



**THE EFFECTS OF MANIPULATIVE VARIABLES ON THE
HARDENING OF DENTAL AMALGAMS**

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DECLARATION

To the best of the author's knowledge this thesis does not contain any material which has been submitted for any other degree or award in any university except where due reference is made in the text or common knowledge is assumed.

If accepted for the award of the degree, the author consents to the thesis being made available for photocopying and loan.

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SUMMARY

The objective of the present research was to investigate the response of three commercial spherical-particle, high-copper amalgams to variations in trituration time and condensation pressure. For comparison the behaviour of a commercial lathe-cut alloy of conventional composition and two commercial admixed alloys, which contained both spherical and lathe-cut particles, were investigated. The response was evaluated in terms of dimensional change during hardening of the amalgam, compressive strength and residual mercury content.

Each of the parameters used in the study indicated that amalgams made from alloys containing lathe-cut particles were more sensitive than spherical particle alloys to changes in the manipulative variables, trituration time and condensation pressure. Amalgams containing non-spherical particles also required higher condensation pressures to achieve satisfactory condensation.

The accurate measurement of dimensional changes which occur during the hardening of dental amalgam was made possible by a new design of dilatometer. With this computerised instrument the stress applied to the test specimen was only 28Pa and the design was such that it was possible to commence measurements of dimensional change within 2 minutes and 45 seconds from the end of trituration. This enabled the very early stages of the hardening reaction to be recorded.

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CHAPTER 1

INTRODUCTION

1.1 Historical Background

Although mercury based alloys, or as they are better known, dental amalgams, were being used in China as restorative materials as early as the seventh century A.D.(Chu Hsi-T'ao 1958), they were not introduced into Europe until 1818, when Regnart, a Frenchman, modified D'Arcet's Mineral Cement. D'Arcet's Mineral Cement consisted of eight parts bismuth, five parts lead, three parts tin, and one part mercury which was added to lower the melting point. This material became liquid at 100°C and was poured directly into the cavity. Regnart found that by the addition of mercury, the fusion temperature of the alloy, and therefore the level of pain suffered by the patient, could be lowered.

In 1826 another Frenchman, Traveau, advocated the use of paté d'argent (silver paste), a mixture of pure silver and mercury. The advantage of using paté d'argent for permanent fillings was that it could be inserted into teeth at room temperature. Prior to the introduction of anaesthesia, any technique which reduced the level of pain suffered by the patient had great public appeal. Consequently, this painless method of filling teeth was quickly seized upon by dental practitioners and profiteers who used the amalgam with little regard to recommended clinical practice.

The most notorious of the profiteers were the Crawcour brothers, who having acquired a superficial knowledge of dentistry in France, emigrated to England and called themselves surgeon-dentists. In

England they were financially successful with their Mineral Succedaneum (actually shavings from French silver coins mixed with mercury), which they claimed could be used to painlessly fill a tooth in about two minutes. From England two of the Crawcour brothers travelled to America where they added the prefix "Royal" to their Mineral Succedaneum and extensively advertised the benefits of their painless method for filling teeth. Their method was very appealing when compared with the plugged gold fillings used by the best American dentists at the time. However, it soon became apparent that their painless technique simply involved placing mounds of amalgam on occlusal surfaces without removing any diseased tissue and with no regard to dental anatomy. The clinical performance of these first amalgams was quite disastrous due mainly to the fact that they underwent a large expansion as they hardened with resulting serious damage to the filled teeth; this led to the withdrawal of amalgams by the American Society of Dental Surgeons in 1845. Members of that organisation were required to pledge, under threat of expulsion, to refrain from using mercury-based restorative materials.

In 1853 Flagg began the first scientific investigation into amalgam by conducting laboratory tests and clinical trials on an amalgam alloy containing 60% silver and 40% tin. Of far greater importance, however, was the work of G.V. Black, published in the first of a series of papers in 1895 (Black 1895a,b,c: 1896a,b: 1897). With carefully controlled experiments using alloys of silver and tin with compositions in the range 42-75% silver, 30-58% Sn, Black observed that these alloys sometimes expanded and sometimes contracted during setting. He also realised that these dimensional changes were of clinical significance and were influenced by such factors as alloy

composition and heat-treatment, particle size, mercury to alloy ratio, size of the filling and packing procedure. He carried out extensive laboratory tests on dimensional change and compressive strength. On the basis of these findings, together with his clinical observations over a period of 40 years, Black was able to make recommendations not only on the clinical procedures which should be adopted eg. cavity design, amalgam mixing and packing, but also on metallurgical factors eg. alloy composition, heat-treatment and mercury-to-alloy ratio. This work resulted in the reacceptance of amalgam as a restorative material and the production, by SS White in 1900, of the first commercial alloy, True Dentalloy. This alloy was based on Black's balanced alloy 70%Ag, 30%Sn which showed a slight expansion during hardening. Many manufacturers were quick to copy Black's balanced alloy and in the early 20th century a diversity of alloys, each claiming unique superiority, were produced.

Souder and Peters from the American National Bureau of Standards set about sorting out and quantifying the physical properties of these alloys. Their published results "An investigation of the Physical Properties of Dental Materials" (Souder and Peters 1920) led to the production of standards and specifications for amalgams, particularly compressive strength and flow, setting and thermal expansion, and electrochemical potential. Test specimen dimensions were defined and a dental interferometer capable of measuring dimensional changes to $0.1\mu\text{m}$ designed. These well defined parameters were used to compare alloys and the effects of varying mercury to alloy ratio, trituration and condensation, and eventually led to the issue of the American Dental Association Specification No 1 in 1929.

Although amalgam research was actively pursued for the next thirty years, amalgam alloy powders changed little in composition, heat-treatment and morphology, in turn due to the strict compositional guidelines contained in ADA Specification No 1. This was in turn due to a misinterpretation of Gayler's work on the silver-tin phase diagram (Gayler 1935), which resulted in the belief that an addition of 15-20% copper would cause amalgams to expand excessively on setting. This proved subsequently not to be the case but early concern over the possibility of excessive expansion led to the application by the ADA of a limit of 5% copper in amalgam alloy formulations.

Clinical improvements did occur however with the introduction of the mechanical triturator in the 1940's and the acceptance of the Eames (or dry) technique in the early 1960's (Eames et al 1961).

Demaree and Taylor (Demaree and Taylor 1962) were the first to study the effect of particle morphology on the mechanical properties of amalgam. They found that a spherical particle alloy with the same composition as conventional low-copper lathe-cut alloys (70.6% Ag, 26.1% Sn, 2.5% Cu) showed improved early strength but no long term improvement in physical properties. Their study resulted in the introduction of the spherical particle low copper commercial alloys Velvalloy and Spheralloy in the mid 1960's.

Allan, Asgar and Peyton (Allan et al 1965) and Wing (Wing 1966) using metallographic techniques investigated the phases present in hardened amalgams prepared from low-copper spherical and lathe-cut alloys. They showed that the microstructures of the fully hardened amalgams were similar, regardless of the shape of the original alloy

particles. It was suggested however, by several researchers (Koran and Asgar 1967, Eden and Waterstrat 1967 , Wing 1970) that low-copper spherical particle alloys had higher early strength (60 minutes), required less mercury and a lower condensation pressure to produce a set amalgam comparable with a very well manipulated lathe-cut alloy. The mechanical properties of the spherical particle alloys were also found to be less sensitive to manipulative variables such as trituration time and condensation pressure.

The first major departure from the composition of Black's balanced alloy resulted from the patenting of an admixed alloy, Dispersalloy, by Youdelis and Innes in 1963. This alloy, which was made from spherical silver-copper eutectic particles (28% Cu, 72% Ag) (Youdelis and Innes 1963) and conventional lathe-cut particles resulted in an amalgam with superior mechanical properties and corrosion resistance than those prepared from conventional alloys.

The success of Dispersalloy, and with it the acceptance by the ADA of high copper amalgams, led to numerous copies of the composition, but so as not to infringe the patent of Dispersalloy other manufacturers produced the alloy as a high copper single composition ternary alloy. These alloys have proven to be clinically superior to both lathe-cut and spherical alloys of conventional composition. The improved properties were attributed to the replacement of the soft and corrodable interconnected network of $\gamma_2(\text{Sn}_{7-8}\text{Hg})$, by a dispersion of $\eta'(\text{Cu}_6\text{Sn}_5)$. It has subsequently been proposed (Durandet 1989) that the $\epsilon(\text{Cu}_3\text{Sn})$ phase present in the original alloy particles acts as nucleation sites for the precipitation of rods of $\eta'(\text{Cu}_6\text{Sn}_5)$ from the solution of β and γ phases in mercury.

At present, dental amalgam alloys are produced with a variety of compositions and morphologies. These can be broadly categorized as lathe-cut low copper (conventional), lathe-cut high copper, spherical low copper, spherical high copper and admixed (a mixture of spherical or lathe-cut high copper and conventional lathe-cut).

These alloys are supplied in manufacturer-prepared capsules containing preweighed quantities of alloy and mercury, thus limiting the preparative variables controlled by the dentist to trituration time and condensation pressure, general guidelines for both of which are usually issued by the manufacturer.

Small variations in trituration time, readily achievable with modern trituration equipment, are sometimes made by dentists on the basis of practical experience of the influence which such variations have on the working time of amalgam. The question of the condensation pressure applied during the packing of an amalgam is, however, more complex and most dentists appear to rely on their training and experience to achieve what they consider to be an appropriate condensation pressure compatible with their own physical strength, the comfort of the patient and the observable response of the amalgam to the tooth cavity. Few, however, have any quantitative information on the condensation stresses which they apply in practice, in spite of the fact that over the last eighty years numerous methods for determining this important manipulative variable have been devised (eg. Black 1908, Ward and Scott 1932, Rakow et al 1978, Lussi and Buergin 1987).

The latter authors have suggested that condensation stresses considerably below the recommended 15MPa were employed by most

dental practitioners. Some of the consequences of applying less than optimum condensation, as reported by several researchers, (Peyton and Liatukus 1961, Hegdahl and Silness 1970, Ekstrand et al 1985) are increased porosity, increased residual mercury, reduced strength and increased setting expansion.

The effect of manipulative variables on the mechanical properties of spherical particle high copper alloys has received little attention in the literature (Clark et al 1981), although the effects on the other alloy types is well documented.

For example Ekstrand, Jorgensen and Holland (Ekstrand et al 1985) noted changes in porosity and dimensional stability as condensation pressure and trituration times were varied. They investigated four different commercial alloys, three of which were lathe-cut, the fourth an admixed lathe-cut and spherical particle alloy. It was concluded, on the basis of dimensional change measurements, that for these four amalgams decreased condensation stress resulted in increased expansion or decreased contraction during hardening; condensation stresses in excess of 14MPa produced the opposite trends.

Wing (Wing 1970) compared one hour and twenty four hour compressive strengths of five spherical particle low copper alloys with those of a conventional lathe-cut low copper alloy using "ten pound" and "three pound" condensation loads (unfortunately the diameter of the plugger in these experiments was not specified so the condensation stresses used cannot be determined). On the basis of these tests, Wing concluded that the strength developed by spherical low copper amalgams is generally unaffected by condensation "load"

whereas the early strength of the lathe-cut amalgam was greatly reduced in amalgams condensed with the lower condensation force. The 24-hour compressive strength ("fully set amalgams") of both spherical and lathe-cut amalgams was found to be independent of condensation load. Wing also reported that the dimensional change of spherical particle low copper amalgams was only slightly affected by both trituration and condensation.

1.2 Dimensional Change

As previously discussed, G.V. Black in 1895 reported observations on the dimensional changes which may occur during the setting of dental amalgam. For his studies Black not only used actual restorations, but he made laboratory measurements, using an optical microscope fitted with an eyepiece micrometer, on the width of the marginal gap produced by shrinkage of amalgam away from the walls of a cylindrical cavity machined into a steel block. Direct measurements of the change in length of a test cylinder of amalgam were also made using a dial plate micrometer. On the basis of these results Black compiled a list of factors that he considered were responsible for his observation that some preparations showed a net expansion by the time the hardening reaction had gone to completion while others showed a net contraction. Alloy composition, heat-treatment and particle size were the major manufacturing variables; trituration time, condensation pressure and mercury to alloy ratio the major manipulative variables controlled by the practitioner in the surgery. Manufacturers quickly responded to Black's recommendations based largely on his dimensional change results, and for the first time were able to produce dental alloys which did not suffer from ageing and

transportation, and gave satisfactory, reproducible clinical results.

Investigations by other workers following on from Black supported the view that composition, heat-treatment and manipulative techniques had a strong influence on these dimensional changes. But as Gray (Gray 1919, Gray 1922) and later Ward and Scott (Ward and Scott 1932) pointed out, the measuring procedures themselves had an influence on the dimensional changes so that the quantitative results obtained using instruments such as micrometers were shown to be suspect. In an attempt to minimise the influence of the measuring instrument on the dimensional change results, a method based on the interferometer principle was proposed by the United States Bureau of Standards in 1929. Using this technique Ward and Scott produced a series of dimensional change curves for a range of alloys, and found that each was sensitive to manipulative factors such as trituration and condensation. However, they had great difficulty in obtaining reproducible results, mainly because of their inability to control the trituration process, which they carried out by hand using a mortar and pestle. Rothen (Rothen 1931) showed that there was a substantial difference in dimensional change results obtained from experiments conducted at room temperature and those carried out at mouth temperature. With this in mind Gayler (Gayler 1933), using a counterbalanced lever dilatometer to minimise the interaction between the measuring device and the specimen, condensed specimens and measured their dimensional change at mouth temperature. Gayler had more success than Ward and Scott in obtaining reproducible results, primarily because she was better able to control condensation and trituration. Her results supported Ward and Scott's view of the importance of these manipulative variables.

With an air operated contactless micrometer, Fusayama (Fusayama et al 1964) was able to commence dimensional change tests immediately after condensation, ie. less than 5 minutes after the end of trituration rather than 15 minutes as specified by the ADA standards at the time. He found that conventional amalgams underwent a significant initial contraction in the first 10 minutes. This had been neglected in the standard tests. Early measurements thus indicated a greater initial contraction and smaller final expansion. Wing (Wing 1964) also warned about the effect on the dimensional changes of the technique adopted for making the measurements, for he was able to demonstrate that for a range of commercially available amalgam alloys, a slight increase in the load applied to the test specimen by the measuring instrument was sufficient to convert a net overall expansion into a net contraction. Similar trends were reported by Vrijhoeff (Vrijhoeff et al 1974), and Muench (Muench 1970) found that the shape of the contact point between the specimen and the measuring device could also affect the dimensional change result. The current technique specified by both the Australian and American Dental Standards Associations was developed by Mahler and Van Eysden (Mahler and Van Eysden 1972) and employs a linear variable displacement transducer which applies a load to the test piece of less than 1.7 gms. Although this technique has been specified, researchers (Darvell 1976, Espevik 1977, Kraft 1976) have continued to try to find faster, more accurate and more reliable methods of measuring dimensional change because of the considerable interest in the reaction mechanisms occurring in the first few minutes of hardening. The accurate monitoring of dimensional change during these initial stages yields information on these early mechanisms, and thus monitoring of dimensional change should commence as soon as

possible after the end of trituration.

As well as providing information on the reaction mechanisms, changes in dimension may have clinical importance and, indeed, commercial dental amalgams are required to conform to standards of dimensional change. These changes are normally measured using a test cylinder 10mm long prepared by compaction (or 'condensation') in accordance with widely accepted standardised procedures. Typical standards (Australian Standard Specification AS2110-1977, ISO/DIS 1559) require that the preparation of the test cylinder should be completed and the cylinder loaded into the dilatometer in times such that the first measurements of dimensional change are made five minutes after the end of trituration. The dimensional change after seven days should then not exceed $\pm 20\mu\text{m cm}^{-1}$.

1.3 Present Work

Although it has generally been asserted that spherical particle alloys are less sensitive to manipulative variables than either lathe-cut or admixed alloys there is little published evidence from carefully controlled studies to support this assertion. The present study was undertaken to assess the sensitivity of three commercial spherical particle high copper amalgams to variations in condensation pressure and trituration time. A commercial lathe-cut amalgam (New True Dentalloy) and two commercial admixed amalgams (Dispersalloy and Permite C) were included for comparison. The parameters studied in this investigation were compressive strength, final mercury content and dimensional change during hardening.

CHAPTER 2

MATERIALS AND EQUIPMENT

2.1 Materials

Six commercially available dental amalgams, listed in Table 1, were used in the investigation. In the case of the lathe-cut low copper alloy, New True Dentalloy, which was not precapsulated, trituration was carried out in a Lojic-type capsule and the recommended mercury:alloy ratio (1:1) was employed. With the other five alloys, capsules of alloy and mercury, as received from the respective manufacturers, were used.

