.

The axial protrusion of Brilliant and Clearfil specimens measured using the transducer are shown in Table 4.8.

The mean protrusion of Isosit specimens, 0.321 mm, was significantly greater ($p < 0.001$) than the mean protrusion of Clearfil specimens, 0.108 mm, which was significantly greater ($p < 0.05$) than the mean protrusion of Brilliant specimens, 0.042 mm.

The range of values for linear polymerisation shrinkage calculated from these means was 0.90 (Isosit), 0.12 (Brilliant) and 0.30 (Clearfil) percent.

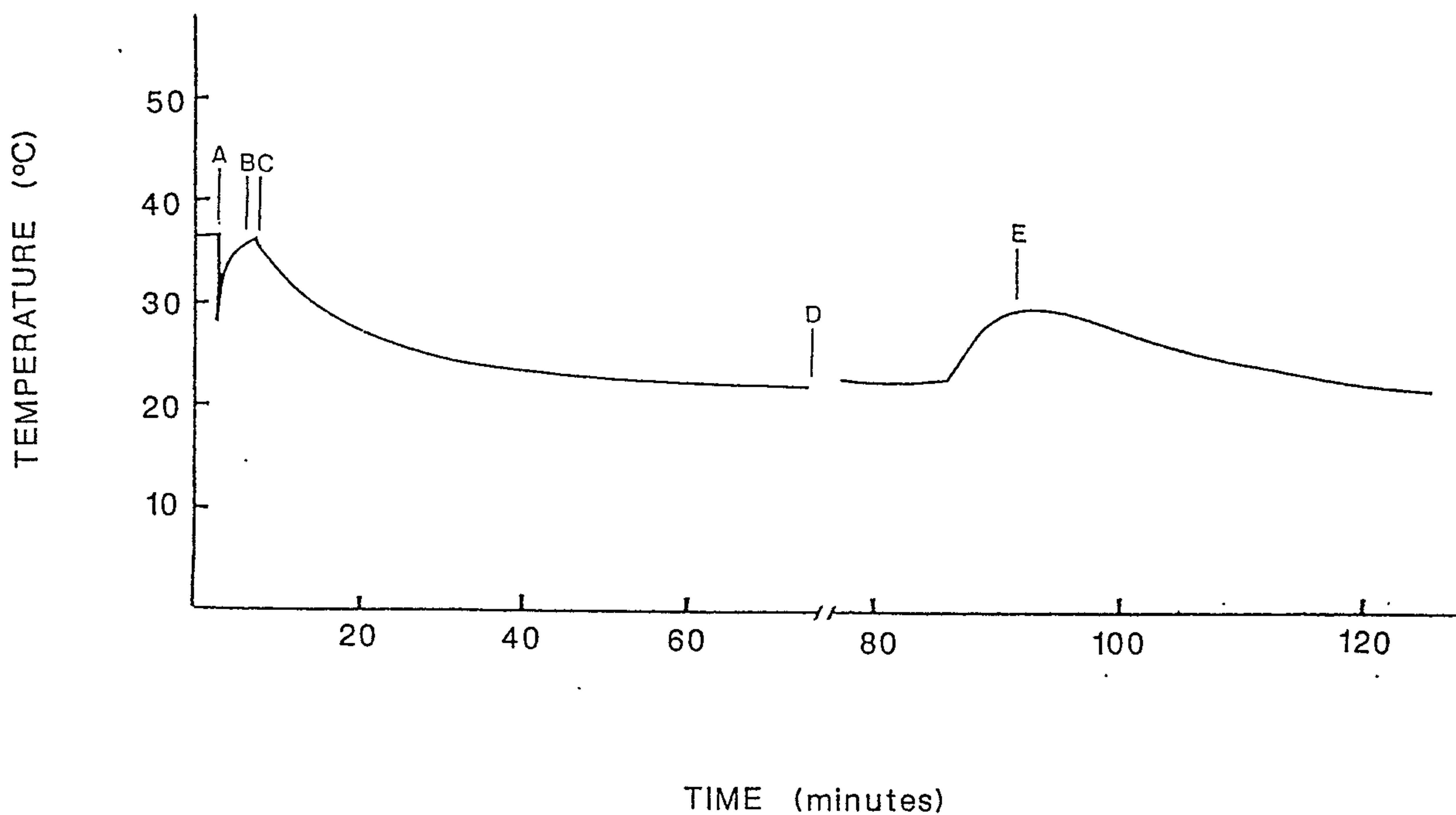


FIGURE 4.8 : Representative recording of the temperature change of the impression taken at mouth temperature and humidity. A - insertion of thermocouple, B - unseat impression, C - remove from oven, D - cool to room temperature, E - setting reaction of die stone.

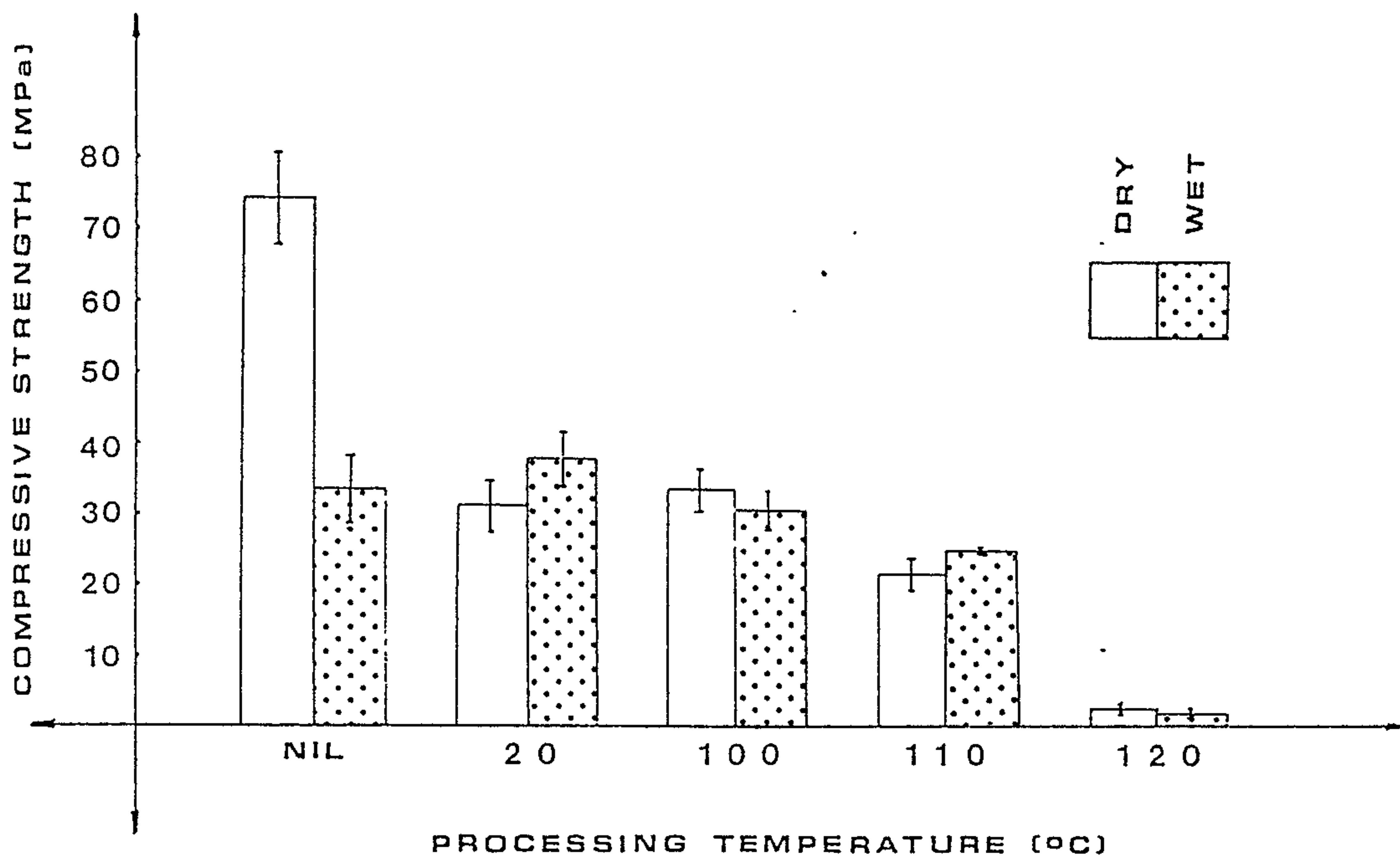


FIGURE 4.9 : Compressive strength of die stone subjected to various processing conditions.

TABLE 4.2 : Axial Protrusion of Isosit specimens for impressions taken at ambient temperature.

PROTRUSION (mm)					
S10	S11	S13	S14	S15	S16
.347	.380	.306	.359	.305	.273
.358	.330	.269	.364	.351	.336
.336	.343	.285	.301	.330	.254
.339	.300	.258	.322	.317	.265
.324	.301	.237	.251	.270	.284
.304	.278	.210	.259	.255	.264
.329	.319	.251	.336	.259	.289
.317	.332	.230	.319	.316	.287
MEAN = 0.301 mm (S.D. = 0.039)					

TABLE 4.3 : Axial protrusion of Isosit specimens for impressions taken at mouth temperature and humidity.

MEAN PROTRUSION (mm)					
S22	S23	S24	S25	S26	S27
.451	.450	.516	.482	.376	.414
.461	.456	.518	.503	.401	.414
.428	.445	.498	.452	.400	.327
.444	.454	.523	.453	.408	.354
.384	.446	.447	.416	.354	.313
.423	.435	.494	.435	.329	.297
.467	.454	.521	.422	.384	.355
.445	.451	.548	.430	.376	.366
MEAN = 0.430 mm			(S.D. = 0.057)		

TABLE 4.4 : Axial protrusion measurements of Isosit from the second pour die.

PROTRUSION (mm)					
S10	S11	S13	S14	S15	S17
.317	.364	.305	.282	.346	.391
.328	.308	.330	.311	.350	.383
.384	.302	.268	.337	.306	.376
.344	.301	.264	.349	.331	.350
.352	.330	.199	.337	.296	.334
.331	.317	.232	.364	.309	.323
.345	.292	.234	.324	.317	.317
.310	.308	.271	.310	.336	.333
MEAN = 0.320 mm (S.D. = 0.038)					

**Table 4.5 : Axial Protrusion of Isosit
(Microscope Measurement)**

SPECIMEN PROTRUSION (mm)					
12	14	16	21	22	23
.302	.286	.368	.365	.354	.363
.336	.307	.343	.365	.354	.357
.322	.310	.380	.401	.303	.372
.362	.305	.358	.407	.302	.370
.331	.293	.370	.334	.251	.355
.333	.274	.379	.344	.292	.360
.325	.263	.397	.304	.293	.343
.327	.266	.338	.294	.356	.345
MEAN = 0.321 mm (S.D. = 0.049)					

**TABLE 4.6 : Mean Difference in Protrusion Measurements
of Isosit Specimens Measured at each point.**

SAMPLE	MEAN DIFFERENCE IN PROTRUSION (μm)			
12	34	40	2	2
14	21	5	19	3
16	25	22	9	59
21	0	6	10	10
22	0	1	41	63
23	6	2	5	2
MEAN = 16 μm (S.D. = 14)				

**TABLE 4.7 : Axial Protrusion of Isosit Specimens.
Transducer and Microscope Measurements.**

SPECIMEN	CURING COND. (⁰ C/min)	PROTRUSION (mm)				
		MICROSCOPE				TRANSDUCER
22	120/10	.354	.354	.303	.302	.327
		.251	.292	.293	.356	.324
23	120/10	.363	.357	.355	.360	.348
		.372	.370	.343	.345	.339
20	100/10	.392	.394	.360	.354	.381
		.399	.380	.427	.412	.391
24	100/10	.386	.383	.353	.343	.347
		.356	.352	.379	.384	.351
26	100/20	.375	.362	.348	.338	.350
		.298	.313	.316	.310	.350
27	100/20	.329	.325	.355	.327	.318
		.349	.323	.337	.335	.330
MEAN		0.349 mm				0.346 mm
S.D.		0.034				0.022

**TABLE 4.8 : Axial Protrusion of Brilliant and Clearfil
(Transducer Measurement)**

CLEARFIL		BRILLIANT	
SPECIMEN	PROTRUSION (mm)	SPECIMEN	PROTRUSION (mm)
BR1	.100	CL1	.075
	.102		.075
BR2	.045	CL2	.190
	.053		.235
BR3	.012	CL3	.228
	.008		.213
BR4	.000	CL4	.028
	.020		.006
BR5	.122	CL5	.035
	.192		.040
BR6	-.072	CL6	.087
	-.085		.085
MEAN PROTRUSION	0.042 mm		0.108 mm
S.D.	0.082		0.084

PART III - DIMENSIONAL STABILITY

4.5 Water Sorption

The amounts of water absorbed by the inlay specimens are shown for the three materials in Tables 4.9 to 4.11. The results expressed as the percentage mass of water per mass of specimen are shown in Table 4.12.

After seven days immersion, the average amounts of water absorbed by specimens were, 3.2 mg (Isosit), 1.3 mg (Brilliant) and 1.5 mg (Clearfil). In Brilliant and Clearfil significant increases ($p < 0.05$) to 2.1 and 2.2 mg respectively were recorded at 30 days, while there was no change in Isosit (3.1 mg). After six months immersion there were no further significant ($p > 0.05$) changes in the three materials.

At six months immersion, Isosit specimens absorbed a significantly higher percentage of water (1.2 percent) than either Clearfil or Brilliant specimens ($p < 0.05$). There was no significant difference ($p > 0.05$) between Brilliant and Clearfil specimens.

Compared with the protrusion measured with dry specimens, that obtained at seven days immersion was greatly decreased in all materials. The difference in protrusion of dry and wet specimens from the die is summarised in Tables 4.13 to 4.15.

The mean linear expansions resulting from water sorption calculated from these measurements were 0.15 (Isosit), 0.08 (Brilliant) and 0.05 (Clearfil) percent at seven days.

After thirty days immersion, there was a further significant difference ($p < 0.1$) in the mean protrusion between dry and soaked specimens in all three inlay materials. This led to a mean linear expansion of 0.41 percent in Isosit which was significantly greater ($p < 0.001$) than either Brilliant or Clearfil specimens, 0.13 and 0.17 percent respectively. There was no significant difference ($p > 0.05$) between Brilliant and Clearfil specimens.

After six months, the protrusion was further decreased all materials. The mean linear expansions calculated were 0.52 (Isosit), 0.31 (Brilliant) and 0.30 (Clearfil) percent. The mean linear expansion of Isosit specimens was significantly greater ($p < 0.001$) than either Clearfil or Brilliant specimens. There was no significant difference ($p > 0.05$) between Clearfil and Brilliant specimens.

4.6 Coefficient of Thermal Expansion

Typical thermal expansion curves of the three materials are shown in figures 4.10 to 4.12 and thermal expansion coefficients calculated from these curves are summarised for three different temperature ranges in Table 4.16. The coefficients increased as the temperature range increased in all materials. At the lowest range the coefficient of Isosit was highest followed by Brilliant and Clearfil. The typical thermal expansion curve of Isosit specimens processed according to the recommended conditions (120°C , 10 minutes) are compared with those processed at 100°C for 10 and 20 minutes in Figure 4.13. Coefficients of thermal expansion obtained from these specimens processed at 100°C are compared in Table 4.17.

The curve of specimens processed at 100°C for 10 minutes showed a plateau at approximately 130 to 140°C, giving a very low coefficient of $16.6 \times 10^{-6}/^{\circ}\text{C}$. This was not observed when the specimens were processed at 100°C for the longer time of 20 minutes.

4.7 Hardness

A representative recording of the hardness test of the standard metal sample is shown in Figure 4.14. The stepped appearance of the loading curve is due to the incremental loading of the specimen by the stepper motor (Section 3.9). At the maximum load, reversal of the direction of the motor to initiate unloading is delayed momentarily which creates an extended plateau on the loading curve as displacement of the indenter continues under a static load. Similarly, the stepped appearance of the recovery curve is due to increasing force on the load cell from elastic recovery of the material during the pauses in unloading.

The depth of penetration at 300 gf on loading and that at 20 gf on unloading are shown in Table 4.18. The mean depth of penetration at 300 gf was 4.3 μm . The known Vickers hardness number, 769-780, represents a depth of 5.4 μm at 300 gf giving an accuracy of $\pm 1 \mu\text{m}$ for these measurements.

Representative recordings of the hardness tests of the three composite inlay materials on the day of processing are shown in Figure 4.15. The depths of penetration at 500 gf on the day of processing and after 7 days immersion are shown in Tables 4.19 to 4.21. Mean depths of penetration, 30 μm (Isosit), 19 μm (Brilliant), 18 μm (Clearfil), did not change significantly

($p < 0.05$) after 7 days. Vickers hardness numbers calculated were 42 (Isosit), 86 (Brilliant) and 117 (Clearfil) at 7 days.

The depths of penetration at 500 gf after six months immersion are shown in Tables 4.22 to 4.24. The mean depth remained unchanged in Isosit (30 μm) but was significantly greater ($p < 0.05$) in Brilliant (24 μm) and Clearfil (23 μm). Vickers hardness numbers calculated were 42 Isosit, 66 (Brilliant) and 72 (Clearfil).

Tables 4.25 to 4.27 compare the depths of penetration at 800 gf (loading) and 20 gf (unloading) at seven days. Mean depths of penetration at 800 gf, 42 μm (Isosit), 29 μm (Brilliant) and 26 μm (Clearfil) recovered to 19 μm (Isosit), 17 μm (Brilliant) and 14 μm (Clearfil) upon unloading. The mean percentage recoveries were 55 percent (Isosit), 41 percent (Brilliant) and 46 percent (Clearfil).

The depths of penetration at 800 gf and 20 gf are shown for six months immersion in Tables 4.28 to 4.30. Mean depths of penetration at 800 gf, 42 μm (Isosit), 33 μm (Brilliant) and 30 μm (Clearfil) recovered to 21 μm (Isosit), 19 μm (Brilliant) and 24 μm (Clearfil) upon unloading. The mean percentage recoveries were 50 percent (Isosit), 42 percent (Brilliant) and 20 percent (Clearfil). The marked decrease in the recovery of Clearfil between 7 days and 6 months, 46 to 20 percent, was shown by a change in the shape of the indentation curve (Figure 4.16).

TABLE 4.9 : Water Sorption of Isosit Specimens

MASS OF ABSORBED WATER (g)			
SPECIMEN	7 DAYS	30 DAYS	6 MONTHS
12/12/12	0.0030	0.0026	0.0029
21/21/21	0.0029	0.0023	0.0025
23/23/23	0.0034	0.0020	0.0024
14/S23/S23	0.0041	0.0038	0.0039
22/S25/S25	0.0030	0.0041	0.0048
16/S26/S26	0.0030	0.0040	0.0043
MEAN	0.0032	0.0031	0.0035
S.D.	0.0005	0.0009	0.0010

TABLE 4.10 : Water Sorption of Brilliant Specimens

MASS OF ABSORBED WATER (g)			
SPECIMEN	7 DAYS	30 DAYS	6 MONTHS
BR1	0.0012	0.0021	0.0020
BR2	0.0009	0.0021	0.0016
BR3	0.0012	0.0022	0.0020
BR4	0.0014	---	0.0027
BR5	0.0014	0.0021	0.0026
BR6	0.0016	0.0020	---
MEAN	0.0013	0.0021	0.0022
S.D.	0.0002	0.0001	0.0004

TABLE 4.11 : Water Sorption of Clearfil Specimens

MASS OF ABSORBED WATER (g)			
SPECIMEN	7 DAYS	30 DAYS	6 MONTHS
CL1	0.0018	0.0018	0.0019
CL2	0.0017	0.0023	0.0020
CL3	0.0020	0.0027	0.0021
CL4	0.0010	0.0019	0.0022
CL5	0.0012	0.0022	0.0023
CL6	0.0015	0.0025	0.0022
MEAN	0.0015	0.0022	0.0021
S.D.	0.0004	0.0009	0.0001

TABLE 4.12 : Water Sorption (% mass)

	ISOSIT	CLEARFIL	BRILLIANT
7 DAYS	1.0	0.4	0.4
30 DAYS	1.0	0.6	0.7
6 MONTHS	1.2	0.6	0.7

TABLE 4.13 : Mean Difference in Axial Protrusion between Dry and Wet Isosit Specimens

MEAN DIFFERENCE IN PROTRUSION (mm)			
SPECIMEN	7 DAYS	30 DAYS	6 MONTHS
12/12/12	0.078	0.164	0.178
21/21/21	0.027	0.138	0.266
23/23/23	0.038	0.213	0.260
14/S23/S23	0.077	0.090	0.102
22/S25/S25	0.035	0.127	0.116
16/S26/S26	0.061	0.139	0.206
MEAN	0.053	0.145	0.188
S.D.	0.022	0.041	0.069

TABLE 4.14 : Mean Difference in Axial Protrusion between Dry and Wet Brilliant Specimens

MEAN DIFFERENCE IN PROTRUSION (mm)			
SPECIMEN	7 DAYS	30 DAYS	6 MONTHS
BR1	0.010	0.030	0.094
BR2	0.042	0.057	0.090
BR3	0.035	0.059	0.130
BR4	0.002	---	0.096
BR5	0.063	0.050	0.142
BR6	0.026	0.028	---
MEAN	0.030	0.045	0.110
S.D.	0.022	0.015	0.023

TABLE 4.15 : Mean Difference in Axial Protrusion between Dry and Wet Clearfil Specimens

MEAN DIFFERENCE IN PROTRUSION (mm)			
SPECIMEN	7 DAYS	30 DAYS	6 MONTHS
CL1	0.009	0.037	0.041
CL2	0.000	0.043	0.080
CL3	0.019	0.077	0.157
CL4	0.027	0.093	0.126
CL5	0.024	0.058	0.116
CL6	0.022	0.058	0.129
MEAN	0.017	0.061	0.108
S.D.	0.010	0.021	0.041

**Table 4.16 : Summary of Mean Coefficients of Thermal Expansion
($\times 10^{-6}/^{\circ}\text{C}$)**

	ISOSIT	BRILLIANT	CLEARFIL
COEFFICIENT (S.D)	30.7 (2.3)	25.1 (5.1)	20.8 (1.5)
TEMPERATURE	20-65 ⁰ C	20-55 ⁰ C	20-65 ⁰ C
COEFFICIENT (S.D)	39.0 (1.2)	39.4 (4.5)	25.4 (0.3)
TEMPERATURE	75-130 ⁰ C	55-112 ⁰ C	65-102 ⁰ C
COEFFICIENT (S.D)	47.0 (5.0)	53.8 (4.0)	31.6 (0.4)
TEMPERATURE	145-238 ⁰ C	125-208 ⁰ C	158-261 ⁰ C

TABLE 4.17 : Summary of mean coefficients of thermal expansion of Isosit specimens ($\times 10^{-6}/^{\circ}\text{C}$).