Alloy	Manufacturer	Details
New True Dentalloy	S.S. White U.S.A.	lathe-cut, low copper
Permite C	Southern Dental Ind Aust	admixed
Dispersalloy	Johnson & Johnson U.S.A.	admixed
Tytin	Kerr Sybron Corp U.S.A.	spherical high copper
Lojic	Southern Dental Ind Aust	spherical high copper
Valiant	L.D. Caulk Co U.S.A.	spherical high copper

Table 1

2.2 Dimensional Change Equipment

As previously discussed, a number of different techniques have been employed by various researchers for measuring dimensional change of dental amalgam during hardening. Each had important limitations which prevented the true picture being obtained of the

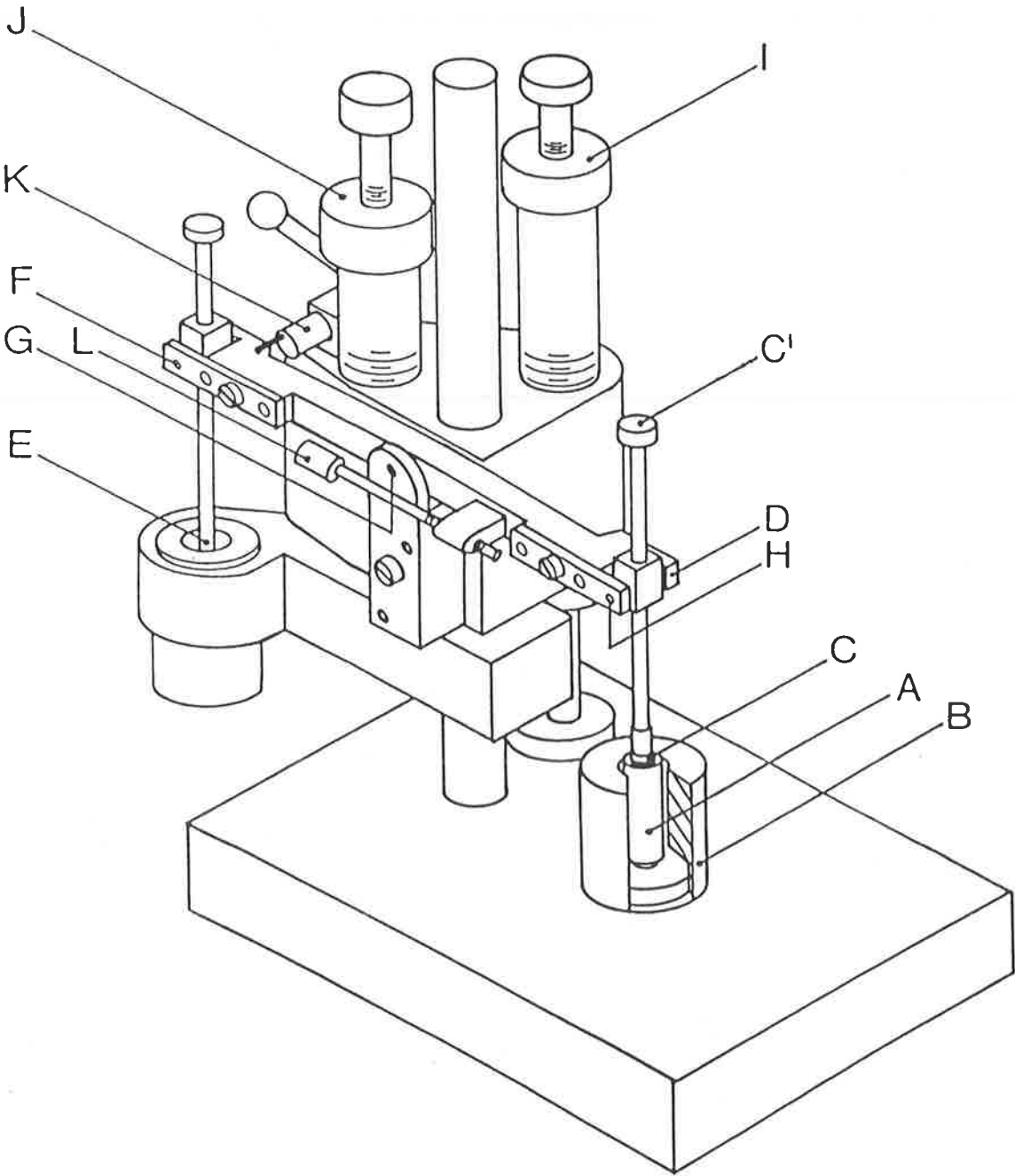
often complex way in which the dimensions of amalgams change during the hardening reaction.

To overcome these limitations a new dilatometer was designed to enable high precision measurements of dimensional change to be commenced within 45 seconds of the production of a standard test specimen of dental amalgam. A computerized data logging system permitted the length of the test specimen to be measured at nominated time intervals thereafter. Also, since Wing (Wing 1964) and Vrijhoeff (Vrijhoeff et al 1974) have reported that the load applied to the amalgam test specimen by the measuring system influences the magnitude of the dimensional change, this instrument was designed to permit its operation at specimen loads as low as 20mg (28 Pa), while maintaining sensitivity such that changes in length of the order of $\pm 0.01\mu\text{m}$ could be readily detected. The mechanical and electrical stability of the whole system were such that measurements of length of a 10mm long Invar test specimen over a period of 7 days showed variations of less than $\pm 0.01\mu\text{m cm}^{-1}$.

Construction and Operation

The general arrangement of the dilatometer is illustrated in Fig. 2.2.1 The cylindrical test specimen, A, of condensed amalgam, the dimensional change of which is to be monitored, is placed in a stainless steel stand, B. The stand is then located centrally beneath the sensing probe, C, attached to one end of the beam, D. The other end of the beam carries the core of a Schaevitz A.C. linear variable differential transformer (LVDT), E. The beam is symmetrical and is suspended by a carbon-steel pivot-shaft pressed into its centre. The pivots fit into 0.19mm synthetic ruby watch jewels, G. Similar

Fig. 2.2.1 General arrangement of dilatometer

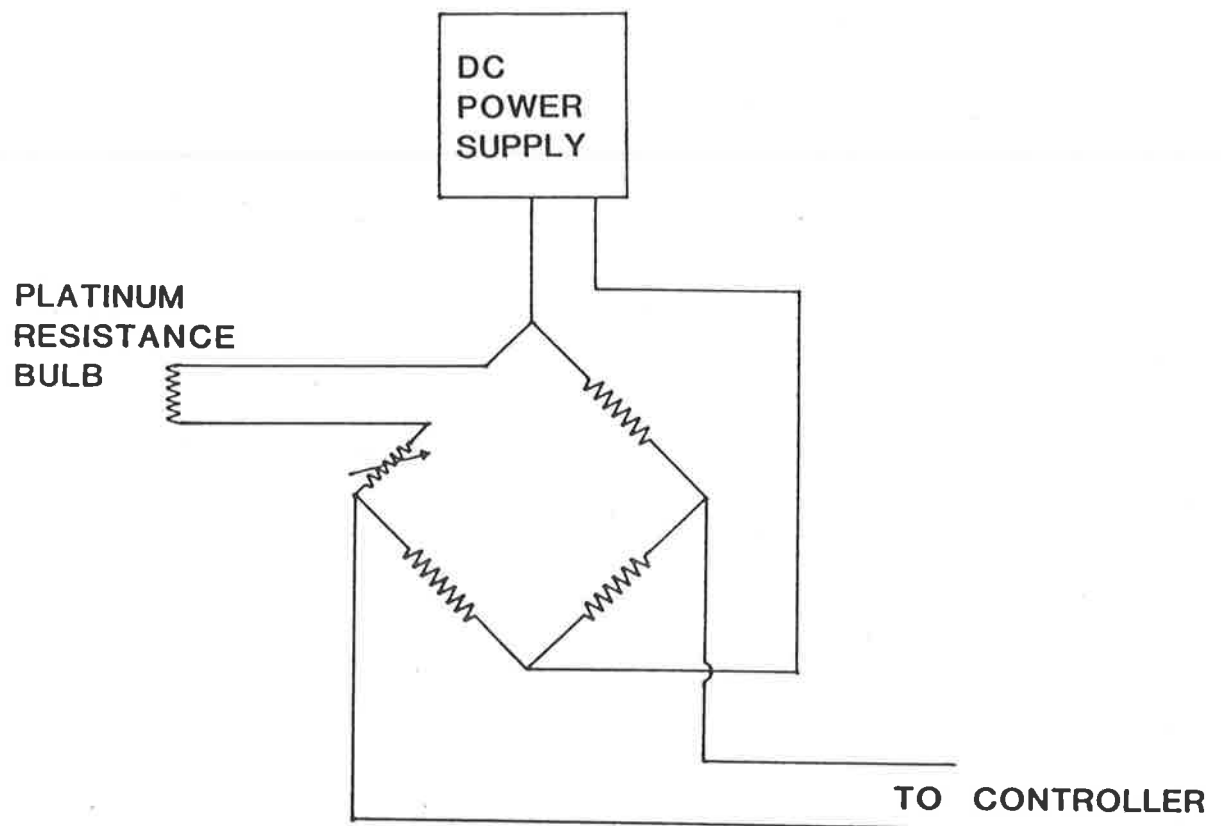


jewelled bearings are used at F and H. The design eliminates the need for extremely close tolerances between the pivots and the jewelled bearings as the pivots are always at the lowest position in the bearings due to gravity. Height adjustments of the LVDT and the beam are made by means of differential pitch lead screws I and J to facilitate the initial alignment and zeroing of the apparatus. When the dilatometer is correctly zeroed the beam is horizontal. The hemispherical sensing probe (as suggested by Muench (Muench 1970)) can be lowered onto the surface of the specimen or lifted clear of it by means of the lever-operated cam, K. The balancing screw, L, attached to the beam permits the net load applied to the specimen by the measuring system to be accurately controlled over wide limits. The magnitude of this net load can be determined directly by placing a pan balance under the sensing probe C. Calibration of the dilatometer and the LVDT can be readily carried out with the aid of a micrometer head mounted so that it makes contact with the upper end, C', of the rod carrying the specimen probe. Calibration tests carried out in this way indicated the LVDT was linear over its entire range of $\pm 1.25\text{mm}$ and the displacement could be resolved to an accuracy of $\pm 0.01\mu\text{m}$.

Temperature Control

The standards specify that the temperature of the test specimen must be maintained at $37\pm 1^\circ\text{C}$ for the duration of the test. To comply with this the dilatometer was housed in a water jacketed oven. A novel method was devised to control the temperature of the oven because of the difficulty in obtaining commercially available control equipment that would accurately control so close to ambient. The technique used is shown schematically in Fig. 2.1.2. A platinum resistance bulb

Fig. 2.2.2 Oven temperature control circuit diagram



(placed adjacent to the specimen in the oven) and a variable resistor are connected across one arm of a Wheatstone Bridge driven by a fixed voltage D.C. power supply. The potential across the balance points is then fed to a standard Eurotherm 0-1200°C chromel-alumel P.I.D. controller. By varying the value of the variable resistor the output to the controller can be amplified over very wide ranges enabling very accurate temperature control e.g. in the present experiment a change of 5°C in the oven temperature produces an apparent temperature change of 300°C at the controller.

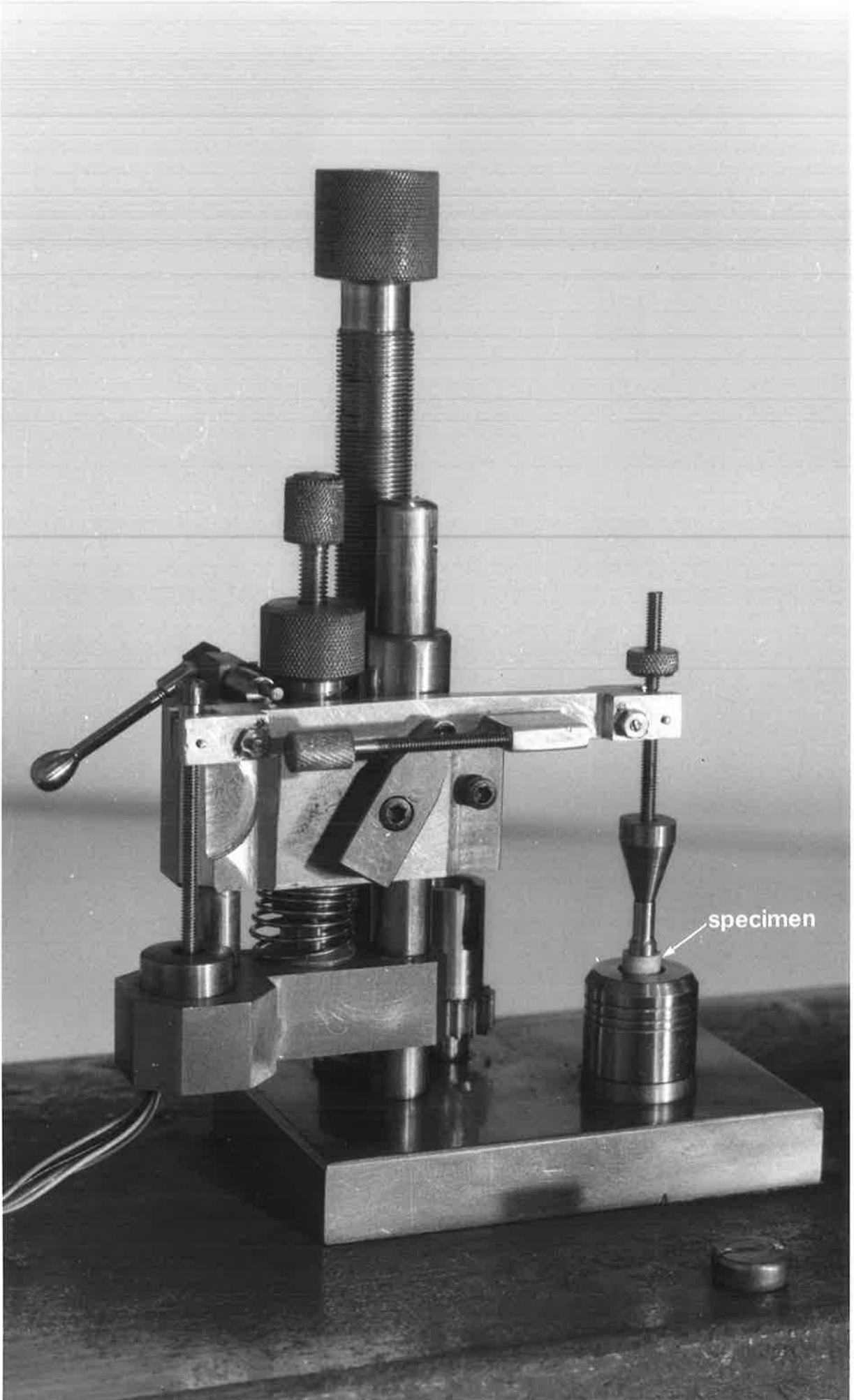
The actual temperature of the oven is monitored using a thermistor probe connected to a digital readout thermometer and the variation in temperature during a seven day experiment is less than $\pm 0.2^\circ\text{C}$ and typically $\pm 0.1^\circ\text{C}$. The front of the oven is enclosed in a large plastic "tent" which is maintained at $36 \pm 1^\circ\text{C}$ to minimise temperature fluctuations when the oven door is opened to insert or remove specimens.

The influence of external vibrations was eliminated by placing the oven on a concrete slab which was mechanically isolated from its surroundings by air bag supports. A consequence of the use of air bags was that the oven tilted when the door was opened and closed and so it was necessary to stand the dilatometer on a gimble. The dilatometer in situ is shown in Fig. 2.2.3.

Data Logging

A computer program DUALDVM1 (Appendix 1) was written for an HP86A (Hewlett Packard) micro-computer for data-logging and

Fig. 2.2.3 Dilatometer in situ



plotting of dimensional change data. The output signal from the LVDT was processed through an HP3421A (Hewlett Packard) multi-channel data-logger and stored on floppy disk using the HP86A. The program made provision for running two dimensional change rigs concurrently, even though their start and finish times may not coincide. Data was logged using an exponential time base, so that readings were taken more frequently at the commencement of the test, when dimensional changes were occurring most rapidly.

At the conclusion of the test, the specimen length was measured and stored on the data file to enable the data to be scaled during plotting.

Plotting of dimensional change ($\mu\text{m cm}^{-1}$) against time (hours) either on screen or using an HP7550A plotter was possible either during the test (using an assumed specimen length of 10mm) or at the completion of the test (using the actual specimen length). The values were plotted using the reading 5 minutes after the end of trituration as the fiducial reading as specified in the standards.

At the completion of five dimensional change tests, the program "AVERAGE" (Appendix 2) was used to average the data and calculate standard deviations for plotting of the final averages.

CHAPTER 3

EXPERIMENTAL

3.1 Specimen Manufacture

The requirements of ADA specification No 1 were rigidly adopted in the preparation and testing of all specimens. Trituration was carried out using an "Ultramat"* high speed mechanical mixer. A quartz operated stopwatch was used to control trituration time because the inbuilt timer on the triturator was found to be inaccurate. Specimens of each mix were condensed in the manner specified in the standard into cylinders 4mm in diameter and 10mm in length using the apparatus shown in Fig. 3.1.1. Trituration and condensation were carried out at $36\pm 1^{\circ}\text{C}$ in the plastic enclosure ("tent") attached to the front of the oven containing the dimensional change equipment (Fig. 3.1.2).