TEMPERATURE RANGE	PROCESSING CONDITIONS		
	120 ⁰ C 10 minutes	100 ⁰ C 10 minutes	100 ⁰ C 20 minutes
20 - 65 ⁰ C	30.7 (2.3)	35.1 (1.4)	26.1 (1.0)
75 - 130 ⁰ C	39.0 (1.2)	44.5 (4.2)	34.0 (2.2)
120 - 140 ⁰ C	32.0 (2.9)	16.6 (8.4)	-----
145 - 238 ⁰ C	47.0 (5.0)	45.0 (3.0)	44.2 (4.4)

() standard deviation

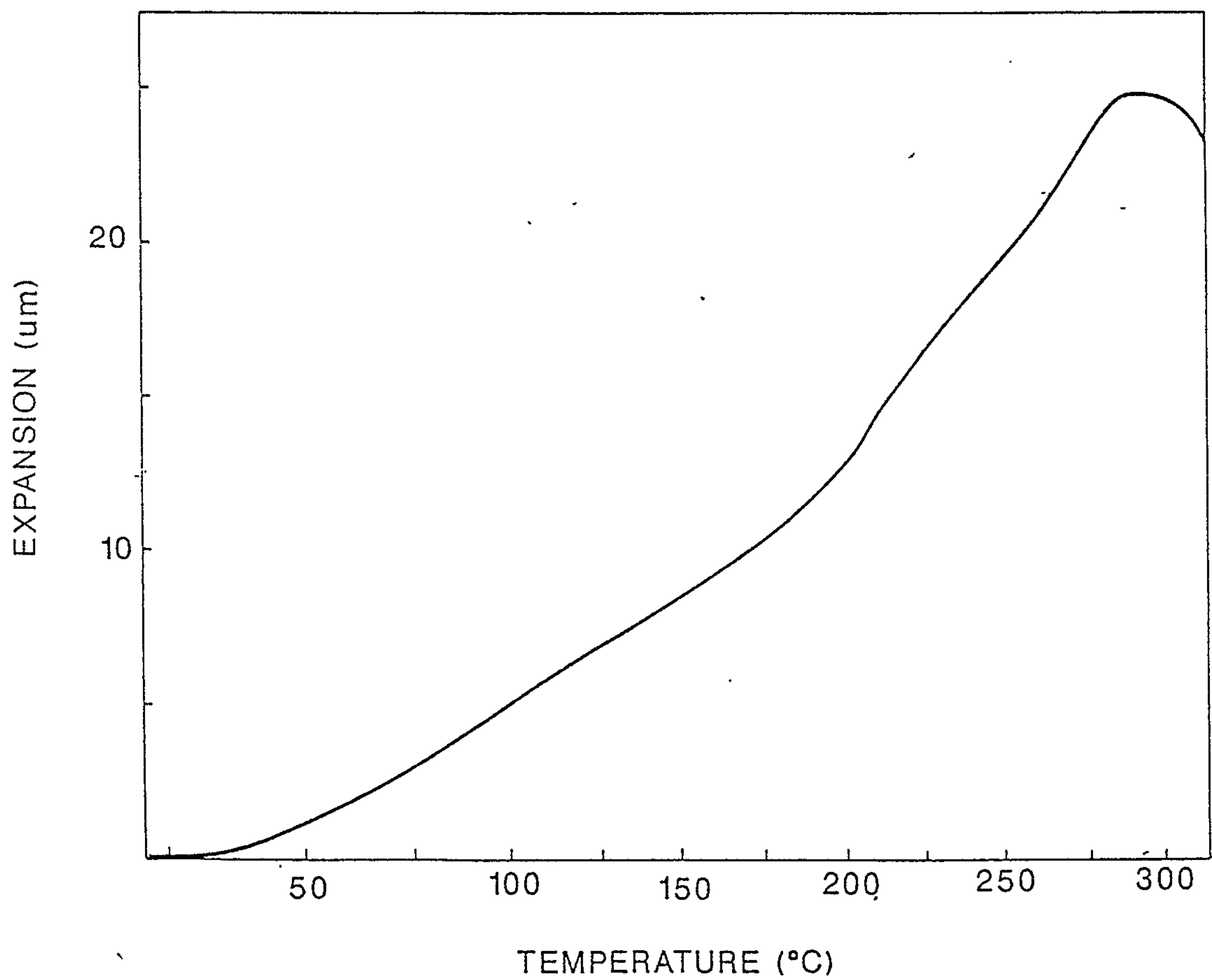


Figure 4.10 : Representative thermal expansion curve of Isosit processed at 120⁰C for 10 minutes.

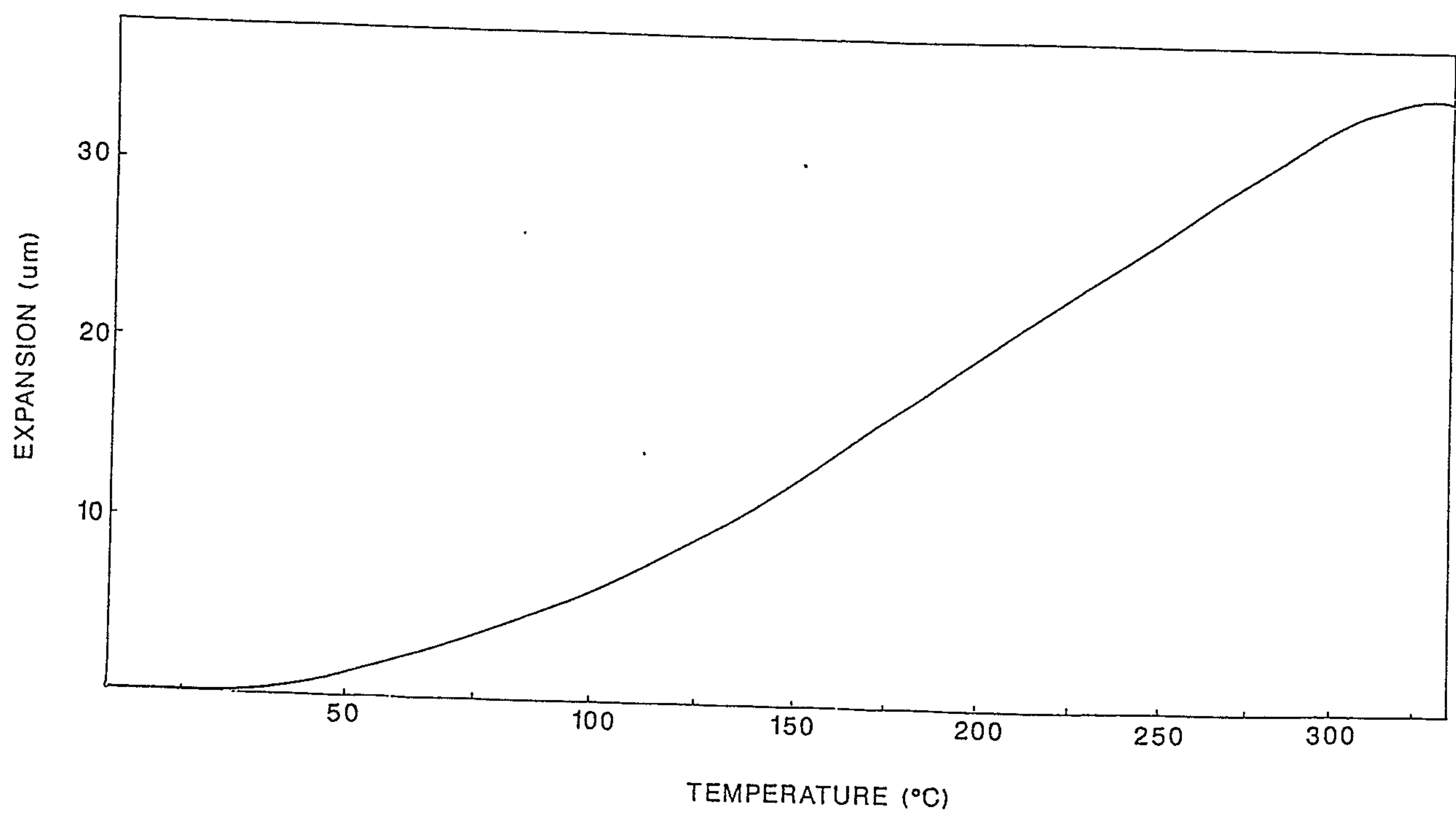


Figure 4.11 : Representative thermal expansion curve of Brilliant.

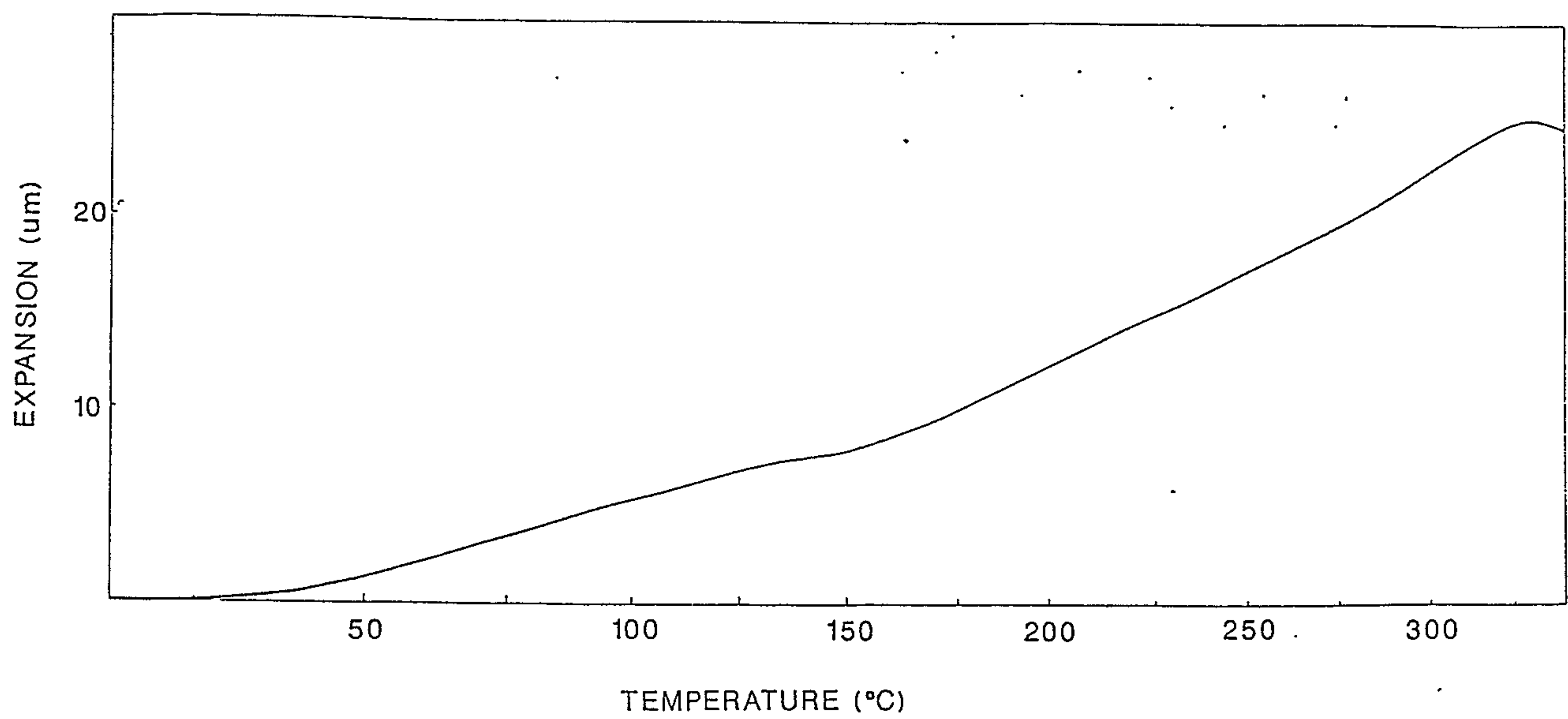


Figure 4.12 : Representative thermal expansion curve of Clearfil.

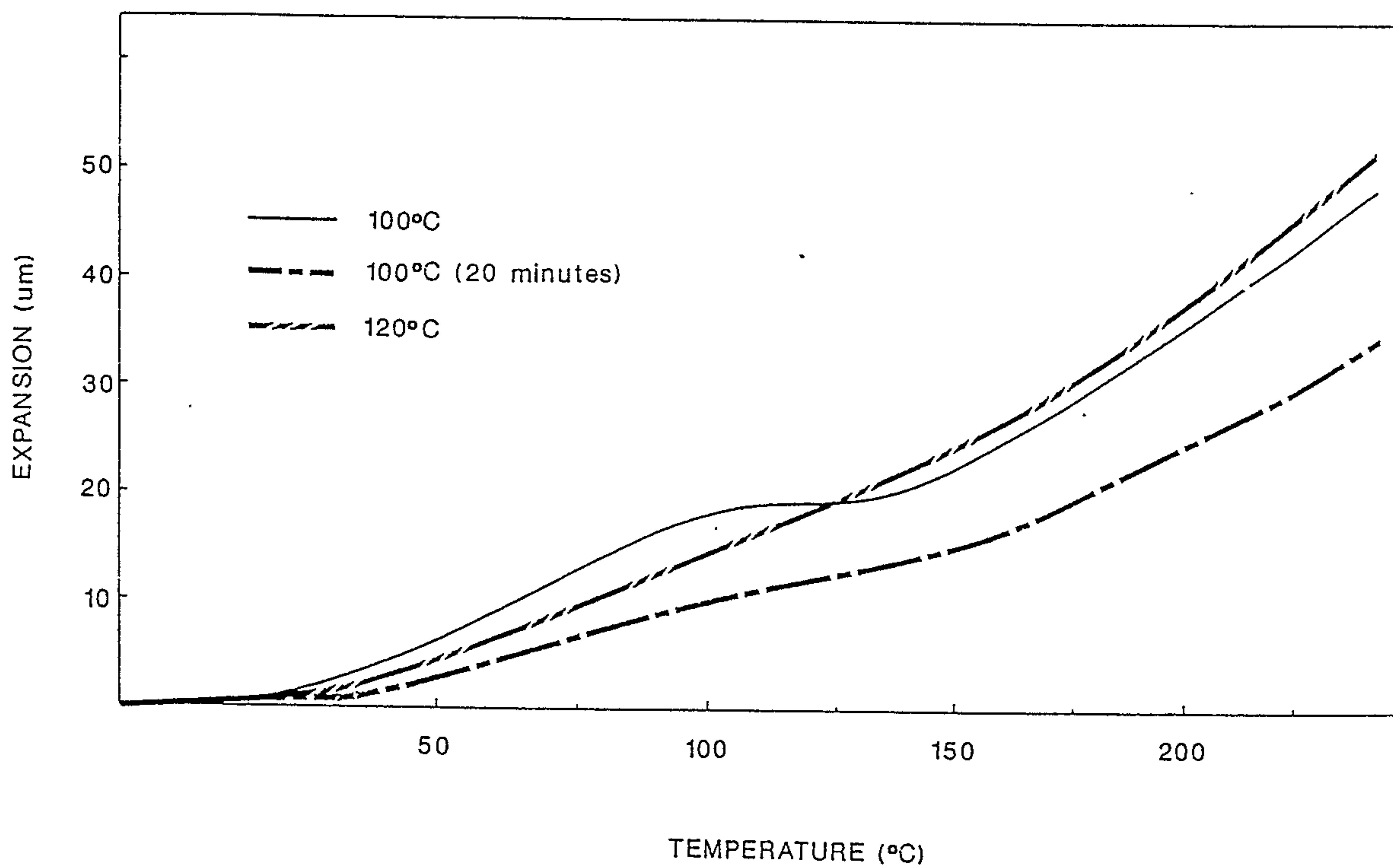


FIGURE 4.13 : Representative thermal expansion curves of Isosit specimens processed at 120°C for 10 minutes and 100°C for 10 and 20 minutes.

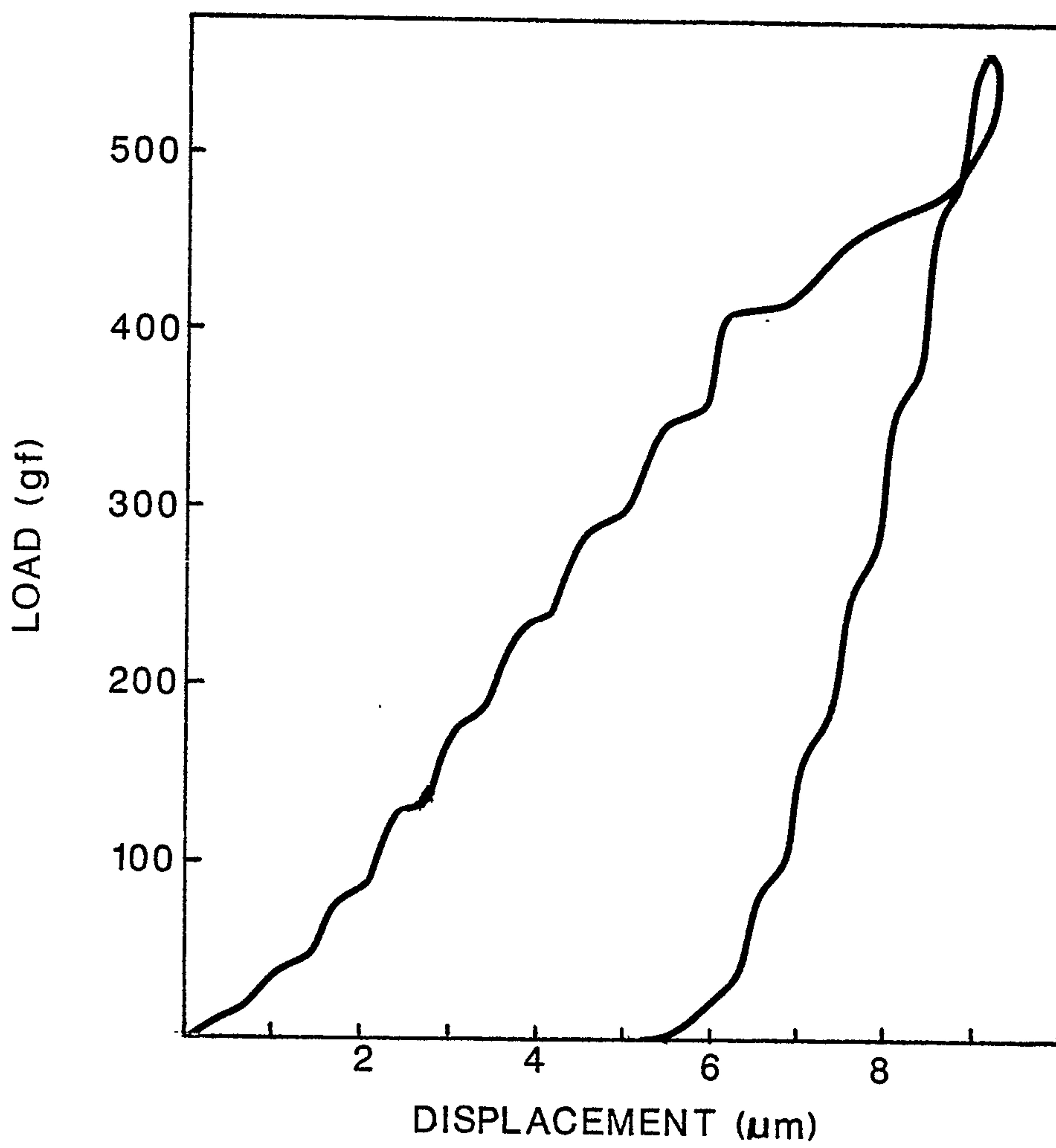


Figure 4.14 : A representative recording of the hardness test of the metal specimen.