To determine the effect of condensation pressure, the dead weight hung from the condensation rig was varied to provide condensation pressures of 3, 7, 14 and 42MPa, and each alloy was triturated according to the manufacturers' recommendations. The use of an hydraulic jack facilitated the controlled application of the weight during condensation.

Trituration times of 2, 5, 8, 12 and 16 seconds were used to evaluate the effect of trituration time, and all samples were condensed using the condensation pressure specified in the standards (14MPa).

* Southern Dental Industries

Fig. 3.1.1 Apparatus for condensing standard specimens

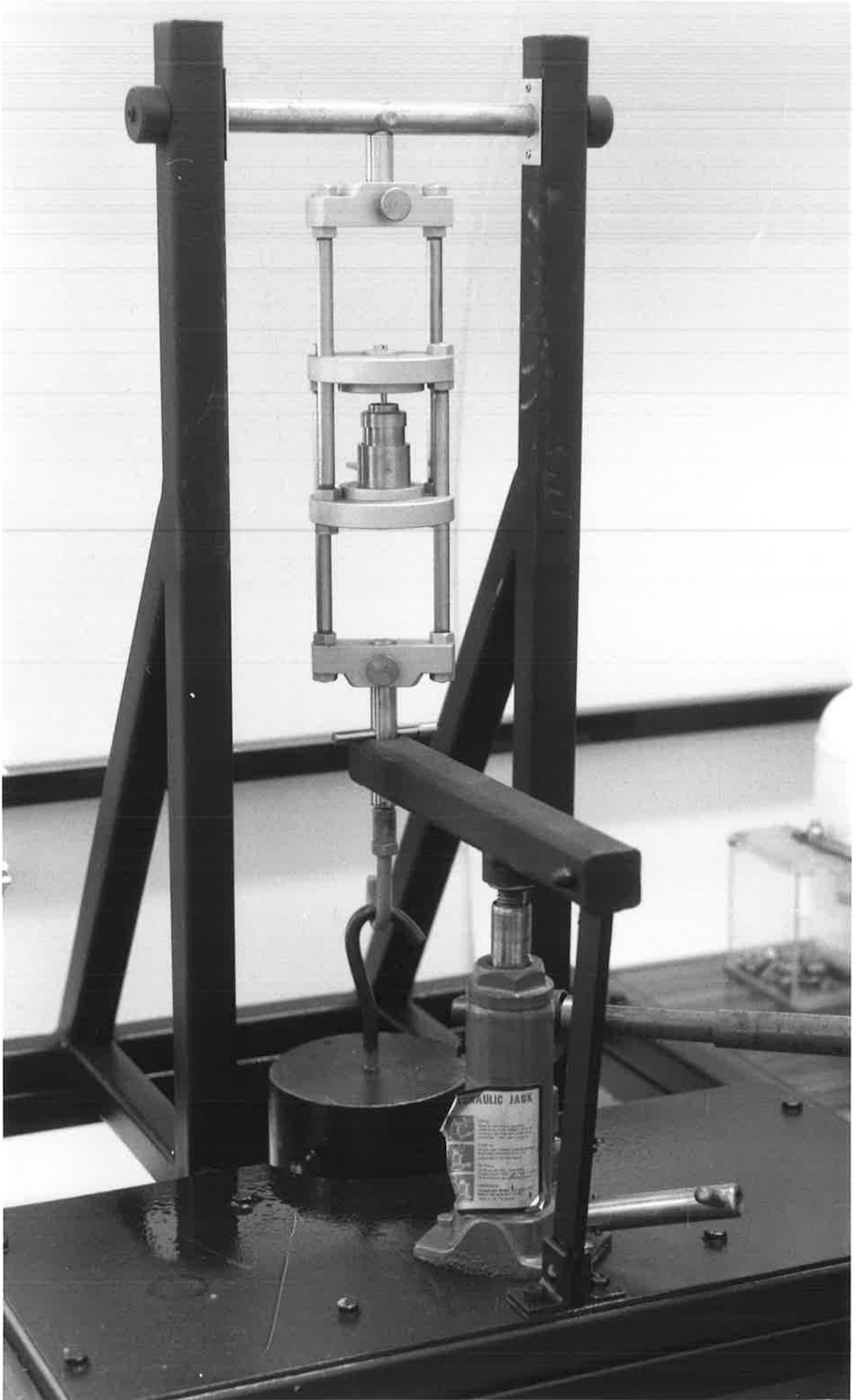


Fig. 3.1.2 Overall view of equipment



3.2 Dimensional Change Measurement

Specimens used for dimensional change measurements were ejected from the condensation die directly into the specimen holder of the dilatometer and the holder placed beneath the sensing probe of the dilatometer. After zeroing of the LVDT, logging of the change in specimen length with time on the microcomputer was commenced, usually less than 45 seconds after the end of condensation.

The computer program used the time at the end of trituration as zero time and the dimensional change at five minutes as zero dimensional change. Tests were run for a minimum of 24 hours, after which, the specimens were removed and their lengths measured and stored on the data file to enable the scaling of the dimensional change data for plotting.

Measurements of dimensional change were repeated 5 times (in accordance with ADA Spec No 1).

3.3 Compressive Strength Measurement

Compressive strength measurements were carried out 60 minutes, 24 hours and 7 days after condensation. Specimens were placed in cellophane bags and stored in the tent at $36\pm 1^\circ\text{C}$ until ready for testing. Immediately prior to testing, the ends of the specimens were ground flat and parallel on 1200 grit silicon carbide paper.

A compression cage fitted with 5 mm diameter platens, installed in an Instron Testing Machine operated at a crosshead speed of 0.5 mm/minute, was used for the tests. Tests were repeated 5 times (in accordance with ADA Spec No 1).

3.4 Final Mercury Content

The alloy powder and mercury were accurately weighed before the manufacture of the dimensional change and compressive strength specimens. The mercury expressed during condensation was collected and weighed and these results were used to calculate the final mercury content of the amalgam using the method of Jorgensen and Nielsen (Jorgensen and Nielsen 1964) viz.

$$\text{final mercury content} = \frac{\text{initial mercury content} - \text{mercury expressed}}{\text{initial weight of mercury} + \text{alloy}}$$

3.5 Scanning Electron Microscopy

The effect of different condensation pressures on the reaction products of a lathe-cut amalgam alloy (New True Dentalloy) and a spherical amalgam alloy (Lojic) were investigated using scanning electron microscopy.

A Philips 505 scanning electron microscope (SEM) with a Kevex Energy Dispersive X-ray analysis system (EDAX) and a Tracor Northern image analysis facility (TN5500) were used in the investigation.

Amalgam specimens were prepared from each of the alloys using condensation pressures of 3, 14 and 42MPa by the standard method and stored in the tent at $36 \pm 1^\circ\text{C}$. After 7 days a longitudinal flat was ground on one side of each specimen using standard metallographic techniques. During grinding and polishing, the specimens were held in a simple jig to ensure that the same depth of material was removed from each specimen. The samples were then immediately placed in the SEM for analysis.

For each sample a back scattered electron image and x-ray maps

of silver, tin, copper and mercury were acquired at five standardized positions around the midpoint of the longitudinal flat, and stored on the TN5500. By carefully manipulating these stored images using software available on the TN5500, the phases present in the microstructure and their proportions could be accurately determined.

CHAPTER 4

RESULTS





4.1 Compressive Strength

4.1.1 Effect of condensation pressure.

The influence of condensation pressure over the range 3MPa to 42MPa on the 1 hour, 24 hour and 7 day compressive strength of amalgams prepared from the low (2%) copper lathe-cut alloy New True Dentalloy and the high copper admix alloys Dispersalloy and Permite C, is shown in Fig. 4.1.1. In Fig. 4.1.2 similar data is presented for the three spherical particle alloys Tytin, Lojic and Valiant. All amalgams were triturated in accordance with the respective manufacturers' recommendation.

It is immediately apparent from Fig. 4.1.1 that for these three amalgams the compressive strength, measured at 1 hour, 24 hours and 7 days, increases significantly as the condensation pressure applied during the fabrication of the test specimen is increased from 3MPa to 42MPa. As previously mentioned (Lussi & Buergin 1987) it has been suggested that the condensation stresses applied by dental practitioners are considerably below the recommended 15MPa. Therefore of particular relevance from a clinical point of view, is the difference in compressive strength over the condensation range 3MPa to 14MPa. With Dispersalloy tested either 24 hours or 7 days after condensation, the compressive strength of the amalgam is increased by 50% (e.g. from 200MPa to 400MPa) over this range of condensation pressure. The conventional lathe-cut amalgam New True

Fig. 4.1.1 The effect of condensation pressure on the compressive strength of lathe-cut and admixed particle amalgams

-  3MPa condensation pressure
-  7MPa condensation pressure
-  14MPa condensation pressure
-  42MPa condensation pressure

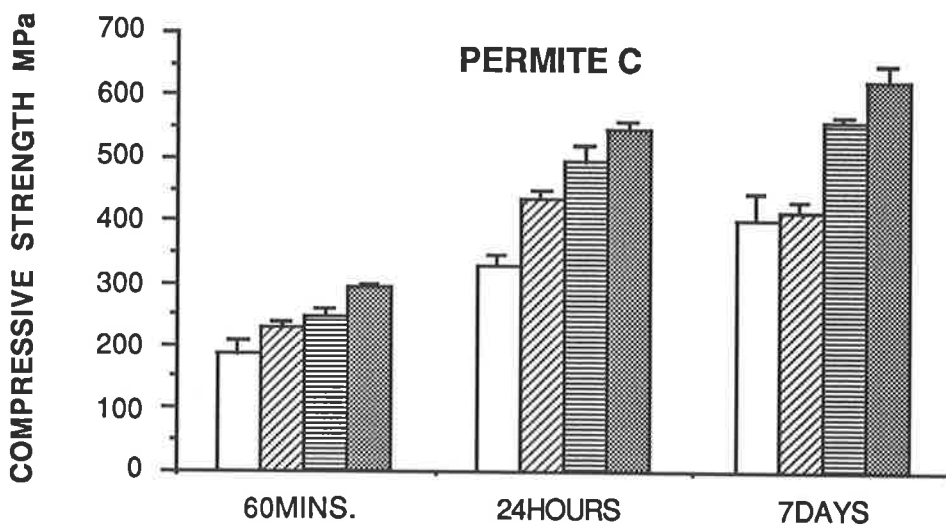
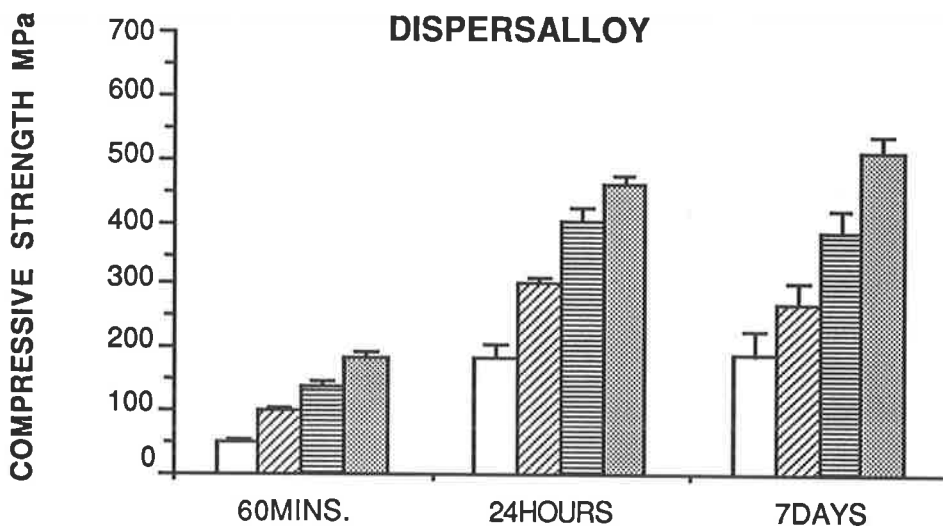
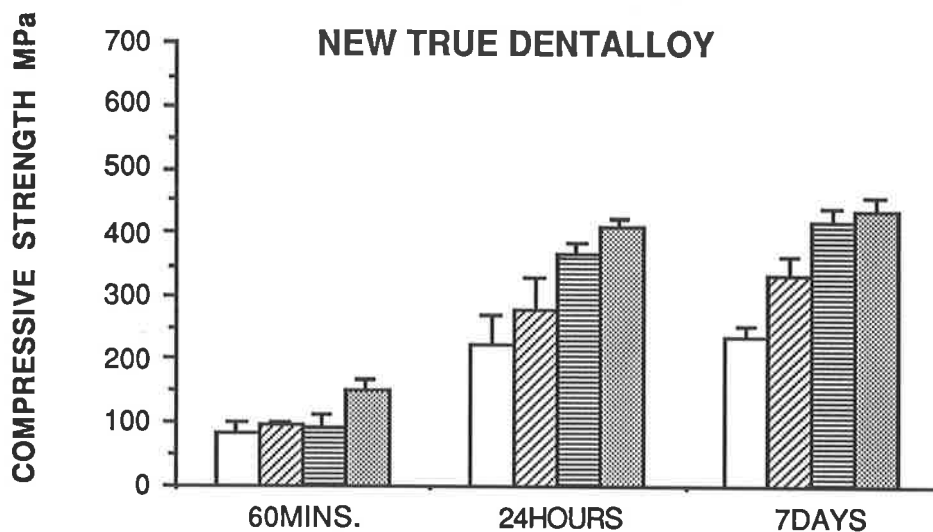




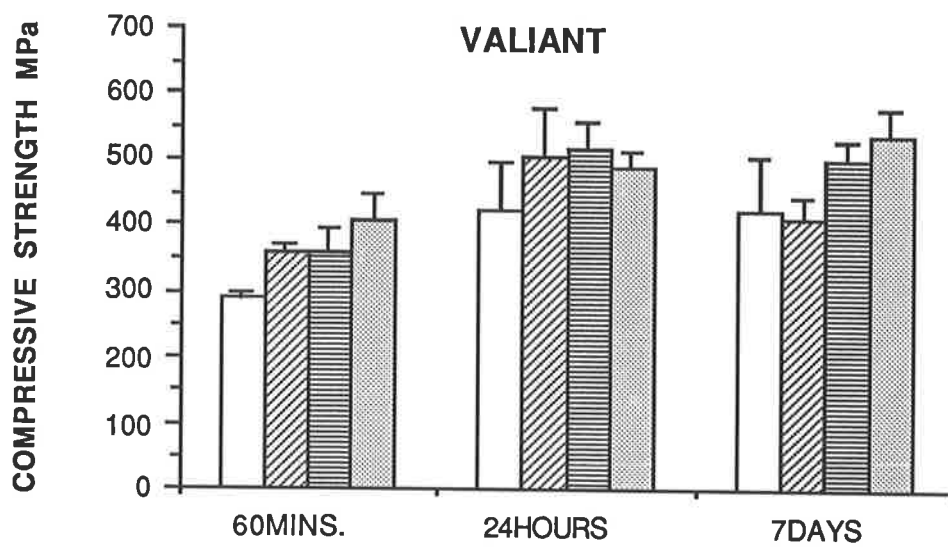
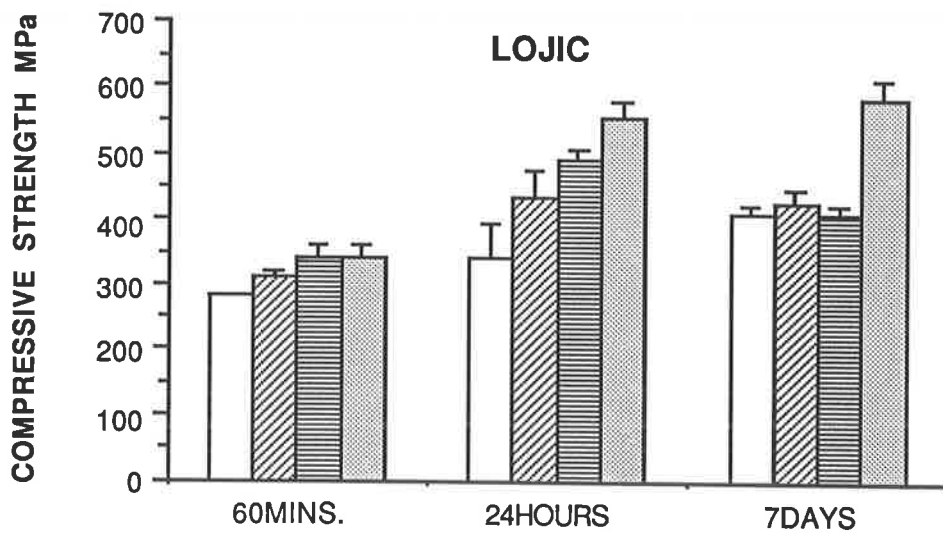
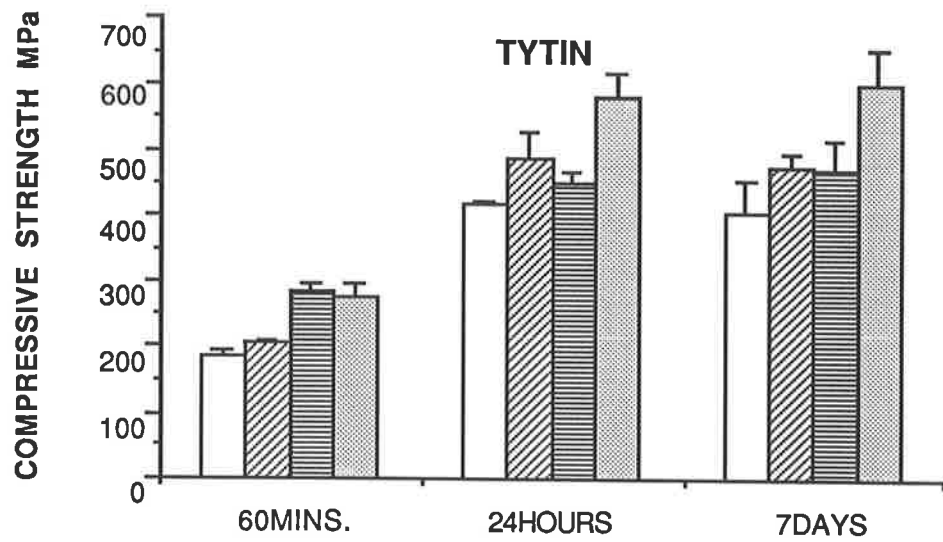


Fig. 4.1.2 The effect of condensation pressure on the compressive strength of spherical particle amalgams

-  3MPa condensation pressure
-  7MPa condensation pressure
-  14MPa condensation pressure
-  42MPa condensation pressure



Dentalloy follows a similar trend. For Permite C, an inherently stronger amalgam, the 24 hour or 7 day compressive strength is about 33% higher for specimens condensed at 14MPa rather than 3MPa.

This variation in compressive strength with condensation pressure is not nearly as marked with the spherical particle alloys, the results of which are shown in Fig. 4.1.2. Tytin and Valiant show an increase in compressive strength of some 15% over this range of condensation pressure and in the case of Lojic, although the 24 hour compressive strength does show some dependence on condensation pressure, the 7 day compressive strength remains virtually constant over the range 3MPa to 14MPa.






It will also be noted that relative to all the other alloys tested, Lojic and Valiant display very high 1 hour compressive strengths even when condensed with a condensation pressure as low as 3MPa.

4.1.2 Effect of trituration time.

The 1 hour, 24 hour and 7 day compressive strengths of each of the amalgams were determined for trituration times of 2, 5, 8, 12 and 16 seconds. These specimens were condensed using the condensation pressure recommended in the standards (14MPa) and their compressive strengths are presented in Figures 4.1.3 and 4.1.4.

In each case severe undertrituration (e.g. 2 seconds)

Fig. 4.1.3 The effect of trituration time on the compressive strengths of lathe-cut and admixed particle amalgams

-  2 seconds trituration time
-  5 seconds trituration time
-  8 seconds trituration time
-  12 seconds trituration time
-  16 seconds trituration time

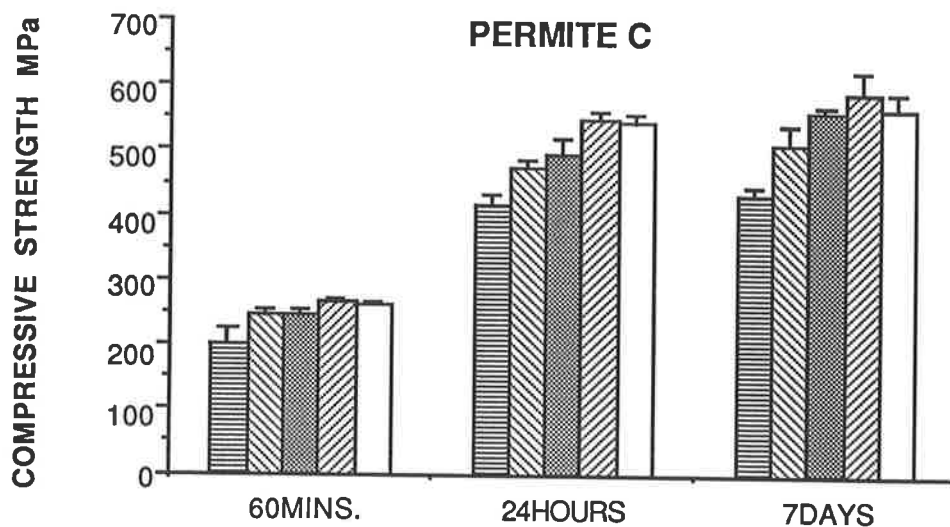
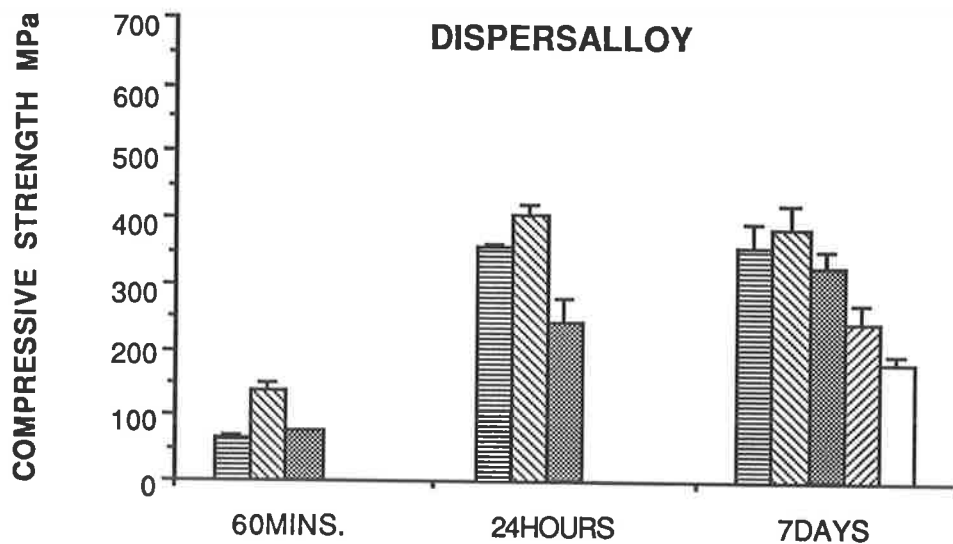
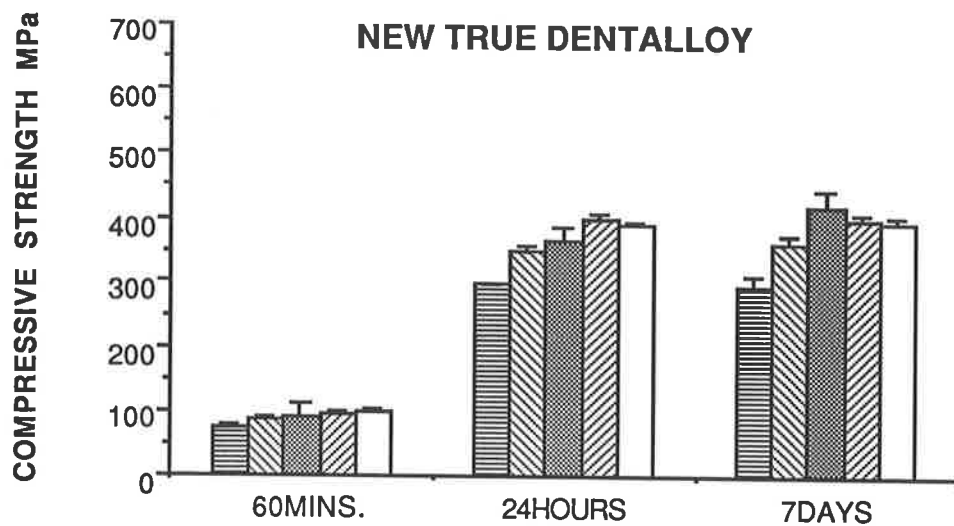





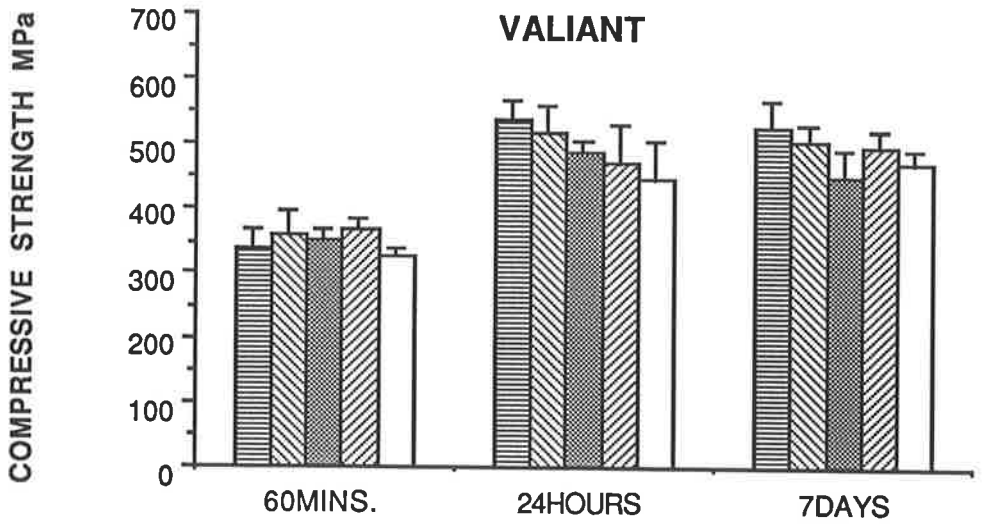
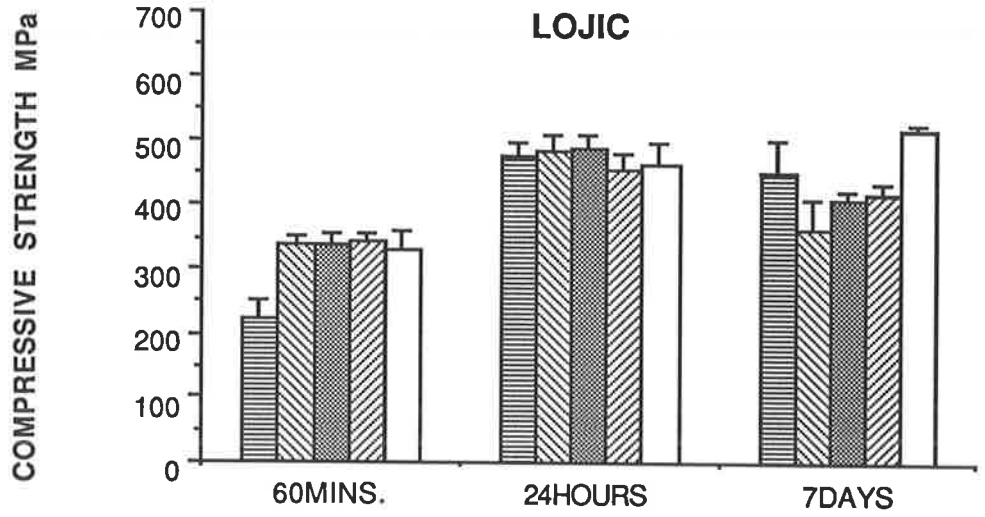
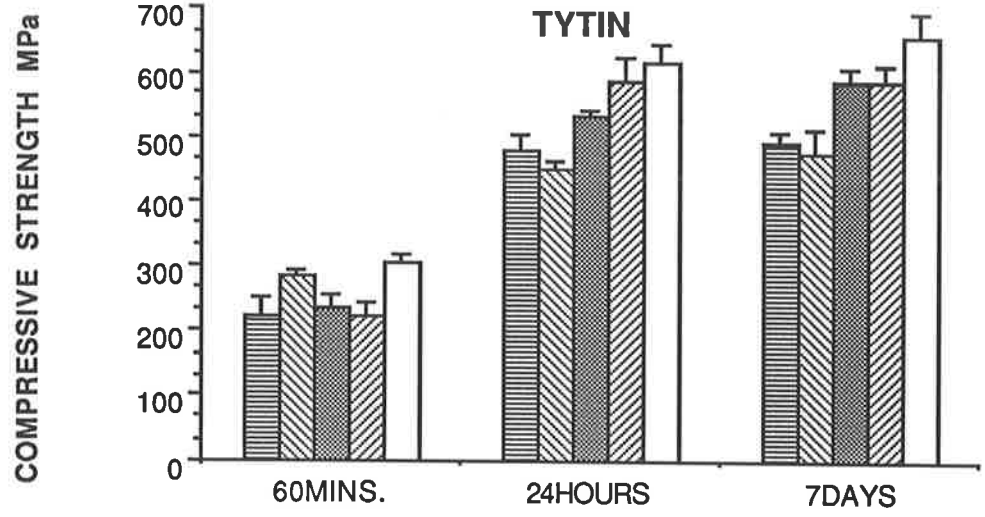


Fig. 4.1.4 The effect of trituration time on the compressive strengths of spherical particle amalgams

-  2 seconds trituration time
-  5 seconds trituration time
-  8 seconds trituration time
-  12 seconds trituration time
-  16 seconds trituration time



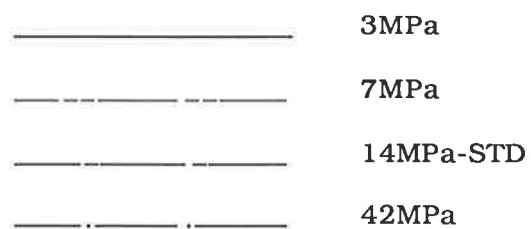
resulted in an amalgam with lower compressive strength. Within the range 5 - 16 seconds, trituration time appeared to have little effect on compressive strength, with the notable exception of Dispersalloy, the 7 day strength of which decreased from 385MPa (5 seconds) to 241MPa (12 seconds). The trituration time recommended by the manufacturer for this alloy is 8 seconds. This was clearly a consequence of overtrituration brought about by the use of a pestle in the capsule. Indeed, after 16 seconds trituration of Dispersalloy the hardening process had proceeded to the stage at which it was no longer possible to condense the amalgam to form a standard test specimen.

4.2 Dimensional Change during hardening

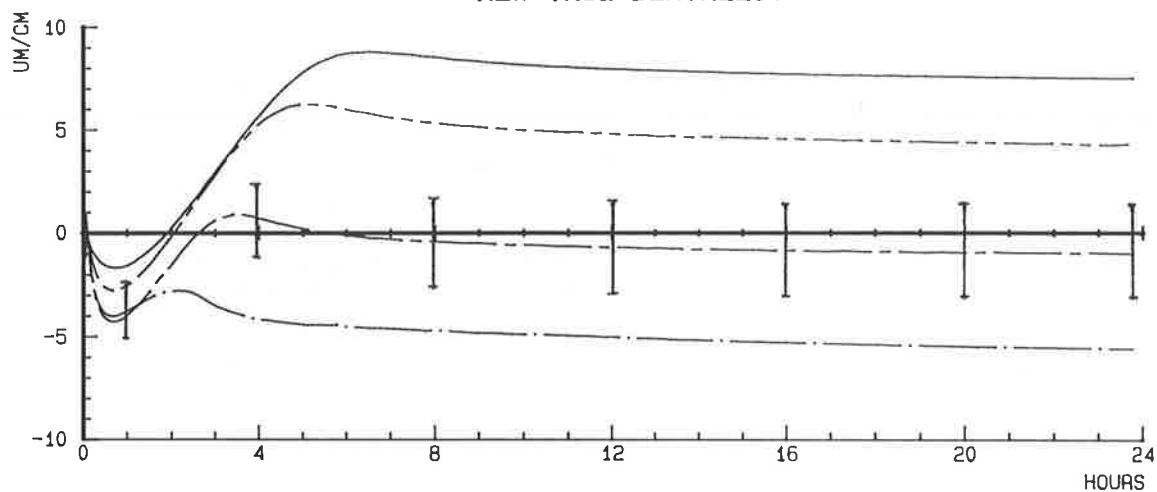
4.2.1 Effect of condensation pressure.

The influence of variations in condensation pressure on dimensional change during hardening was investigated for amalgams prepared from each of the six alloys. Condensation pressures of 3, 7, 14 and 42MPa were employed and dimensional change was plotted as a function of time for 24 hours after the preparation of each test specimen. The fiducial reading was taken at 5 minutes as specified in ADA Spec No 1. In Fig. 4.2.1 the results obtained for the group of non spherical particle alloys New True Dentalloy, Dispersalloy and Permite C are presented and for comparison the very different behaviour of the group of spherical particle alloys (Tytin, Lojic and Valiant) is shown in Fig. 4.2.2. The reproducibility of the dimensional change curves for each amalgam alloy is indicated by the magnitude of the error bars ($\pm 1\sigma$) shown for amalgams prepared according to the respective manufacturer's specification.