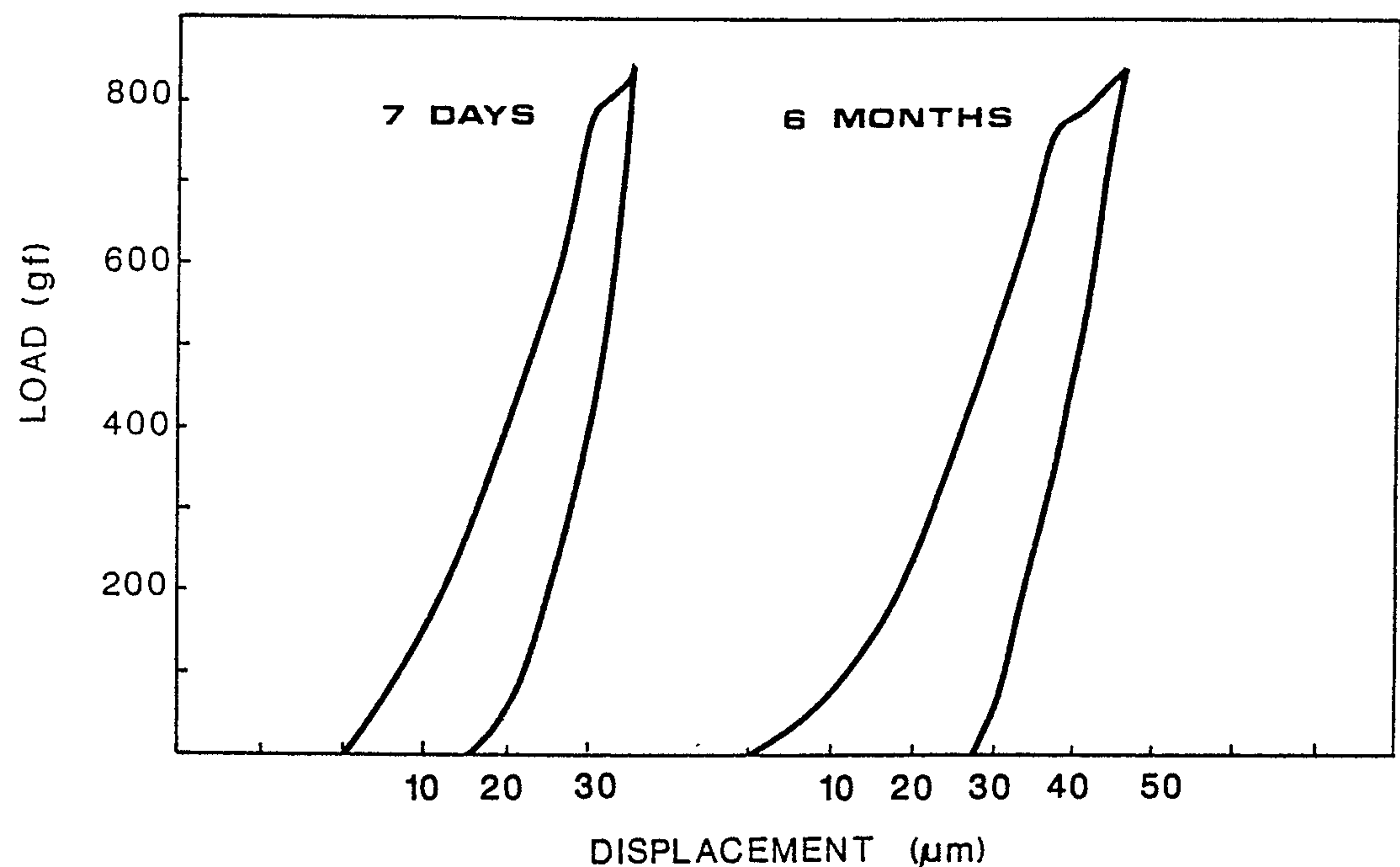


Figure 4.15 : Representative recordings of the hardness tests of Isosit, Clearfil and Brilliant specimens on the day of processing.

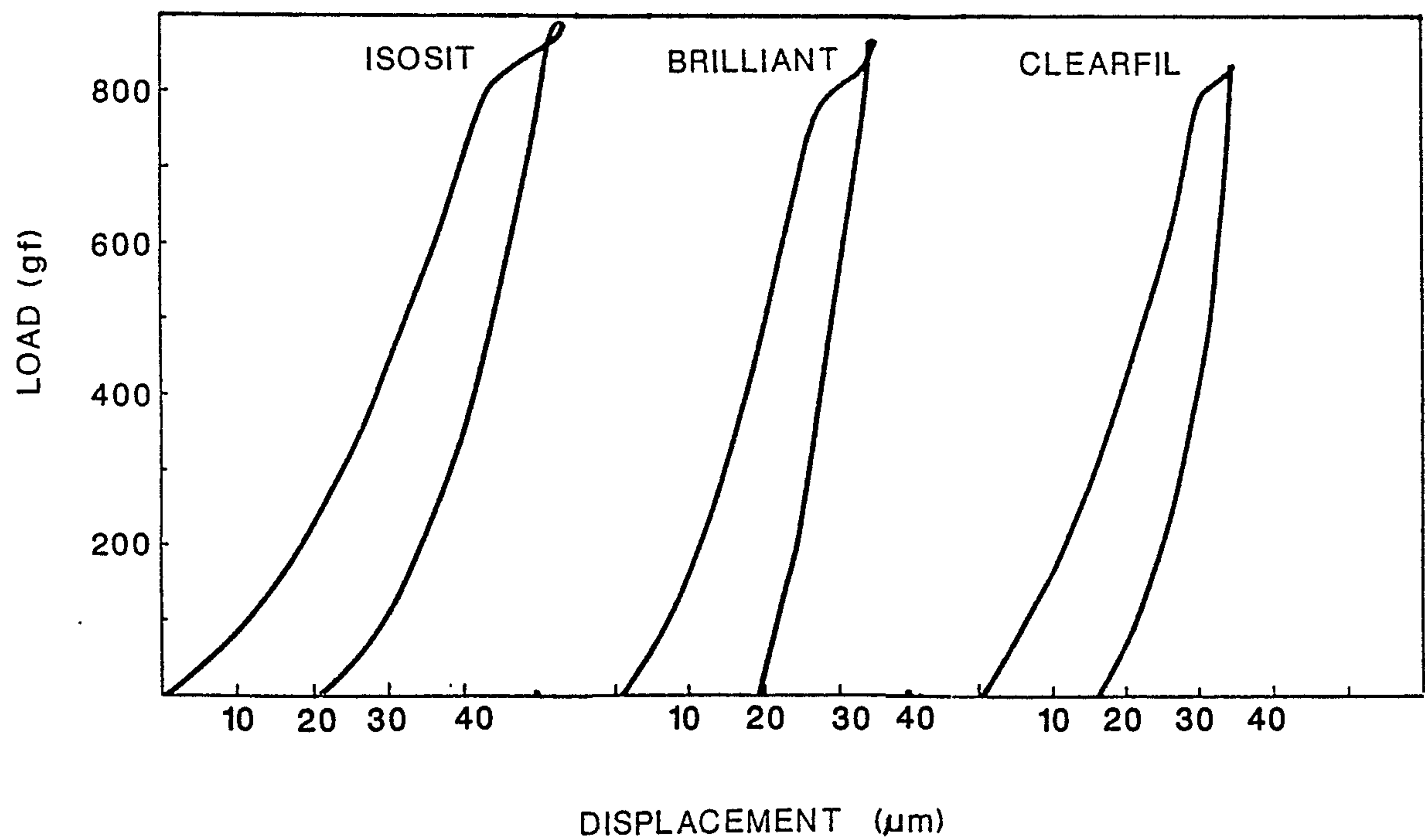


Figure 4.16 : Representative recordings of the hardness tests of Clearfil at seven days and six months.

Table 4.18 : Depths of penetration (standard metal specimen).

Depth (μm)			
loading (300 gf)		unloading (20 gf)	
4.4	4.0	5.2	6.2
4.0	4.7	6.3	5.5
3.8	5.0	5.3	5.2
MEAN = 4.3		MEAN = 5.6	
S.D. = 0.5		S.D. = 0.5	

Table 4.19 : Depths of penetration at 500 gf (Isosit).

SAMPLE	DISPLACEMENT (μm)											
	4 hours						7 days					
H1	26	37	27	32	37	30	34	32	27	28	28	32
H2	38	25	33	29	32	26	28	28	29	30	31	38
H3	37	27	27	28	27	27	34	29	25	25	28	33
MEAN	30						30					
S.D.	4						3					

Table 4.20 : Depths of penetration at 500 gf (Brilliant).

SAMPLE	DISPLACEMENT (μm)											
	4 hours						7 days					
B11	17	21	16	15	20	20	21	20	20	21	17	20
B12	23	19	19	19	18	18	24	20	21	20	20	21
B13	19	23	16	15	18	19	26	20	24	19	20	17
MEAN	19						21					
S.D.	2						2					

Table 4.21 : Depths of penetration at 500 gf (Clearfil).

SAMPLE	DISPLACEMENT (μm)											
	4 hours						7 days					
C11	25	15	16	18	16	16	19	19	18	16	19	16
C12	18	14	17	17	16	19	27	17	16	15	17	17
C13	18	18	22	16	18	19	21	21	20	19	17	16
MEAN	18						18					
S.D.	3						3					

Table 4.22 : Depths of penetration at 500 gf.
(Isosit, six months)

SAMPLE	DISPLACEMENT (μm)					
12	46	30	27	28	29	39
21	36	32	32	22	25	31
23	28	34	32	27	23	25
MEAN = 30						S.D. = 6

**Table 4.23 : Depths of penetration at 500 gf.
(Brilliant, six months)**

SAMPLE	DISPLACEMENT (μm)					
B1	40	28	26	25	25	27
B2	36	22	18	21	22	22
B3	21	19	21	19	16	18
MEAN = 24				S.D. = 6		

**Table 4.24 : Depths of penetration at 500 gf.
(Clearfil, six months)**

SAMPLE	DISPLACEMENT (μm)					
C1	32	17	29	17	18	23
C2	25	26	24	19	28	21
C3	29	23	26	19	22	18
MEAN = 23				S.D. = 5		

**Table 4.25 : Depths of penetration at 800 gf and 20 gf.
(Isosit, seven days)**

SAMPLE	DISPLACEMENT (μm)											
	800 gf						20 gf					
H1	48	45	38	35	38	44	24	17	17	16	18	20
H2	37	39	40	41	53	46	18	18	14	19	19	24
H3	48	44	37	35	38	50	17	20	19	18	19	28
MEAN	42						19					
S.D.	5						3					

**Table 4.26 : Depths of penetration at 800 gf and 20 gf.
(Brilliant, seven days)**

SAMPLE	DISPLACEMENT (μm)											
	800 gf						20 gf					
B11	29	29	29	29	24	28	14	17	22	23	16	17
B12	34	28	29	27	29	28	19	12	18	19	15	11
B13	33	28	35	25	25	25	17	11	23	16	18	17
MEAN	29						17					
S.D.	3						4					

**Table 4.27 : Depths of penetration at 800 gf and 20 gf.
(Clearfil, seven days)**

SAMPLE	DISPLACEMENT (μm)											
	800 gf						20 gf					
C11	30	26	25	24	28	24	13	18	13	8	13	12
C12	34	23	22	22	27	22	26	9	10	9	8	10
C13	33	28	28	30	22	21	20	16	18	14	13	13
MEAN	26						14					
S.D.	4						5					

**Table 4.28 : Depth of penetration at 800 gf and 20 gf.
(Isosit, six months)**

SAMPLE	DISPLACEMENT (μm)											
	800 gf						20 gf					
12	66	43	38	40	40	55	35	18	15	20	17	28
21	47	42	42	31	35	44	22	21	24	16	17	14
23	38	45	43	37	31	35	18	28	22	22	17	19
MEAN	42						21					
S.D.	8						5					

Table 4.29 : Depth of penetration at 800 gf and 20 gf.
(Brilliant, six months)

SAMPLE	DISPLACEMENT							(μm)						
	800 gf							20 gf						
B1	51	38	35	33	27	37		32	23	22	22	18	19	
B3	51	33	30	30	31	30		26	26	14	16	15	14	
B5	30	27	30	30	23	26		16	14	17	15	12	13	
MEAN	33							19						
S.D.	8							5						

Table 4.30 : Depth of penetration at 800 gf and 20 gf.
(Clearfil, six months)

SAMPLE	DISPLACEMENT (μm)											
	800 gf						20 gf					
C4	41	23	37	22	22	28	33	17	30	20	15	26
C5	33	34	31	25	34	26	24	25	25	20	31	24
C6	45	29	33	26	27	24	34	22	32	17	25	15
MEAN	30						24					
S.D.	7						6					

CHAPTER FIVE - DISCUSSION

PART I - MATERIALS

5.1 Filler Content

Heating composites to 575°C to determine filler content has been the conventional method used by many researchers (Ruyter and Sjovik 1981; Pilliar et al. 1986; Hosoda et al. 1988). The TG results of Brilliant and Clearfil showed that the organic components of these materials were burnt out by 600°C and the filler contents, 75 percent (Brilliant) and 86 percent (Clearfil), corresponded closely to the values provided by the manufacturer. However, the mass decrease continued to a higher temperature with Isosit and the filler content 72 percent, determined at 600°C was less than the value claimed by the manufacturer, 74 percent (Ivoclar 1988).

The continuing mass decrease of Isosit at higher temperatures may indicate the breakdown of ytterbium, which is present in significant amounts (Hosoda et al. 1988; Pennycook 1990) and has a melting point of 824°C. However, this may also indicate the difficulty of burning the polymer phase, particularly the prepolymerised particles present in Isosit (Hosoda et al. 1988) which are likely to be highly cross linked. The rate of degradation of polymers is generally rapid at temperatures above about 400°C. At such temperatures the pyrolysis products may be entirely volatile. However in some cases, notably cross linked polymers, much of the main chain carbon is incorporated in an involatile char. Such carbonaceous residues resist much higher temperatures as the surface chars have low thermal

conductivities and may protect the underlying material from the effects of heat for a long time (Hall 1981). Further development of the TG method appears to be useful in characterising the structure of dental composites.

It is known that the loss of HCl from poly(vinyl chloride) is observed as a separate step, usually at about 250°C, during decomposition using TG analysis (Billmeyer 1971; Hall 1981). The present TG analysis of Brilliant and Clearfil showed a two stage breakdown which appears to reflect the presence of co-monomer (TEGDMA) in the polymer matrix. Therefore, the TG method will also be useful in the study of copolymers used in dental composites.

The large exothermic peak at about 95°C on the DTA curve of Isosit specimens containing N-Liquid indicated rapid decomposition of the initiator. Therefore, polymerisation of Isosit is possible at a lower temperature than that recommended (120°C) by the manufacturer. The complex nature of the DTA curves between about 300 and 600°C represents depolymerisation and subsequent repolymerisation of the monomer fragments.

5.2 Processing Conditions

There is clearly a disparity between the three materials in terms of the conditions under which they are processed.

For the conditions recommended for Isosit (120°C, 10 minutes) the timing of the processing cycle did not start until this processing temperature was reached. Consequently, the total processing time, from the start of heating to the end of

processing was significantly longer, 17 minutes. Also, when the Ivomat was not allowed to cool between processing procedures the heating rate increased and the total processing time decreased. As temperatures within the Ivomat were greater than 120°C it can be more correctly stated that Isosit is heated to a temperature range between 120 and 128°C and held in this temperature range for 10 minutes.

The manufacturer of Brilliant claims that a processing temperature as high as 110°C is reached. Clearly this was not the case as the maximum temperature reached in the processing time was 89°C and the average temperature at which the material was held was about 75°C. The most consistent heating temperature, 100°C, was obtained with Clearfil as a dry laboratory oven preheated to 100°C was used. However, this should be confirmed with the dry heating oven supplied with this material.

The processing time was shortest in Brilliant (8.5 minutes) and much longer for Clearfil (15 minutes).

The effect of pressure on the inlay materials is an area that has not been investigated. An obvious benefit of pressure would be the reduction in porosity of the specimens (Pennycook 1990). However, the effect of pressure on the structure and physical properties has not been clearly established.

PART II - Dimensional Accuracy

5.3 Impression Taking and Die Fabrication

The effect of impression taking on the dimensional accuracy of Isosit specimens was studied by comparing the percentage linear dimensional change of specimens prepared from impressions taken at 37°C and at ambient temperature. The difference obtained (0.3 percent) can be attributed to the thermal contraction of the impression material from 37 to 20°C, and confirms the results of previous studies (Peppit and Earnshaw 1981; Peppit 1984) which have demonstrated that the polymerisation shrinkages of addition-cured silicone impression materials were much less than 0.1 percent in most of the materials tested and they exhibited high coefficients of thermal expansion ($136-156 \times 10^{-6}/^{\circ}\text{C}$). The comparison of the linear percentage dimensional change of specimens processed in stone dies prepared from an impression taken at 20°C with that of specimens directly processed in the metal die showed there was no significant difference in the linear percentage dimensional change (0.9 percent). This also confirms that the thermal contraction rather than polymerisation shrinkage of the impression material is a contributing factor to the accuracy of composite inlays. It is important to remember, however, that the polymerisation shrinkage of polysulphide and condensation-cured silicone impression materials is relatively large, 0.2-0.5 percent (Peppit and Earnshaw 1981).

At the recommended processing temperature the stone specimen lost its compressive strength significantly (Figure 4.9). The significant loss of strength is due to dehydration of calcium sulphate dihydrate (gypsum) to hemihydrate (Hasratininsih

1987). As there was no difference in the linear percentage dimensional change (0.9 percent) of specimens processed in stone dies prepared from an impression taken at 20°C with that of specimens directly processed in the metal die, it can be concluded that breakdown of the stone die will not significantly affect dimensional accuracy of inlays. However, the effect of this on the accuracy of Class II and III inlays requires further investigation because of the complexity of their shape and the direction of polymerisation shrinkage forces.

A second pour die is required for the finishing of Isosit because of the weakening of the stone die during the processing. There was no significant difference in the linear dimensional change of specimens processed in either the first or second pour dies indicating that this procedure did not affect the dimensional accuracy of specimens.

In conclusion, the dimensional change during the impression taking and stone die fabrication is attributed to the cooling of the impression material, which was about 0.3 percent in this study.

5.4 Polymerisation Shrinkage

The mean percentage linear polymerisation shrinkages, 0.9 percent (Isosit), 0.1 percent (Brilliant) and 0.3 percent (Clearfil) indicate that although polymerisation shrinkage is the major factor affecting the dimensional accuracy of Isosit specimens, it is extremely small with Brilliant. The effects of impression and polymerisation shrinkage were equal for Clearfil.

The large difference in polymerisation shrinkage between Isosit and the other two materials may reflect the lower filler content of Isosit. However, other researchers have shown a poor correlation between filler content and linear polymerisation shrinkage (Asmussen 1975). In fact, Clearfil containing 86 percent filler gave a higher shrinkage value (0.3 percent) than Brilliant (0.1 percent) which had a filler content of 75 percent. Therefore, several other factors may be influencing this.

Isosit is a urethane dimethacrylate based polymer whereas the other two materials are Bis GMA based polymers diluted with TEGDMA (Section 3.1). Although the type of monomer has little effect (Bayne 1989), the presence of a diluting co-monomer will influence polymerisation shrinkage because of the larger size of the dilutant molecules (Asmussen 1975) and its effect on the viscosity of the material (Cowperthwaite et al. 1979; Dulik et al. 1981; Munksgaard et al. 1987). The amount of TEGDMA is 5 percent in Brilliant while that in Clearfil is estimated to be similar to Clearfil photoposterior which is highly diluted, 15 percent (Hosoda 1989).

The size and shape of filler particles and its effect on the viscosity of the material are also important considerations (Munksgaard et al. 1987). Isosit is claimed to be homogenously microfilled (Ivoclar 1988) but other studies have shown that it contains prepolymerised particles (Hosoda et al. 1988; Pennycook 1990). Both Brilliant and Clearfil are hybrid composites (Hosoda et al. 1988), the latter having a slightly larger particle size (Hosoda et al. 1988).

Isosit is cured by heat whereas the other two materials are initially light cured. Heat curing will generally achieve a higher degree of polymerisation than light curing (Bayne 1989)

and therefore a higher degree of polymerisation shrinkage may result. In light cured specimens the surface is cured first which makes the apparent shrinkage small.

Isosit is also unique in that it is cured under pressure and this reduced porosity in the material (Pennycook 1990). Brilliant and Clearfil were not cured under pressure and the inherent porosity may decrease the apparent shrinkage by a change in the size of the internal voids rather than a change in the overall specimen size.

The cement space provided by polymerisation shrinkage, 23 μm (Isosit), 3 μm (Brilliant) and 8 μm (Clearfil) is inadequate particularly in relation to the latter two materials; including the contribution during impression taking and stone die fabrication (0.3 percent), the cement space obtained will be 31 μm (Isosit), 11 μm (Brilliant) and 16 μm (Clearfil). A systematic way of compensating, considering shrinkage of impression material, expansion of the stone die and polymerisation shrinkage is necessary to provide adequate cement space. Use of die spacer has been widely accepted for this purpose in small metal castings (McAloon 1989).

PART III - DIMENSIONAL STABILITY

5.5 Water Sorption

The percentage mass of water absorbed by the inlay materials after six months immersion, 1.2 (Isosit) and 0.7 percent (Brilliant and Clearfil), falls within the lower end of the range of values for composite filling resins (Pearson 1979; Fan et al. 1985). The higher water sorption value for Isosit

may be due to its lower filler content as absorption occurs primarily by diffusion through the resin matrix (Braden et al. 1976; Braden and Clarke 1984). Pearson (1979) suggested that urethane dimethacrylate based polymers may be more hydrophillic than Bis GMA based polymers which may contribute to this increased value. However, Ohtani (1987) recently gave water sorption values, in percentage mass, of 2.36 ± 0.23 for urethane dimethacrylate and 2.48 ± 0.45 for Bis GMA. Ohtani's measurement was based on a method developed earlier by Hirasawa et al. (1983) which used an electronic micrometer. They found a direct correlation between the mass of absorbed water and linear expansion of dental composites, and this relationship was confirmed with binary copolymers by Ohtani. She concluded that the expansion measurement was suitable for estimating the amount of water absorbed, as the expansion was less affected by the elution from the composites than the water sorption measurement.

Isosit absorbed water rapidly, reaching a value approaching its maximum within seven days immersion. In contrast, both Brilliant and Clearfil absorbed water more slowly achieving approximately half their six month values at seven days.

The effect of water absorption on dimensional stability became increasingly evident. The dimensional changes at thirty days, 0.41 (Isosit), 0.13 (Brilliant) and 0.17 (Clearfil) were further increased to 0.52, 0.31 and 0.30 respectively, at six months. From three to six months there was no further gain in the percentage mass of water absorbed. As Ohtani suggests, the measurement of dimensional change is a sensitive way of estimating the amount of water absorbed. However, a further change in shape may accompany stress relaxation (Feilzer et al. 1990).