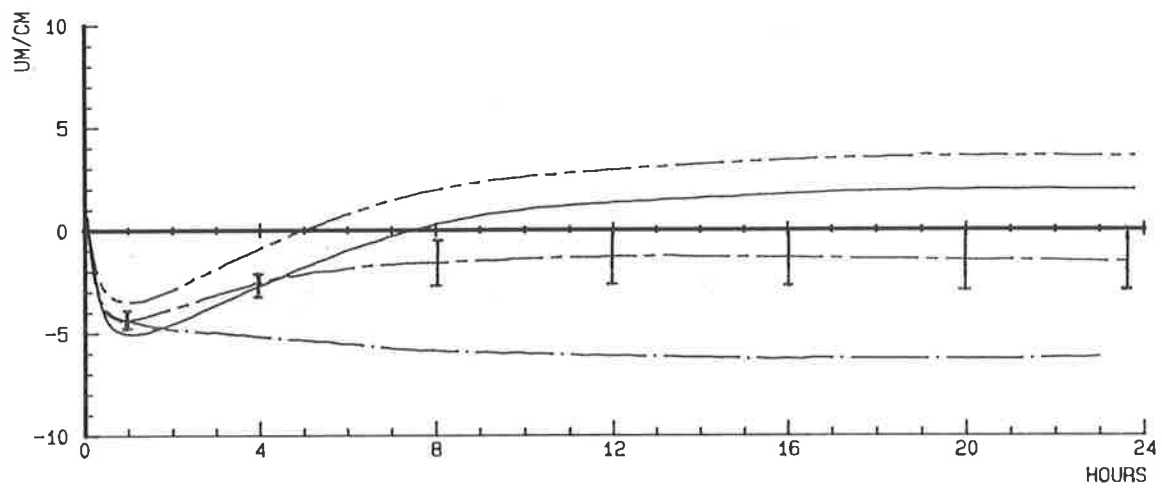
Fig. 4.2.1 The effect of condensation pressure on the dimensional change curves of lathe-cut and admixed amalgams



NEW TRUE DENTALLOY



DISPERSALLOY



PERMITE C

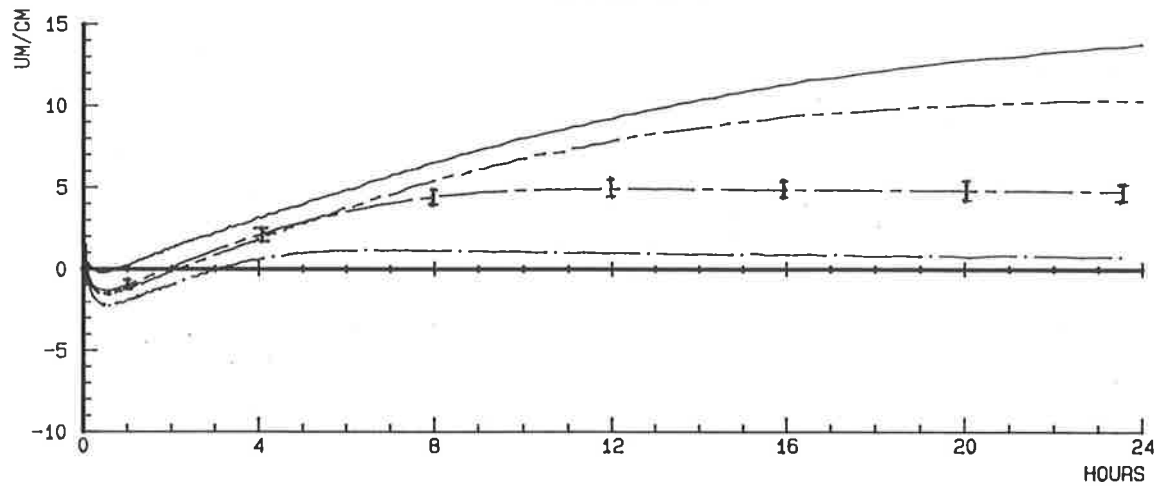
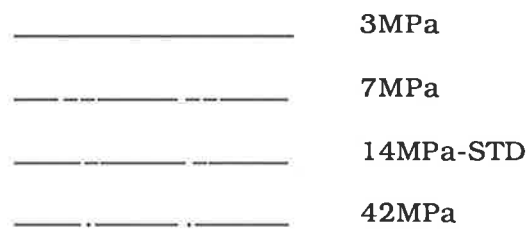
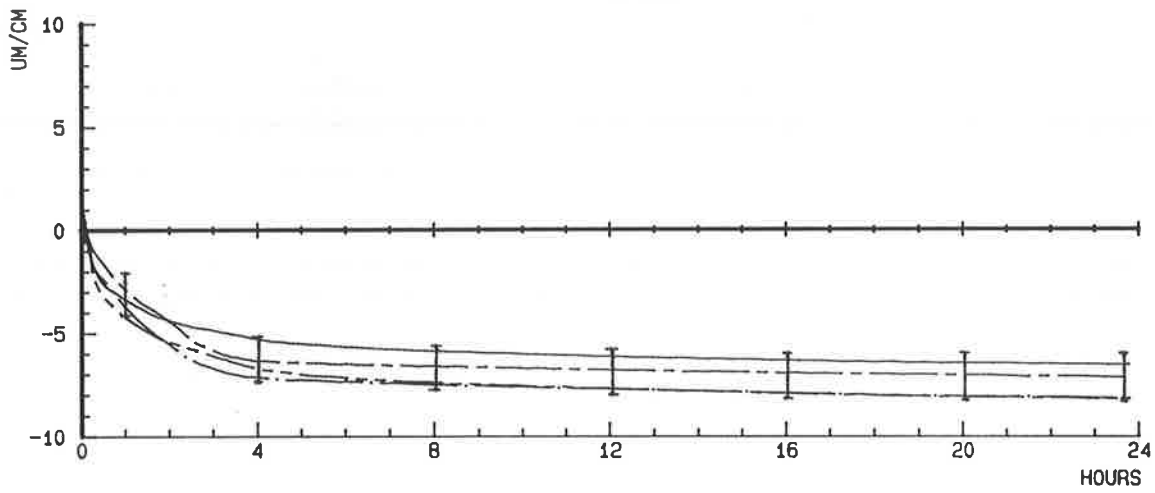


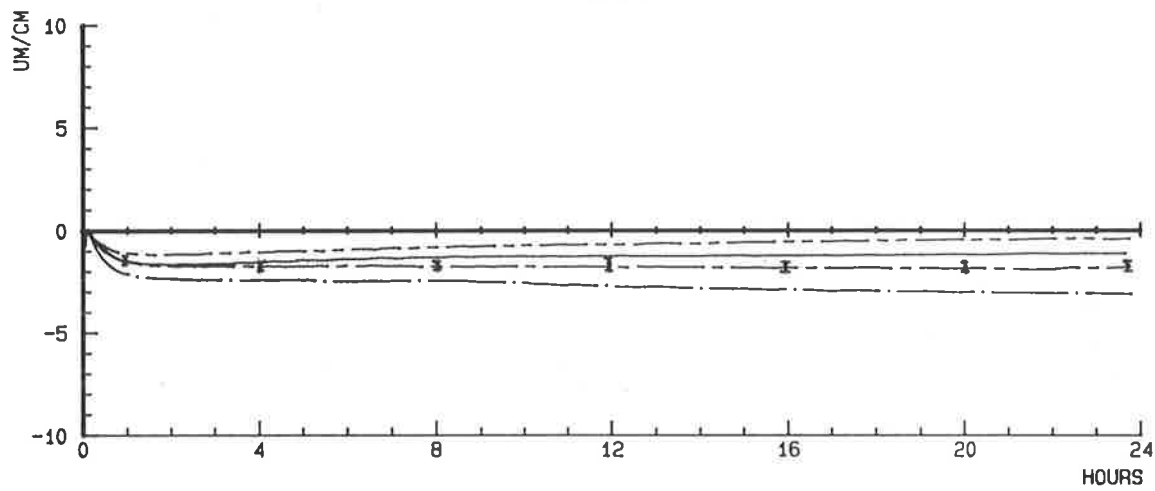
Fig. 4.2.2 The effect of condensation pressure on the dimensional change curves of spherical particle amalgams



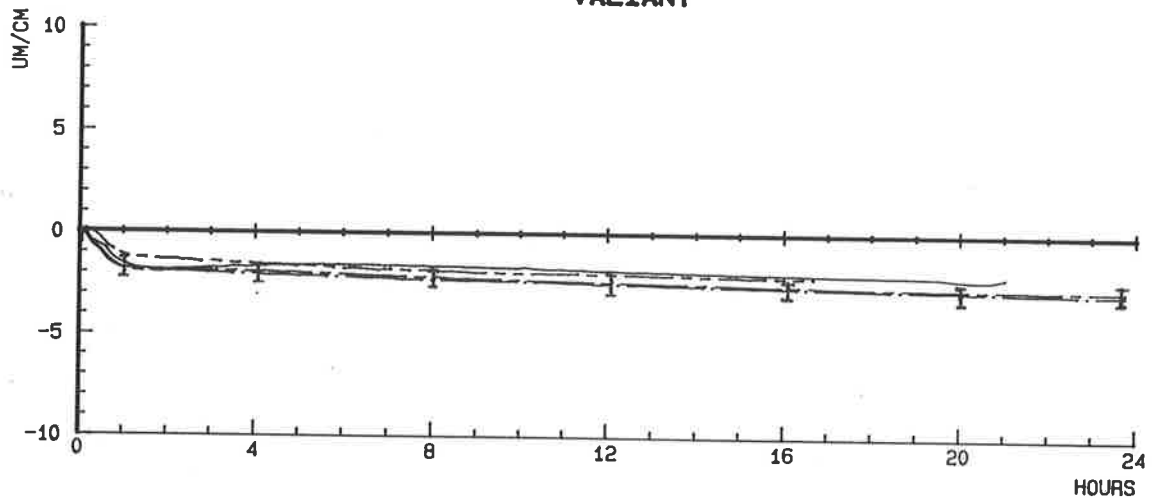
TYTIN



LOJIC



VALIANT



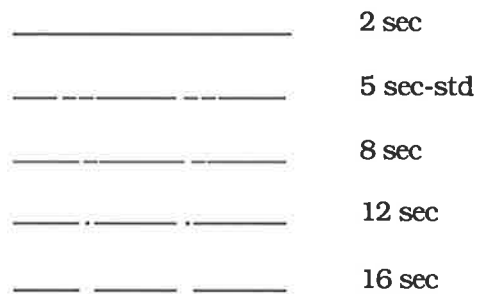
Two major differences in the response of the two groups of alloys can be seen. Firstly, the dimensional change of the lathe-cut and admixed amalgams (Fig. 4.2.1) display a marked sensitivity to variations in condensation pressure, to the extent even that in some cases the sign of the dimensional change is altered. For example, Dispersalloy condensed at a pressure of 7MPa shows a slight expansion after 24 hours whereas the same material condensed with a condensation pressure of 14MPa contracts slightly. In contrast the dimensional change of the spherical particle amalgams (Fig. 4.2.2) shows very little reaction to changes in condensation pressure over the entire range investigated (3-42MPa).

Secondly, New True Dentalloy, Dispersalloy and Permite C (Fig. 4.2.1) clearly show that specimens prepared using a condensation pressure of 14MPa continue to change in length for at least 8 hours after preparation. On the other hand, the spherical particle alloys (Fig. 4.2.2) reached dimensional stability much more rapidly. Lojic and Valiant, for example, reached a stable length approximately one hour after preparation while Tytin took three hours to achieve dimensional stability.

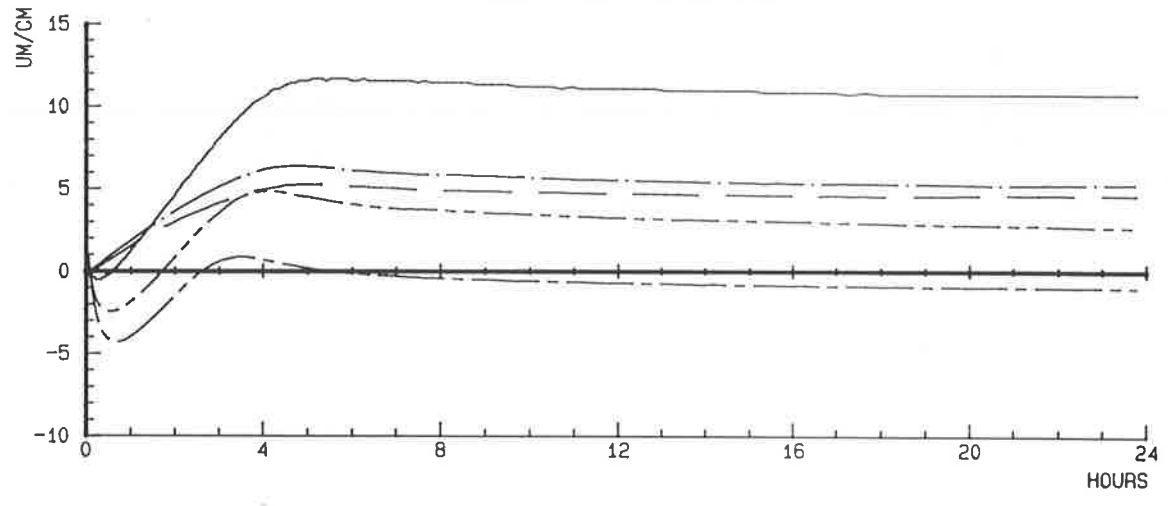
4.2.2 Effect of trituration time

The sensitivity of dimensional change curves to trituration time was assessed for trituration times of 2, 5, 8, 12 and 16 seconds, all specimens being prepared using the same condensation pressure (14MPa). The trituration times recommended by the manufacturers are either 5 seconds (Dispersalloy, Tytin and Valiant) or 8 seconds (New True

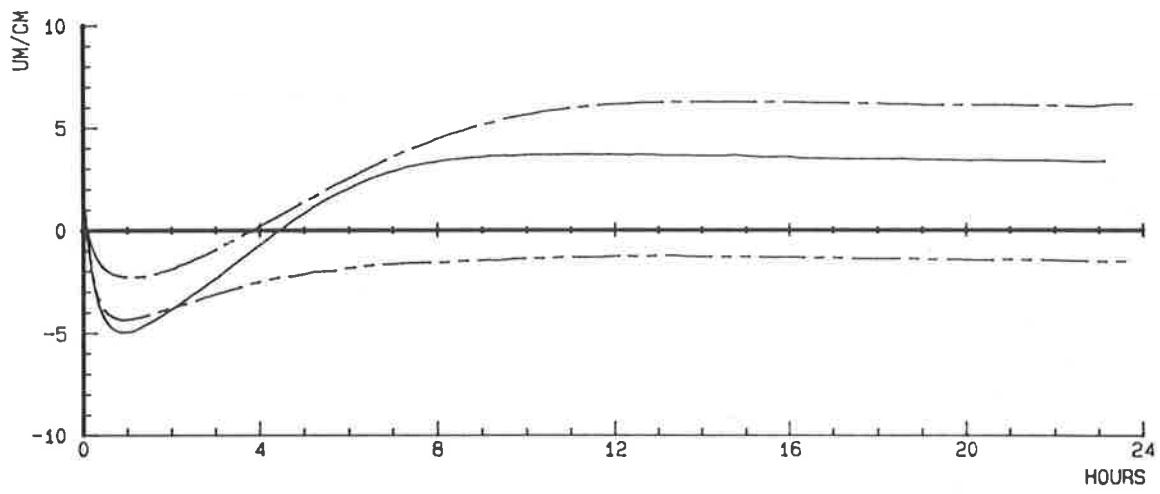
Fig. 4.2.3 The effect of trituration time on the dimensional change curves of lathe-cut and admixed particle amalgams