The linear dimensional changes are consistent with results reported for composite filling materials (Bowen et al. 1982). In this study the authors commented that hygroscopic expansion of composite filling materials was favourable as it would compensate for the polymerisation shrinkage. Although the expansion due to water sorption was not as great as the polymerisation shrinkage in Isosit, it was greater than the polymerisation shrinkage in both Brilliant and Clearfil. This dimensional change occurs slowly over a period of months and is outside the control of the practitioner. Any dimensional discrepancy initially present with composite inlays will be filled by the luting cement. Therefore, such an uncontrollable dimensional change occurring in the inlays is undesirable and may in fact stress both cement and tooth structure. Expansion due to water sorption is therefore a major factor which detracts from the dimensional stability of composite inlays.

5.6 Thermal Expansion

For the temperature range 20 to 65°C it can be seen that the coefficient of thermal expansion of Isosit ($31 \times 10^{-6}/^{\circ}\text{C}$) is the highest followed by Brilliant ($25 \times 10^{-6}/^{\circ}\text{C}$) and Clearfil ($21 \times 10^{-6}/^{\circ}\text{C}$). This closely reflects the differences in filler content of these materials, 69 (Isosit), 75 (Brilliant) and 86 (Clearfil) percent. The latter is probably the highest found in dental composites currently available (Bryant and Mahler 1986; Ruyter and Oyased 1987). Despite this, the coefficient is still considerably higher than human tooth structure ($11.4 \times 10^{-6}/^{\circ}\text{C}$) reported by Souder and Peters (1920) and Souder and Paffenbarger (1942). A thermal expansion coefficient of $20\text{--}30 \times 10^{-6}/^{\circ}\text{C}$ would result in a linear dimensional change of 0.122 to 0.183 percent over the 4 to 65°C

temperature range. Although this is the lowest among the dimensional changes studied in this present study, it is equivalent to a dimensional change of 10 to 15 μm for an inlay having the diameter of 8 mm adopted for the present study.

Decreasing the curing temperature of Isosit from 120 to 100°C resulted in a significant increase in the coefficient of thermal expansion to $35 \times 10^{-6}/^{\circ}\text{C}$ for the range 20–65°C (Table 4.17). The thermal expansion curve was also characterised by a marked plateau (Figure 4.13). It is most likely that the plateau is a result of continued polymerisation of the specimen counteracting the thermal expansion. The incomplete polymerisation is not due to the lowered temperature but due to the short processing time, as the plateau was not evident when the processing time was prolonged to 20 minutes.

Below this plateau the coefficients of thermal expansion are significantly higher than those obtained from specimens processed at either 120°C or 100°C (20 minutes). The lower degree of polymerisation and increased monomer content of these specimens is likely to be responsible for the increased coefficient of thermal expansion. In this regard, it is interesting that the overall expansion coefficients are lower in the specimens processed at 100°C for 20 minutes. Polymerisation of Isosit is possible at a temperature lower than 100°C (Section 4.1) and a longer curing time at a lower temperature may enhance the degree of polymerisation.

Miyazaki et al. (1984) attributed a similar plateau on the thermal expansion curve of a composite filling material to a glass transition temperature. However, on the basis of the results obtained in the present study this seems unlikely; glass transition temperatures of all three inlay materials

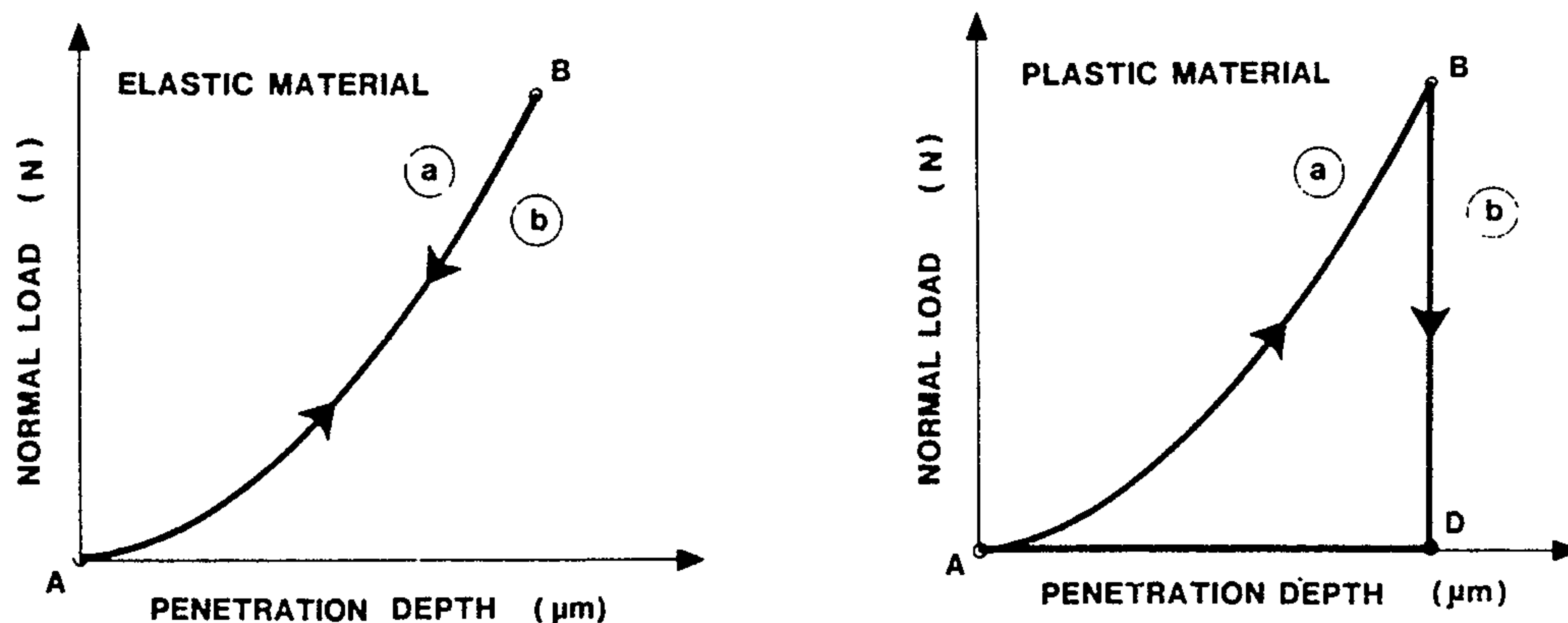
were represented by a sudden drop in the thermal expansion curve above 290°C, although this temperature is recorded under dynamic heating conditions and should be considerably higher than that measured under static conditions. However, this is markedly higher than the range of glass transition temperatures of 87-120°C for conventional posterior composite filling materials (Greener and Bakir 1986) and is consistent with other studies that have shown that raising the curing temperature will increase the glass transition temperature (Wu an Yu 1985).

5.7 Hardness

Conventional microhardness testing of composite resins uses a Knoop indenter to overcome the problems of distortion of the residual indentation by elastic recovery (Phillips 1990c). However, the published Knoop harness data for these three composite inlay materials is inconsistent (Hosoda et al. 1989; Inoue et al. 1989; Takahashi et al. 1989). A Rockwell hardness test with a spherical ball 2 mm in diameter has been used (Dennison and Craig 1972; Greener et al. 1984; Wendt 1987b). However, because of the geometry of the indenter a much higher indenting force, 15 to 45 kg, is usually required and this may create problems with fracturing of small test specimens.

The geometry of the Vickers indenter allows for a relatively small loading force of up to 1 kg, giving a deeper indentation covering a greater surface area than the Knoop indenter. This will reduce the influence of filler particle size and surface structure on the hardness test results. Continuous measurement of the depth of indentation and indenting load during loading and unloading not only eliminates the problems associated

with elastic recovery but also measures the recovery process at the same time. A plot of load versus displacement of the indenter tip will provide an indentation curve for the material (Loubet et al. 1986). Hypothetical indentation curves for elastic and rigid plastic materials (Loubet et al. 1986) are shown below.



The indentation curve of an elastic material will have transposed loading (a) and unloading (b) components as elastic recovery of the material maintains force on the indenter tip during unloading. In contrast, the indentation curve of a rigid plastic material will have separate loading (a) and unloading (b) components as there is no elastic recovery of the material and the force on the indenter becomes instantly nil at the point of unloading (B). The indentation curves of composite inlay materials in this study were similar to other elastoplastic materials (Loubet et al. 1986) with separate loading and unloading components indicating both elastic recovery and permanent plastic deformation.

The Vickers hardness number of Isosit after seven days immersion, 42, was slightly lower than the value provided by the manufacturer, 50 to 60 (500 gf for 30 seconds, Ivoclar 1988), but was consistent with the range of Vickers number, 43

to 61 (Hirasawa et al. 1975) for macrofilled composite filling materials with a range of filler content similar to Isosit, 70 to 78 percent by mass (Dennison and Craig 1972, Hosoda et al. 1987a). The hardness values for Brilliant (86) and Clearfil (117) reflected the increased filler content of these materials, 75 and 86 percent respectively (Section 4.1).

The decrease in hardness of Brilliant (66) and Clearfil (72) after six months immersion may indicate early degradation of the materials. In contrast the hardness of Isosit remained unchanged after six months. Both Brilliant and Clearfil are based on Bis GMA and contain TEGDMA. The latter has a hydrophillic bond in the ethylene glycol unit predisposing the composite to breakdown due to hydrolysis (Hino and Arai 1989; Fuji and Arai 1990). As Soderholm (1981, 1983) has shown the water reaches to the interface between the filler particles and matrix and will break the silane bond.

The percentage elastic recovery of the three materials after seven days immersion (41 to 55 percent) was less than the value given by Dennison and Craig (1972), 70 to 87 percent. However, the results are not comparable as they used a Rockwell hardness test with a load 30 kg and recovery was measured ten minutes after loading.

The elastic behaviour of polymers reflects a deformation of the structure on a molecular level. Elastic properties are imparted by cross linking which suppresses viscoelastic behaviour and allows coiled polymer chains to translate past each other reversibly when deformed (Kinloch and Young 1983; Sperling 1986a). This will be further modified by reinforcing fillers which form a variety of physical and chemical bonds with the polymer chains permitting local chain movement but restricting actual flow (Sperling 1986b).

Therefore, the reduction in the percentage elastic recovery of Clearfil, 46 to 20 percent, after six months immersion indicates structural changes occurring in both cross linking of the polymer and filler-matrix bonding. Soderholm and Roberts (1990) looked at the effect of hydration and dehydration on the tensile strength of composite filling materials and concluded that absorbed water had a plasticising effect on the resin matrix. Other studies (Zui and Arai 1986; Matukawa and Arai 1988; Fuji and Arai 1990) have shown a decrease in tensile and transverse strength associated with surface degradation and the appearance of by-products from the degradation of the filler matrix bond after three to six months immersion. Similarly, analysis of eluted material has shown that stress corrosion and elemental leaching may result in breakdown of the silica network of the glass (Charles 1958; Soderholm 1983).

The lack of change in elastic recovery of Isoosit and Brilliant after six months immersion may be a reflection of structural differences between these materials, such as the presence and concentration of copolymer, the degree of cross linking and silane treatment. It is important that the present hardness test has recorded an early change (6 months) in elastic recovery as other studies of composite filling materials have shown a reduction in the tensile and transverse strengths over a much longer duration of three years (Zui and Arai 1986; Matukawa and Arai 1988; Fuji and Arai 1990).