NEW TRUE DENTALLOY



DISPERSALLOY



PERMITE C

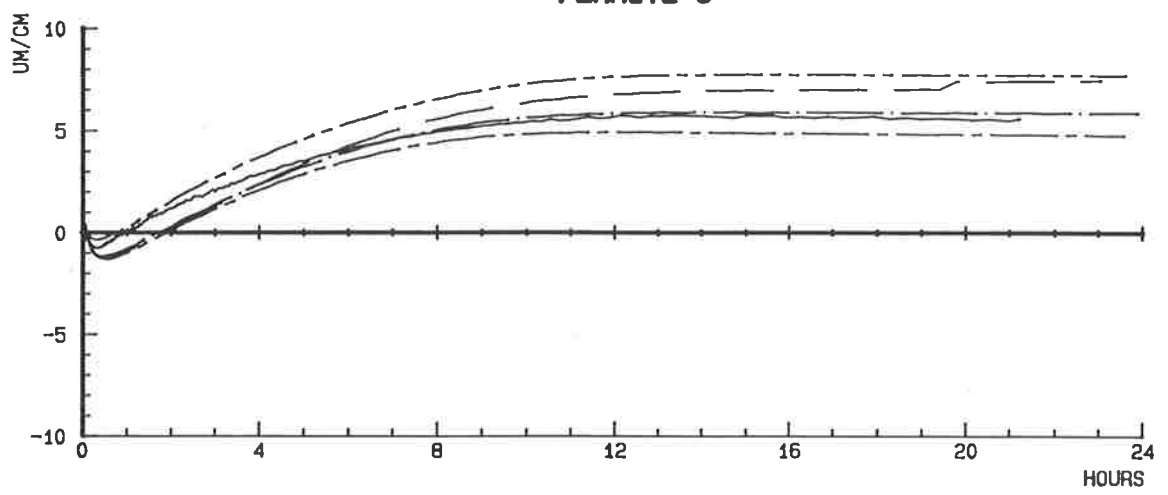
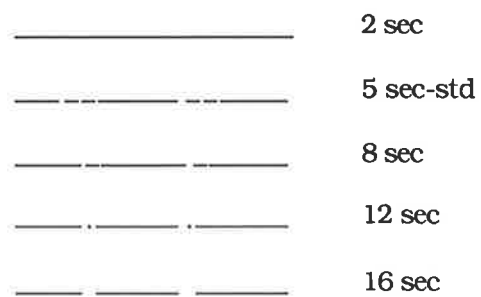
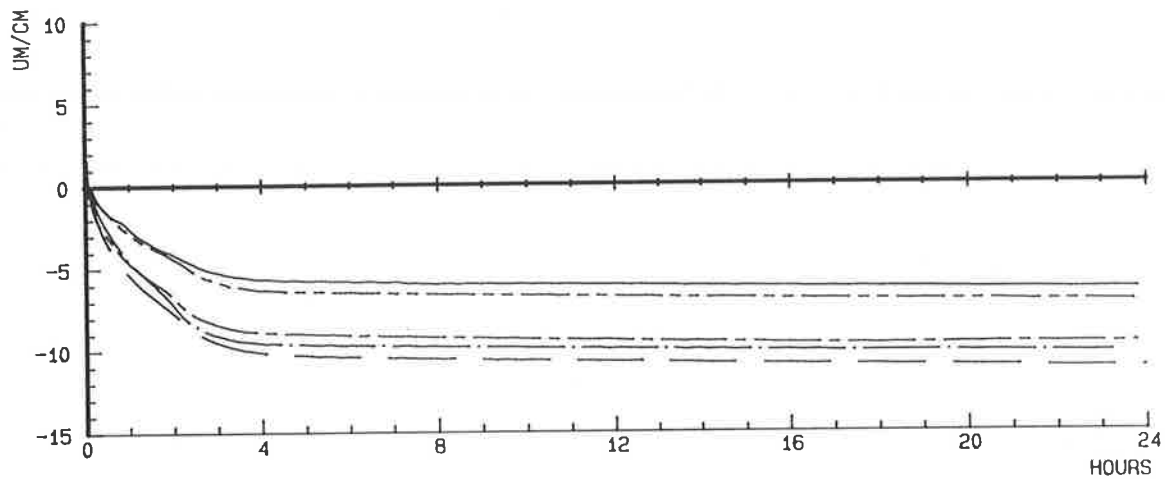


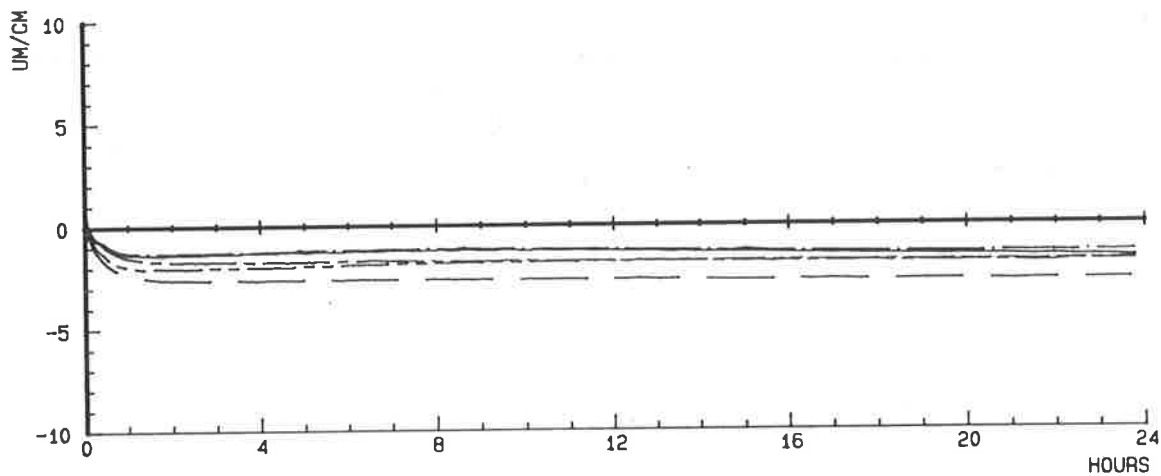
Fig. 4.2.4 The effect of trituration time on the dimensional change curves of spherical particle amalgams



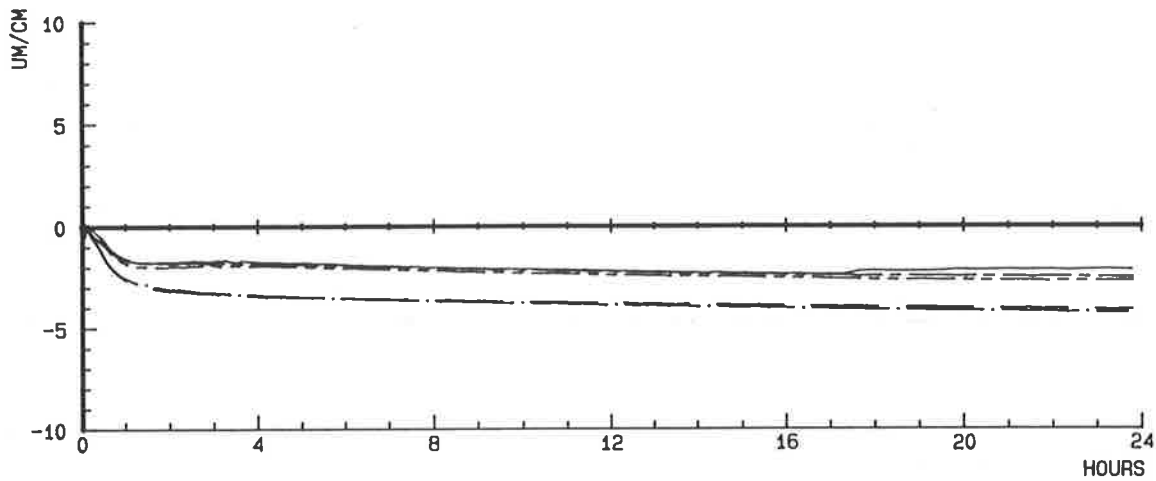
TYTIN



LOJIC



VALIANT



Dentalloy, Permite C and Lojic). The effect on the dimensional change curves of New True Dentalloy, Dispersalloy and Permite C resulting from changes in trituration time are shown in Fig. 4.2.3. From these results it is evident that the dimensional change curves of New True Dentalloy and Dispersalloy are extremely sensitive to changes in trituration time. For example, with New True Dentalloy, an increase in trituration time from 2 to 8 seconds results in reduced expansion throughout the hardening reaction, so that the dimensional change curves are effectively shifted downwards. Further increases in trituration time from 8 to 16 seconds reversed this trend and the curves are shifted upwards, i.e. now lie above the 8 second curve. The influence of trituration time on the dimensional change curves of Dispersalloy and Permite C is similar to that of New True Dentalloy, although the response of Permite C is less marked.

In marked contrast, changing the trituration time for the spherical particle alloys Tytin, Lojic and Valiant had no significant effect on dimensional change (Fig. 4.2.4). For each of these alloys the dimensional change curves, for specimens triturated for between 5 seconds and 12 seconds, were virtually independent of trituration time.

4.2.4 Effect of particle deformation during trituration

As noted previously, changes in trituration time have a significant effect on the dimensional change curves of amalgams containing lathe-cut particles. This is due to either improved mixing of the alloy particles with mercury, or increased deformation of the particles as a result of particle-particle

interactions and particles colliding with the end walls of the capsule. To separate these effects a series of experiments was carried out using the lathe-cut powder, New True Dentalloy. A standard quantity of the powder was put into a Logic type capsule without mercury and vibrated using the Ultramat triturator for a predetermined length of time. Only deformation of the alloy particles could occur during this time. The recommended quantity of mercury was then added to the alloy powder, and the mercury and powder were mixed using the Ultramat triturator.

The dimensional change curves for specimens condensed from these amalgams together with those of specimens triturated in the normal manner are presented in Figs. 4.2.5. In Fig. 4.2.5(a) New True Dentalloy was vibrated for 11 seconds without mercury followed by 5 seconds with mercury. For comparison the dimensional change curves for specimens triturated normally for 5 seconds and 16 seconds are included. It is evident from these curves that the amalgam which had been plastically deformed by vibrating in the Ultramat prior to being triturated with mercury, behaved in the same way as the amalgam which had been triturated normally for 5 seconds. This suggests that the plastic deformation induced by vibrating the powder prior to the addition of mercury had little or no influence on the dimensional change curve.

In contrast, the dimensional change curve in Fig. 4.2.5(b) for the alloy vibrated for 8 seconds prior to being triturated with mercury for 8 seconds, is similar to that of the amalgam triturated normally for 16 seconds. In this case the particle deformation induced by vibrating prior to mixing with mercury

Fig. 4.2.5(a) The effect of vibrating lathe-cut particles for 11 seconds without Hg, followed by 5 seconds trituration with Hg

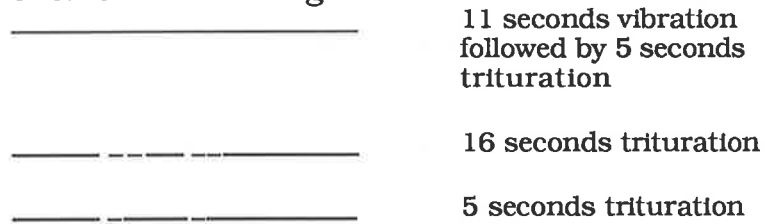


Fig. 4.2.5(b) The effect of vibrating lathe-cut particles for 8 seconds without Hg, followed by 8 seconds trituration with Hg

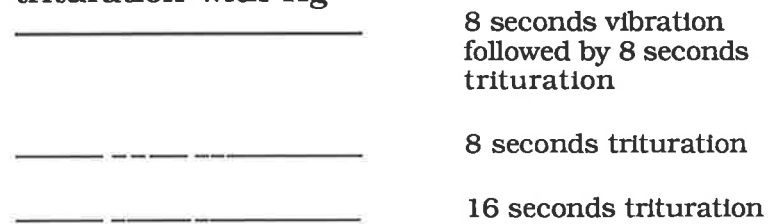
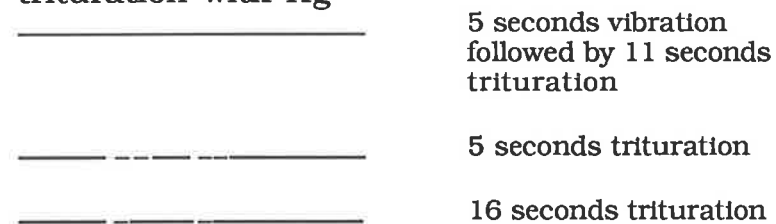
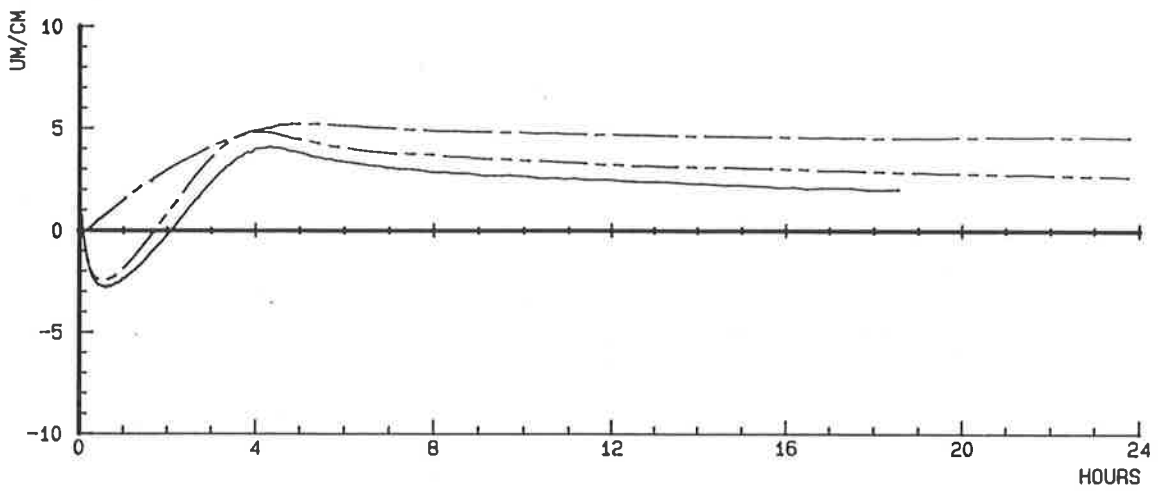
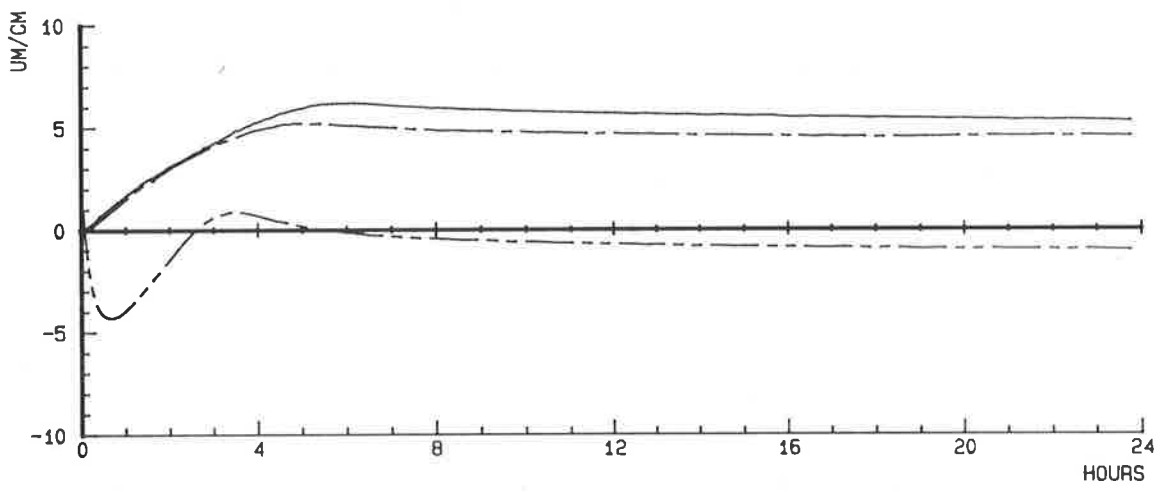
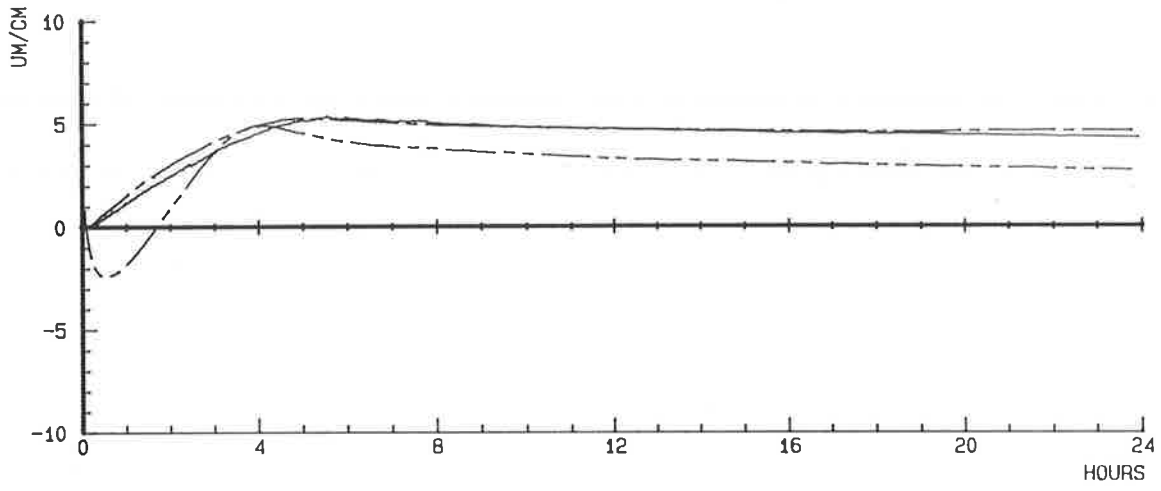


Fig. 4.2.5(c) The effect of vibrating lathe-cut particles for 5 seconds without Hg, followed by 11 seconds trituration with Hg





has influenced the dimensional change curve. Similarly, in Fig. 4.2.5(c) the amalgam vibrated for 5 seconds and then triturated with mercury for 11 seconds behaves as if it had been triturated with mercury for the full 16 seconds.

Mixing is considered to be complete when all the alloy particles are adequately wetted with mercury and the agglomerated amalgam pellet is homogeneous. If mixing is incomplete, the hardening reaction, and therefore the dimensional change curve, is strongly influenced by the heterogeneous nature of the mix. This is clearly the case in Fig. 4.2.5(a) where the severe deformation of the particles resulting from 11 seconds vibration without mercury, had little, if any, affect on the dimensional change curve. However in Figs. 4.2.5(b) and 4.2.5(c) it is the degree of particle deformation that has the strongest influence on the dimensional change curve, indicating that mixing in both of these latter cases must have been complete.