The elastic recovery of a composite will provide an indication of its ability to withstand permanent deformation and therefore may influence the wear performance of the material. Previous studies (Moser and Greener 1972, 1973; Harrison and Draughan 1976; McKinney and Wu 1985) have shown a poor correlation between in vitro and in vivo wear performance and

physical properties such as hardness. It is doubtful whether the instantaneous and delayed elastic recoveries and permanent deformation have been differentiated in the conventional hardness tests.

The extension of the concept of hardness tests such as the one developed in the present study may elucidate a relationship between elastic performance and wear performance of dental composites which is the major limitation of current dental composites (Ekfeldt and Olivo 1990) and may in fact be the most significant factor affecting their dimensional stability.

CHAPTER SIX - SUMMARY AND CONCLUSIONS

The dimensional accuracy and stability of composite inlays has been studied by developing a stainless steel die to assess the dimensional changes occurring during impression taking and fabrication, and after fabrication.

Three composite inlay resins, Isosit, Brilliant and Clearfil, representing a cross section of the materials available were studied. There was clearly a disparity between the three materials in terms of the conditions under which they were processed; Isosit was cured under heat and pressure up to about 130°C whereas Brilliant and Clearfil were light cured and then post cured with dry heat up to 90 and 100°C respectively.

There was a net reduction in inlay dimensions during the fabrication for all three materials. The percentage linear dimensional changes are summarised below.

Dimensional Accuracy

MATERIAL	ISOSIT	BRILLIANT	CLEARFIL
THERMAL CONTRACTION OF THE IMPRESSION	-0.30 %	-0.30 %	-0.30 %
POLYMERISATION SHRINKAGE	-0.90 %	-0.12 %	-0.30 %
TOTAL DIMENSIONAL CHANGE	-1.20 %	-0.42 %	-0.60 %

Polymerisation shrinkage was the major factor affecting the dimensional accuracy of Isosit. In contrast, thermal contraction of the impression was the major factor affecting the dimensional accuracy of Brilliant. Both factors equally affected the dimensional accuracy of Clearfil.

In considering the 8 mm diameter inlay studied here, these linear dimensional changes will result in a cement space of 31 μm for Isosit, 11 μm for Brilliant and 16 μm for Clearfil. These spaces are too small and conventional compensation techniques such as the use of die spacer are required to allow for more space for cementation.

There was a net increase in inlay dimensions after fabrication for all three materials. The percentage linear dimensional changes are summarised below.

Dimensional Stability

MATERIAL	ISOSIT	BRILLIANT	CLEARFIL
EXPANSION DUE TO WATER SORPTION	0.52 %	0.31 %	0.30 %
THERMAL EXPANSION OF THE INLAY (5-50°C)	0.14 %	0.11 %	0.09 %
TOTAL	0.66 %	0.42 %	0.39 %

Water sorption is the major factor affecting the dimensional stability of these materials. In contrast to gold and ceramic inlay materials, the magnitude is significant and clinically undesirable because it occurs after cementation of the inlay, continuing over the six month duration of this study, and is

outside of the control of the dentist. Furthermore the long term dimensional stability associated with wear and breakdown due to water sorption is questionable.

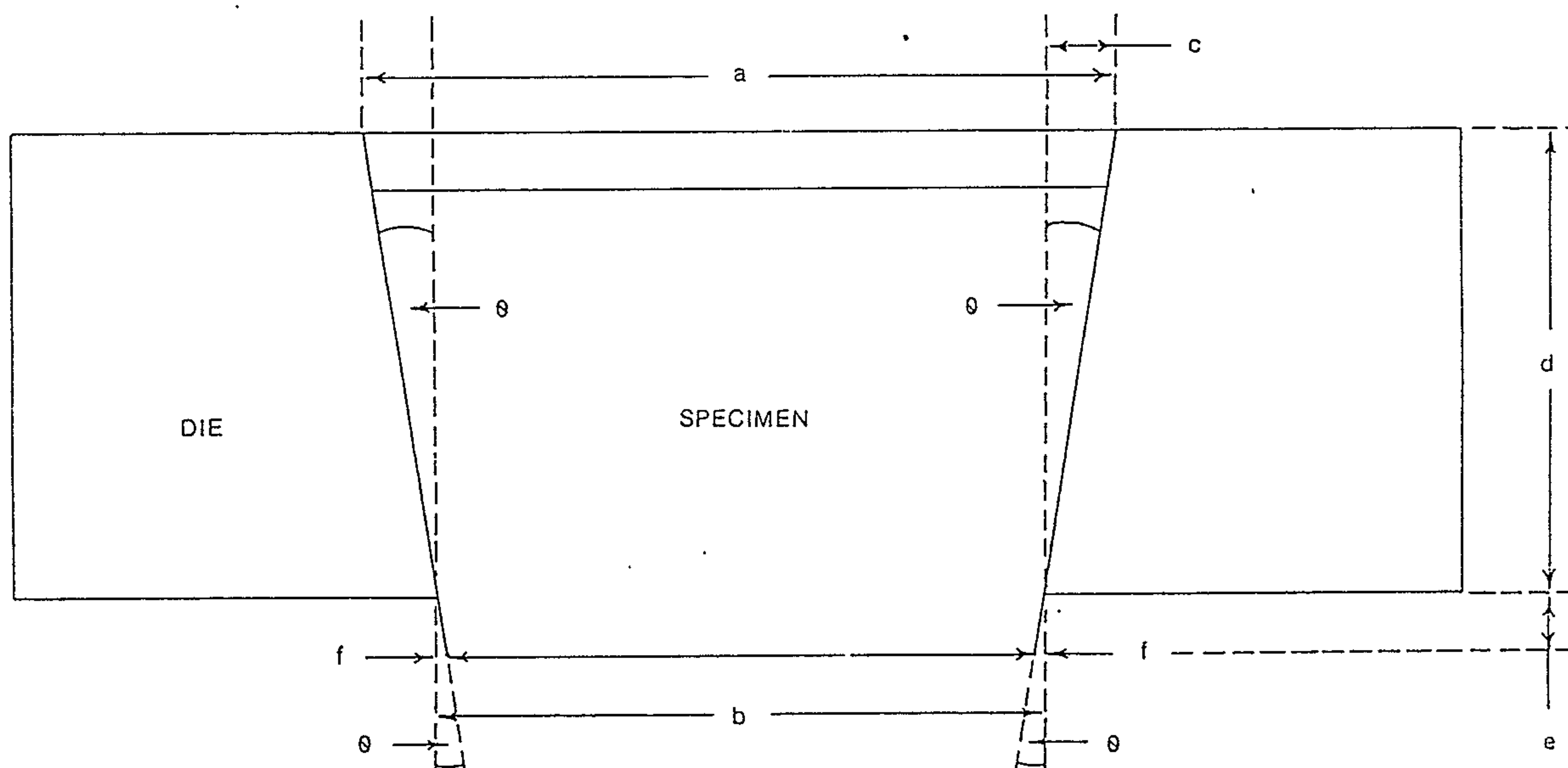
A hardness testing device was developed to continuously measure the applied load and depth of indentation during loading and unloading of specimens. The accuracy of this device was verified using a standard metal specimen and indentation depths were within 1 μm of the known hardness values. The method also allowed for the measurement of the elastic recovery of specimens.

The Vickers hardness number for each material; Isosit, 42, Brilliant, 86, Clearfil, 117, reflected the difference in filler content determined by thermogravimetric method for these materials; 69, 75 and 86 percent respectively.

The hardness testing method also provided a sensitive measure of structural changes occurring as the result of water sorption. There was a significant decrease in the hardness of all three composite inlay resins and a significant decrease in the elastic recovery of Clearfil after six months immersion.

The importance of these changes in relation to the degradation of these materials requires further investigation. The further development of the hardness tester will enable such a study and a relationship between structural changes and in vivo and in vitro wear performance of composite inlay and filling resins may be better understood.

Wear and degradation of the restorations is perhaps the most important factor to be considered in the assessment of the dimensional stability of composite inlays as it will largely determine the clinical success of the restorations. Water sorption will play an important role in their degradation and at present it is probably the major limitation of these materials.

APPENDIXA-1. DIAGRAM OF THE GEOMETRIC RELATIONSHIP BETWEEN INLAY PROTRUSION AND LINEAR DIMENSIONAL CHANGE.

A-2. DERIVATION OF THE FORMULA FOR CALCULATION OF PERCENTAGE
LINEAR DIMENSIONAL CHANGE OF INLAYS.

CONVERGENCE

$$T = \frac{d}{2 \times c} \quad \text{however} \quad c = \frac{a - b}{2}$$

$$T = \frac{d}{a - b}$$

$$\tan \theta = \frac{c}{d} = \frac{f}{e}$$

$$f = \frac{c \times e}{d} \quad \text{however} \quad c = \frac{a - b}{2}$$

$$f = \frac{\frac{a - b}{2} \times e}{d}$$

LINEAR DIMENSIONAL CHANGE

$$S = 2 \times f$$

$$S = 2 \times \frac{a - b}{2} \times \frac{e}{d}$$

$$S = \frac{(a - b) \times e}{d} \quad \text{however} \quad T = \frac{d}{a - b}$$

$$S = \frac{e}{T}$$

PERCENTAGE LINEAR DIMENSIONAL CHANGE

$$\% S = \frac{S}{b} \times 100 = \frac{e}{T \times b} \times 100$$

B. RESULTS OF PRELIMINARY EXPERIMENTS TO DETERMINE THE MOST
SUITABLE LOADING CONDITIONS FOR PROTRUSION MEASUREMENT

MEAN PERCENTAGE PROTRUSION ON STANDARD DEVIATION
FOR LOADS APPLIED FOR 30 SECONDS

	300 g	500 g	1 kg	2 kg
12	10.2	10.4	3.5	6.5
13	5.6	2.7	5.0	5.5
14	6.3	3.6	3.4	2.9
15	3.0	6.2	4.7	4.1
16	6.2	8.1	4.9	5.0
17	2.1	4.7	2.9	2.8
Mean	5.6	6.0	4.1	4.5
(S.D.)	2.9	2.9	0.9	1.5

MEAN PERCENTAGE PROTRUSION ON STANDARD DEVIATION
FOR LOADS APPLIED FOR 60 SECONDS

	300 g	500 g	1 kg	2 kg
12	5.1	4.2	5.1	1.6
13	3.9	7.5	4.3	5.8
14	4.1	2.6	3.7	2.4
15	4.8	3.5	4.8	4.2
16	6.0	4.5	5.2	4.6
17	2.0	3.7	3.2	1.8
MEAN	4.3	4.3	4.2	3.4
(S.D.)	1.4	1.7	0.8	1.7

The 1 kg load applied for 30 seconds and the 2 kg load applied for 60 seconds provided the most reproducible measurements of protrusion. Although there was no significant difference ($p>0.05$) between these loading conditions, the 2 kg load for 60 seconds was used in the measurements of dimensional accuracy as it had the lowest mean value.

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