4.3 Final mercury content

4.3.1 Effect of condensation pressure.

The results of the determination of final mercury content after condensation at 3, 7, 14 and 42MPa are summarised in Table 4.3.1. To achieve adequate wetting of the lathe-cut and admixed alloy particles during trituration, an initial mercury to alloy ratio of 1:1 is required. Spherical particle alloys on the other hand, because of their much lower surface to volume ratio only require an initial mercury to alloy ratio of 0.7:1. The higher

Alloy	% Mercury				
	As Received	3MPa	7MPa	14MPa	42MPa
New True Dentalloy	50.0	47.9	45.3	41.6	32.8
Dispersalloy	50.0	47.3	45.9	40.7	31.4
Permite C	47.9	45.1	42.6	37.6	30.9
Lojic	43.2	42.2	40.7	38.4	32.3
Valiant	43.0	41.6	39.0	36.8	32.1
Tytin	42.5	40.8	38.6	37.0	31.0

Table 4.3.1 Effect of condensation pressure on final mercury content

final mercury content of the lathe-cut and admixed amalgams is due to their higher initial mercury content coupled with their marked sensitivity to condensation pressure, particularly in the range of greatest clinical importance, 3 to 14MPa. The final mercury content of New True Dentalloy, for example, decreases by 6.3% from 47.9% to 41.6% over this range, whereas the spherical amalgam Tytin only decreases by 3.8% over the same range of condensation pressures.

4.3.2 Effect of trituration time

Table 4.3.2 shows the residual mercury levels of the alloys after trituration times of 2, 5, 8, 12 and 16 seconds. As mentioned previously Dispersalloy could not be satisfactorily condensed if triturated for longer than 8 seconds because as the trituration time was increased the setting reaction became more advanced and the amalgam became more difficult to condense. As might be expected the poorer condensation gave rise to an increase in the level of residual mercury.

Alloy	% Mercury					
	As Received	2 sec	5 sec	8 sec	12 sec	16 sec
New True Dentalloy	50.0	42.3	41.3	41.6	37.5	36.7
Dispersalloy	50.0	39.1	40.7	46.1		
Permite C	47.9	38.7	39.3	37.6	38.9	38.9
Lojic	43.2	35.2	38.4	38.4	38.9	39.0
Valiant	43.0	35.4	36.8	37.8	38.5	39.7
Tytin	42.5	37.8	37.0	36.5	36.3	36.2

Table 4.3.2 Effect of trituration time on final mercury content

New True Dentalloy showed a decrease in residual mercury with increasing trituration time. This may well be due to the fracturing and rounding of the lathe-cut particles enabling better condensation to occur.

Permite C, although an admixed alloy like Dispersalloy, is different from Dispersalloy in several ways. The Permite C capsule does not contain a pestle, and the high copper particles are the alloy Lojic not silver-copper eutectic as used in Dispersalloy. It would appear that these particles react to a greater extent than the silver-copper eutectic particles. The slight changes in the final mercury levels with increasing trituration time for Permite C could be considered to be the result of the aggregated effects of the separate constituents from which the amalgam alloy is composed.

The change in final mercury levels for the spherical particle alloys is not nearly as marked as for New True Dentalloy and Dispersalloy.

4.4 Scanning Electron Microscopy

Scanning electron microscopy was used to determine the effect of condensation pressure on the proportions of the phases present in a fully hardened lathe-cut amalgam (New True Dentalloy) and a spherical particle high copper amalgam (Lojic). In Table 4.4.1 the results obtained for New True Dentalloy are presented and the results for Lojic are shown in Table 4.4.2.

For Lojic, the extent of reaction as evinced by the proportion of unreacted alloy particles is virtually independent of condensation pressure up to 14MPa and exhibits only a small change when the pressure is further increased to 42MPa. New True Dentalloy, on the other hand, shows a marked increase in the proportion of unreacted alloy particles as the condensation pressure is increased, particularly in the range from 3MPa to 14MPa.

The spherical particle amalgams contained less porosity than the lathe-cut amalgams at all condensation pressures. In fact, the spherical particle amalgam condensed at the lowest condensation pressure (3MPa) contained less porosity than the lathe-cut alloy condensed at the highest condensation pressure (42MPa).

NEW TRUE DENTALLOY

Phases	Proportions %		
Original alloy	13.9	31.7	38.2
γ_1 (Ag_2Hg_3)	74.6	60.1	53.1
γ_2 (Sn_{7-8}Hg)	9.1	6.2	5.5
Porosity	2.5	1.9	3.3
Condensation Pressure	3MPa	14MPa	42MPa

Table 4.4.1

LOJIC

Phases	Proportions %		
Original alloy	26.0	28.6	34.38
γ_1	31.82	31.78	27.38
η' (Cu_6Sn_5)	9.08	6.8	6.1
reaction rim	31.92	32.1	30.66
Porosity	1.16	0.66	1.46
Condensation Pressure	3MPa	14MPa	42MPa

Table 4.4.2

CHAPTER 5

DISCUSSION

It is clear from this investigation that the compressive strength and dimensional change of some amalgams are very sensitive to condensation pressure and trituration time whereas others show very little dependence on these manipulative variables. At one end of the spectrum of sensitivity are the lathe-cut alloy New True Dentalloy and the admixed alloy Dispersalloy. Following condensation at low condensation pressures e.g. (3MPa, 7MPa), after 24 hours these amalgams displayed an expansion whereas at the very high condensation pressure of 42MPa, the sign of the dimensional change was reversed, and they clearly showed a contraction over the duration of the hardening reaction. Permite C, also an admixed alloy, showed a similar trend, in as much as it showed a large expansion (approx $15\mu\text{m cm}^{-1}$) at low condensation pressure but only a slight expansion at the very high condensation pressure.

With the non spherical alloys New True Dentalloy and Dispersalloy, departures from the manufacturers' recommended trituration time of 8 seconds also had a marked effect on dimensional change. For either shorter or longer trituration times both alloys displayed an expansion at 24 hours whereas specimens triturated according to the manufacturer's specification showed either a zero (New True Dentalloy) or slightly negative (Dispersalloy) dimensional change. At the shorter trituration times, (e.g. 2 seconds) mixing is inadequate and the amalgam expands during setting. As the trituration time is increased to 8 seconds mixing is improved and the expansion is reduced. Further increasing the trituration time results in amalgams which expand on setting due to the increased deformation

of the alloy particles during trituration.

Although there are some differences between the absolute values in dimensional change reported in the present investigation with those in a previous study of Ekstrand (Ekstrand et al 1985), it is clear that there is substantial agreement on the point that with Dispersalloy, dimensional change during hardening is sensitive to both trituration time and condensation pressure.

The dimensional change curves of the spherical particle high copper amalgam alloys on the other hand, are of a quite different shape and are comparatively insensitive to wide variations in either trituration time or condensation pressure. It should be noted that the admixed alloy Permite C, while little affected by trituration time, displays the same shape and marked sensitivity to condensation pressure which appears characteristic of the non spherical particle alloys.

Compressive strength and residual mercury content of lathe-cut and admixed alloys are also significantly affected by condensation pressure. A previous study (Clark et al 1981) suggested that lathe-cut and admixed alloys show large increases in compressive strength with increased condensation pressure, but the strength of spherical particle alloys was relatively insensitive to condensation pressure. This was confirmed in the present investigation.

Alterations in trituration times (within reasonable limits e.g. 5 - 16 seconds), had little effect on the compressive strength of any of the alloys, with the exception of Dispersalloy, which became overtrituated after 8 seconds as a consequence of using a pestle.

The increased condensability of spherical particle alloys particularly at low condensation pressures (Ohashi et al 1985) and lower initial mercury levels, account for the lower final mercury levels in the spherical amalgams at all condensation pressures and trituration times. The final mercury content of the lathe-cut and admixed alloys is very dependent on the condensation pressure but much less so on trituration time. The consequence of a higher final mercury content, as evinced by scanning electron microscopy, is an increase in the proportion of reaction products formed during hardening. This results in reduced strength (Okabe et al 1985) and increased creep rate and γ_2 formation (Mahler and Adey 1979).

It follows that the clinical performance of amalgams containing lathe-cut particles will also be affected by both trituration time and condensation pressure. With modern high energy amalgamators the trituration time can be readily controlled. However, the condensation pressure applied by the dental practitioner during the packing of an amalgam restoration is difficult to control because it is affected by the size and shape of the restoration, the position within the cavity being packed (e.g. cavity floor, walls or against a matrix band), the size and shape of the plugger, the fitness/tiredness of the operator and the comfort of the patient. Because of the sensitivity of lathe-cut amalgams, these variations in condensation pressure will result in an inhomogeneous restoration whose dimensional changes during hardening will vary from one point to another. This will lead to high levels of residual stress, and as a consequence a reduction in strength. It is quite clear, however, that spherical particle amalgams would not suffer from these problems because they are relatively insensitive to variations in condensation pressure and require much lower

condensation forces to achieve adequate strength.

CHAPTER 6

CONCLUSION

The results of this investigation show that the dimensional change and compressive strength of non spherical amalgam alloys are critically dependent on the condensation pressures and trituration times used. Spherical particle high copper alloys, on the other hand, are stronger and relatively insensitive to either of these variables.

Since the condensation pressure applied by dentists during the placement of an amalgam restoration has been reported to be considerably lower than the 14MPa specified in ADA Spec. No. 1 (Lussi and Buergin (1987); Rakow (1978)), the actual clinical properties of non-spherical amalgams may be significantly inferior to those predicted from measurements made in accordance with ADA Spec. No. 1. By contrast, the clinical properties of amalgams prepared from spherical particle high copper alloys, which are comparatively insensitive to manipulative variables, would be expected to be closer to those obtained in the laboratory using testing procedures specified by ADA No. 1.

Clearly the spherical particle high copper amalgam alloys tested have superior strength and are far less sensitive to manipulative variables (particularly condensation pressure) than either conventional lathe cut or admixed amalgam alloys. Consequently, the clinical use of spherical particle amalgam alloys would seem to be preferred over lathe-cut or admixed amalgam alloys.



APPENDIX 1
COMPUTER PROGRAMME

DUALDVM1

```
05 ! PROGRAMME DUALDVM1
10 ! TWO RIG DVM DATA COLLECTION
20 CLEAR
30 IMAGE "MV RIG1",X,K,2X,K,"DAYS",X,K,"HRS",X,K,
  "MIN",X,K,"SEC"
40 IMAGE 40X,"MV RIG2",X,K,2X,K,"DAYS",X,K,"HRS",X,K,
  "MIN",X,K,"SEC"
50 ENABLE KBD 1+4+32+64
60 GOSUB 1000 ! READY TIMER ROUTINE
70 DT1=99999999 @ DT2=99999999 @ J=0
80 GOSUB 1320 ! TO CHECK CURRENT STATUS OF RIGS
90 L=2
100 ON KEY# 14,"STOP" GOTO 250
110 IF NAME1$="DONE" THEN ON KEY# 1,"START
  RIG1"GOSUB 260 ! TO START RIG1
120 IF NAME1$ <> "DONE" THEN ON KEY# 1,"END
  RIG1"GOSUB 2300 ! FINISH RIG1
130 IF NAME2$="DONE" THEN ON KEY# 3,"START
  RIG2"GOSUB 1600 ! TO START RIG2
140 IF NAME2$ <> "DONE" THEN ON KEY# 3,"END
  RIG2"GOSUB 2550 ! FINISH RIG 2
150 ON KEY# 2,"PLOT" GOSUB 2800 ! TO PLOT
  DIMENSIONAL CHANGE DATA
160 KEY LABEL
170 IF L=1 THEN 90
180 IF NAME1$ <> "DONE" THEN 230
190 IF NAME2$ <> "DONE" THEN 230
200 KEY LABEL
210 GOTO 90
```



```
220 IF L=1 THEN CLEAR
230 IF L=1 THEN 90 ELSE K=0
240 IF K=1 THEN 230 ELSE 240
250 END
260 ! TO START RIG 1
270 OFF KEY# @ CLEAR
280 OFF TIMER# 1 @ OFF TIMER# 2
290 CLEAR
300 DISP "NAME OF RIG1 DATA FILE";
310 INPUT NAME1$
320 CREATE NAME1$,500,16
330 ASSIGN# 2 TO NAME1$
340 NP1=0 @ V5MIN1=0
350 TEST1=0
360 ON KEY# 14,"CRASH" GOSUB 900
370 IF TEST1=1 THEN 630
380 DT1=10000 ! TIMER INTERVAL INITIALLY 10 SECONDS
390 CLEAR
400 DISP "PRESS K1 TO START TIMER AT END OF
      TRITURATION"
410 ON KEY# 1,"TIMER" GOTO 440
420 KEY LABEL
430 IF TEST1=1 THEN 630 ELSE 430
440 OFF KEY# 1 @ CLEAR
450 STARTIME1=INT (TIME) @ STARTDATE1=DATE
460 CLEAR @ KEY LABEL
470 PRINT# 2,1 ; STARTIME1,STARTDATE1
480 PRINT# 2,2 ; NP1,DT1
490 ASSIGN# 2 TO *
```

```
500 ASSIGN# 3 TO "CURNT R1R2"
510 PRINT# 3,1 ; NAME1$
520 ASSIGN# 3 TO *
530 CLEAR
540 DISP "PRESS K2 WHEN SPECIMEN READY"
550 ON KEY# 2,"SP1 READY" GOTO 580
560 KEY LABEL
570 IF TEST1=1 THEN 630 ELSE 570
580 OFF KEY# 2 @ CLEAR
590 ON TIMER# 1,DT1 GOSUB 670 ! DVM DATA
    COLLECTION RIG1
600 IF TEST1=1 THEN 620 ELSE T1=INT (TIME)-
    STARTIME1+(DATE-STARTDATE1)*86400
610 IF T1<330 THEN 600
620 IF NAME2$ <> "DONE" THEN ON TIMER# 2,DT2 GOSUB
    1970 ! DVM DATA COLLECTION RIG2
630 TEST1=0
640 L=1 @ K=1
650 CLEAR
660 RETURN
670 ! DVM DATA COLLECTION ROUTINE RIG1
680 IF NAME1$="DONE" THEN 870
690 OFF TIMER# 1
700 DT1=INT (DT1*1.02) @ DT1=MIN (DT1,99999999)
710 T1=INT (TIME )-STARTIME1+(DATE -STARTDATE1)*
    86400
720 OUTPUT 809 ;"DCV01"
730 ENTER 809 ; V1
740 V1=V1*1000
```

```
750 ASSIGN# 1 TO NAME1$ @ NP1=NP1+1 @ PRINT# 1,
      NP1+5 ; T1,V1 @ PRINT# 1,2 ; NP1,DT1
760 ASSIGN# 1 TO *
770 IF V5MIN1<> 0 THEN 830
780 IF T1<300 THEN 820
790 IF NP1=1 THEN V5MIN1=V1 @ GOTO 810
800 V5MIN1=LV1+(300-LT1)*(V1-LV1)/(T1-LT1)
810 ASSGIN# 1 TO NAME1$ @ PRINT# 1,3 ; 10,V5MIN1 @
      ASSIGN# 1 TO *
820 LV1=V1 @ LT1=T1
830 TM1=INT (T1/60) @ TS1=T1-TM1*60
840 TH1=INT (TM1/60) @ TM1=TM1-TH1*60
850 TD1=INT (TH1/24) @ TH1=TH1-TD1*24
860 DISP USING 30 ; V1,TD1,TH1,TM1,TS1
870 L=2 @ K=1
880 ON TIMER# 1,DT1 GOSUB 670
890 RETURN
900 ! TO CRASH PROGRAM RIG1
910 OFF TIMER# 1 @ OFF KEY#
920 ASSIGN# 3 TO "CURNT R1R2"
930 PRINT# 3,1 ; "DONE"
940 ASSIGN# 3 TO *
950 PURGE NAME1$
960 NAME1$="DONE"
970 TEST1=1
980 L=1 @ K=1
990 RETURN
1000 ! READY TIMER ROUTINE
1010 DIM MONTH(12)
```

```
1020 FOR I=1 TO 12 @ READ MONTH(I)@ NEXT I
1030 DATA 31,28,31,30,31,30,31,31,30,31,30,31
1040 GOSUB 1180 ! CURRENT TIME ROUTINE
1050 DISP THMS,DAY;MNTH;YR
1060 IF DATE <82000 THEN 1310
1070 DISP "TIME";THMS;" ";DAY;MNTH;YR
1080 DISP "DATE: DAY, MONTH,YEAR" @ INPUT
      DAY,MNTH,YR
1090 IF YR>1900 THEN YR=YR-1900
1100 IF YR MOD 4=0 THEN MONTH(2)=29
1110 DAYS=YR*1000+DAY
1120 FOR I=1 TO MNTH-1 @ DAYS=DAYS+MONTH(I) @
      NEXT I
1130 DISP "TIME;HRS,MIN,SECS FROM MIDNIGHT";
1140 INPUT THS,TMS,TSS
1150 SETTIME THS*3600+TMS*60+TSS,DAYS
1160 THMS=THS*100+TMS+TSS/1000
1170 RETURN
1180 ! CURRENT TIME ROUTINE
1190 YR=INT (DATE /1000) @ DAY=DATE -YR*1000
1200 FOR I=1 TO 12
1210 DAY=DAY-MONTH(I)
1220 IF DAY<= 0 THEN DAY=DAY+MONTH(I) @ GOTO 1240
1230 NEXT I
1240 IF I<= 12 THEN 1280
1250 YR=YR+1
1260 IF YR MOD 4=0 THEN MONTH(2)=29 ELSE
      MONTH(2)=28
1270 GOTO 1200
```

```
1280 MNTH=I
1290 THS=INT (TIME /3600) @ TMS=INT (TIME /60)-THS*60
    @ TSS=INT (TIME )-THS*3600-TMS*60
1300 THMS=THS*100+TMS+TSS/100
1310 RETURN
1320 ! TO CHECK THE CURRENT STATUS OF BOTH RIGS
1330 ASSIGN# 1 TO "CURNT R1R2"
1340 READ# 1,1 ; NAME1$ ! FOR RIG NUMBER 1
1350 READ# 1,2 ; NAME2$ ! FOR RIG NUMBER 2
1360 ASSIGN# 1 TO *
1370 ! CURRENT STATUS OF RIG1
1380 IF NAME1$="DONE" THEN 1480
1390 ASSIGN# 1 TO NAME1$
1400 READ# 1,1 ; STARTIME1,STARTDATE1
1410 READ# 1,2 ; NP1,DT1
1420 READ# 1,3 ; LENGTH1,V5MIN1
1430 CLEAR
1440 IF NP1>= 600 THEN DISP "MORE THEN 600 READINGS
    - DATA NOT STORED" @ GOTO 1480
1450 READ# 1,NP1+5 ; LASTT1
1460 ASSIGN# 1 TO *
1470 ON TIMER# 1,DT1 GOSUB 670
1480 ! CURRENT STATUS OF RIG2
1490 IF NAME2$="DONE" THEN 1590
1500 ASSIGN# 1 TO NAME2$
1510 READ# 1,1 ; STARTIME2,STARTDATE2
1520 READ# 1,2 ; NP2,DT2
1530 READ# 1,3 ; LENGTH2,V5MIN2
1540 CLEAR
```

```
1550 IF NP2>= 600 THEN DISP "MORE THEN 600
      READINGS - DATA NOT STORED" @ GOTO 1590
1560 READ# 1,NP2+5 ; LASTT2
1570 ASSIGN# 1 TO *
1580 ON TIMER# 2,DT2 GOSUB 1970
1590 RETURN
1600 ! TO START RIG 2
1610 OFF KEY# @ OFF TIMER# 1 @ OFF TIMER# 2 @
      CLEAR
1620 CLEAR
1630 DISP "NAME OF RIG2 DATA FILE";
1640 INPUT NAME2$
1650 CREATE NAME2$,500,16
1660 ASSIGN# 4 TO NAME2$
1670 NP2=0 @ V5MIN2=0
1680 TEST2=0
1690 ON KEY# 14,"CRASH" GOSUB 2200
1700 IF TEST2=1 THEN 1940
1710 DT2=10000 ! TIMER INTERVAL INITIALLY 10 SECONDS
1720 CLEAR
1730 DISP "PRESS K1 TO START TIMER AT END OF
      TRITURATION"
1740 ON KEY# 1,"TIMER" GOTO 1770
1750 KEY LABEL
1760 IF TEST2=1 THEN 1940 ELSE 1760
1770 OFF KEY# 1
1780 STARTIME2=INT (TIME ) @ STARTDATE2=DATE
1790 PRINT# 4,1 ; STARTIME2,STARTDATE2
1800 PRINT# 4,2 ; NP2,DT2
```

```
1810 ASSIGN# 4 TO *
1820 ASSIGN# 3 TO 'CURNT R1R2"
1830 PRINT# 3,2 ; NAME2$
1840 ASSIGN# 3 TO * @ CLEAR @ KEY LABEL
1850 DISP "PRESS K2 WHEN SPECIMEN READY"
1860 ON KEY# 2,"SP2 READY" GOTO 1890
1870 KEY LABEL
1880 IF TEST2=1 THEN 1940 ELSE 1880
1890 OFF KEY# 2 @ CLEAR
1900 ON TIMER# 2,DT2 GOSUB 1970 ! DVM DATA
    COLLECTION RIG2
1910 IF TEST2=1 THEN 1930 ELSE T2=INT (TIME )-
    STARTIME2+(DATE -STARTDATE2)*86400
1920 IF T2<330 THEN 1910
1930 IF NAME1$ <> "DONE" THEN ON TIMER# 1,DT1 GOSUB
    670 ! DVM DATA COLLECTION RIG1
1940 L=1 @ TEST2=0 @ K=1
1950 CLEAR
1960 RETURN
1970 ! DVM DATA COLLECTION ROUTINE RIG2
1980 OFF TIMER# 2
1990 DT2=INT (DT2*1.02) @ DT2=MIN (DT2,99999999)
2000 IF NAME2$="DONE" THEN 2190
2010 T2=INT (TIME )-STARTIME2+(DATE -STARTDATE2)
    *86400
2020 OUTPUT 809 ;"DCV02"
2030 ENTER 809 ; V2
2040 V2=V2*1000
2050 ASSIGN# 6 TO NAME2$ @ NP2=NP2+1 @ PRINT#
```

```
        6,NP2+5 ; T2,V2 @ PRINT# 6,2 ; NP2,DT2
2060 ASSIGN# 6 TO *
2070 IF V5MIN2<> 0 THEN 2130
2080 IF T2<300 THEN 2120
2090 IF NP2=1 THEN V5MIN2=V2 @ GOTO 2110
2100 V5MIN2=LV2+(300-LT2)*(V2-LV2)/(T2-LT2)
2110 ASSIGN# 6 TO NAME2$ @ PRINT# 6,3 ; 10,V5MIN2
        @ ASSIGN# 6 TO *
2120 LV2=V2 @ LT2=T2
2130 TM2=INT (T2/60) @ TS2=T2-TM2*60
2140 TH2=INT (TM2/60) @ TM2=TM2-TH2*60
2150 TD2=INT (TH2/24) @ TH2=TH2-TD2*24
2160 DISP USING 40 ; V2,TD2,TH2,TM2,TS2
2170 ON TIMER# 2,DT2 GOSUB 1970
2180 L=2 @ K=1
2190 RETURN
2200 ! TO CRASH PROGRAM RIG2
2210 ASSIGN# 3 TO "CURNT R1R2"
2220 PRINT# 3,1 ; "DONE"
2230 ASSIGN# 3 TO *
2240 PURGE NAME2$
2250 NAME2$="DONE"
2260 TEST2=1
2270 OFF TIMER# 2 @ OFF KEY#
2280 L=1 @ K=1
2290 RETURN
2300 ! TO FINISH RIG1
2310 OFF TIMER# 1 @ OFF TIMER# 2
2320 ASSIGN# 2 TO "CURNT R1R2"
```



```
2330 READ# 2,1 ; NAME1$
2340 PRINT# 2,1 ; "DONE"
2350 ASSIGN# 2 TO *
2360 ASSIGN# 2 TO NAME1$
2370 READ# 2,3 ; LENGTH1,V5MIN1
2380 READ# 2,2 ; NP1,DT1
2390 CLEAR
2400 OUTPUT 809 ;"DCV01"
2410 ENTER 809 ; V1
2420 V1=V1*1000
2430 NP1=NP1+1
2440 T1=INT (TIME )-STARTIME1+(DATE -STARTDATE1)
      *86400
2450 PRINT# 2,NP1+5 ; T1,V1 @ PRINT# 2,2 ; NP1
2460 ASSIGN# 2 TO *
2470 DISP "LENGTH OF SPECIMEN ";NAME1$;" IN MMS"; @
      INPUT LENGTH1
2480 PRINT# 2,3 ; LENGTH1,V5MIN1
2490 ASSIGN# 2 TO *
2500 DISP "DONE1"
2510 NAME1$="DONE"
2520 L=1 @ K=1
2530 IF NAME2$ <> "DONE" THEN ON TIMER# 2,DT2 GOSUB
      1970
2540 RETURN
2550 ! TO FINISH RIG2
2560 OFF TIMER# 2 @ OFF TIMER# 1
2570 ASSIGN# 2 TO "CURNT R1R2"
2580 READ# 2,2 ; NAME2$
```

```
2590 PRINT# 2,2 ; "DONE"
2600 ASSIGN# 2 TO *
2610 ASSIGN# 2 TO NAME2$
2620 READ# 2,3 ; LENGTH2,V5MIN2
2630 READ# 2,3 ; NP2,DT2
2640 CLEAR
2650 OUTPUT 809 ;"DCV02"
2660 ENTER 809 ; V2
2670 V2=V2*1000
2680 NP2=NP2+1
2690 T2=INT (TIME )-STARTIME2+(DATE -STARTDATE2)
      *86400
2700 PRINT# 2,NP2+5 ; T2,V2 @ PRINT# 2,2 ; NP2
2710 DISP "LENGTH OF SPECIMEN ";NAME2$;" IN MMS"; @
      INPUT LENGTH2
2720 PRINT# 2,3 ; LENGTH2,V5MIN2
2730 ASSIGN# 2 TO *
2740 DISP "DONE2"
2750 NAME2$="DONE"
2760 L=1 @ K=1
2770 IF NAME1$ <> "DONE" THEN ON TIMER# 1,DT1 GOSUB
      670
2780 RETURN
2790 ! TO PLOT THE DIMENSIONAL CHANGE DATA
2800 L=1 @ K=1
2810 OFF TIMER# 1 @ OFF TIMER# 2
2820 OFF KEY# @ CLEAR
2830 DIM TP(600),VP(600),PLOTNAME$[20],FACTOR(20),
      TF(600),VF(600)
```

```
2840 ON KEY# 2,"STOP PLOT" GOTO 4760
2850 KEY LABEL
2860 DISP "TO STOP THE PLOT AT ANY TIME PRESS KEY K2"
2870 DISP "IS HP PLOTTER TO BE USED?(Y/N)";@ INPUT HP$
2880 IF HP$ <> "Y" THEN GOSUB 3770
2890 IF HP$="Y" THEN GOSUB 2910
2900 RETURN
2910 TO PLOT DIMENSIONAL CHANGE USING DUALDVM
PROGRAM ON HP PLOTTER
2920 DISP "TO STOP THE PLOT AT ANY TIME PRESS KEY K2"
2930 DISP "TURN ON PLOTTER AND LOAD PAPER"
2940 DISP "WHEN THE PLOTTER IS READY PRESS THE 'SPACE
BAR'AND'RETURN'";
2950 DISP "TO CONTINUE";
2960 INPUT A$
2970 PLOTTER IS 805 @ CLEAR
2980 GOSUB 3180 ! TO READ FILENAMES
2990 DISP "TITLE FOR PLOT";@ INPUT TITLE$
3000 DISP "U/CM: min,max,tick interval, no. of ticks per label";
3010 INPUT Umin,Umax,Utick,Umajor
3020 DISP "HOURS: min,max,tick interval, no. of ticks per label";
3030 INPUT Tmin,Tmax,Ttick,Tmajor
3040 GOSUB 3570 ! TO SET UP AXES
3050 FOR NO=1 TO NPLOTS
3060 PLOTNO=NO
3070 GOSUB 3260 ! TO READ DATA FILES
3080 GOSUB 3370 ! TO PLOT DATA
3090 NEXT NO
3100 OUTPUT 805 ;"PG;"
```

```
3110 ! TO STOP PLOT OF DIMENSIONAL CHANGE DATA
3120 OFF KEY# @ CLEAR
3130 PLOTTER IS 1 @ ALPHALL
3140 DISP "END NO FURTHER PLOTS TO BE DONE";
3150 ON TIMER# 1,DT1 GOSUB 670 @ ON TIMER# 2,DT2
      GOSUB 1970 ! DVM DATA COLLECTION      ROUTINES
3160 L=1 @ K=1
3170 RETURN
3180 ! TO READ FILENAMES
3190 DISP "INPUT NUMBER OF PLOTS";@ INPUT NPLOTS
3200 IF NPLOTS<1 THEN GOTO 3130
3210 FOR I=1 TO NPLOTS
3220 DISP "PLOT";I;"FILENAME,PLOTNAME SCALE FACTOR
      RELATIVE TO u/CM AXIS";
3230 INPUT FILENAME$(I),PLOTNAME$(I),FACTOR(I)
3240 NEXT I
3250 RETURN
3260 ! TO READ FILEDATA
3270 ASSIGN# 1 TO FILENAME$(NO)
3280 READ# 1,2 ; NPTS
3290 READ# 1,3 ; LENGTH,V5MIN
3300 FOR I=1 TO NPTS
3310 READ# 1,I+5 ; TP(I),VP(I)
3320 VF(I)=(VP(I)-V5MIN)*FACTOR(NO)*1.27/LENGTH
3330 TF(I)=TP(I)/3600
3340 NEXT I
3350 ASSIGN# 1 TO *
3360 RETURN
3370 ! TO PLOT THE DIMENSIONAL CHANGE DATA
```

```
3380  SETUU
3390  OUTPUT 805 ;"SP";PLOTNO+1;""
3400  OUTPUT 805 ;"LT";8-PLOTNO;""
3410  MOVE TF(1),VF(1)
3420  FOR J=1 TO NPTS
3430  DRAW TF(J),VF(J)
3440  IF J=NPTS THEN GOTO 3460
3450  NEXT J
3460  ! TO SET THE LABEL FOR THE PLOT NAME
3470  SETGU @ DEG
3480  MOVE 105,87-(PLOTNO-1)*3
3490  DRAW 120,87-(PLOTNO-1)*3
3500  OUTPUT 805 ;"IN;SP1;"
3510  MOVE 123,87-(PLOTNO-1)*3
3520  CSIZE 2
3530  LORG 2
3540  LDIR 0 @ LABEL PLOTNAME$(NO)
3550  OUTPUT 805 ;"SPO;"
3560  RETURN
3570  ! TO SET UP AXES
3580  OUTPUT 805 ;"IN;IP;SP1;"
3590  FRAME
3600  SETGU
3610  LOCATE 15,130,15,90 ! LOCATES PLOTTING
    BOUNDARIES IN GU
3620  SCALE Tmin,Tmax,Umin,Umax
3630  CSIZE 3
3640  LAXES Ttick,Utick,Tmin,Umin,Tmajor,Umajor
3650  AXES Ttick,0,Tmin,0,Tmajor,0
```

```
3660 SETGU @ DEG
3670 MOVE 5,90
3680 LORG 7
3690 LDIR 90 @ LABEL "U/CM"
3700 MOVE 130,5
3710 LORG 7
3720 LDIR 0 @ LABEL "HOURS"
3730 MOVE 100,90
3740 LORG 7 @ CSIZE 6
3750 LABEL TITLE$
3760 RETURN
3770 ! TO PLOT DIMENSIONAL CHANGE USING DUAL DVM
PROGRAM ON SCREEN
3780 CLEAR
3790 ! SET UP FOR PLOT ROUTINE
3800 L1=20 @ L2=200 @ L3=15 @ L4=90
3810 DISP "1) AT COMPLETION OF PLOTTING TO STORE THE
PLOT AND RETURN TO"
3820 DISP " DATA COLLECTION PRESS KEY K9"
3830 DISP "2) To abandon plotting at any time or to return to
data"
3840 DISP " collection at any time press key K2"
3850 ON KEY# 9,"STORE PLOT" GOTO 4020
3860 DISP "Add to LAST PLOT (Y/N)";@ INPUT B$
3870 IF B$ <> "Y" THEN DISP "TITLE FOR PLOT";@ INPUT
TITLE$
3880 GOSUB 4110 ! TO READ FILENAMES
3890 IF B$="Y" THEN GOSUB 4660 ! TO READ LAST PLOT
DATA
```

```
3900 IF B$="Y" THEN 3960
3910 DISP "U/CM: min,max,tick interval, no of ticks per label"
3920 INPUT Umin,Umax,Utick,Umajor
3930 DISP "HOURS: min, max, tick interval, no. of ticks per
label";
3940 INPUT Tmin,Tmax,Ttick,Tmajor
3950 GOSUB 4460 ! TO SET UP AXES
3960 FOR NO=1 TO NPLOTS
3970 PLOTNO=NO
3980 GOSUB 4190 ! TO READ DATA FILES
3990 GOSUB 4300 ! TO PLOT DIMENSIONAL CHANGE DATA
4000 NEXT NO
4010 WAIT 300000 @ GOTO 4070
4020 OFF KEY# 9
4030 ASSIGN# 4 TO "LAST PLT D"
4040 PRINT# 4 ; Tmin,Tmax,Umin,Umax
4050 ASSIGN# 4 TO *
4060 GSTORE "LAST PLOT"
4070 ON TIMER# 1,DT1 GOSUB 670 @ ON TIMER# 2, DT2
GOSUB 1970 ! DVM DATA COLLECTION ROUTINES
4080 ALPHALL @ CLEAR @ OFF KEY# ! RETURNS TO ALPHA
MODE
4090 L=1 @ K=1
4100 RETURN
4110 ! TO READ FILENAMES
4120 DISP "INPUT NUMBER OF PLOTS";@ INPUT NPLOTS
4130 IF NPLOTS<1 THEN GOTO 4070
4140 FOR I=1 TO NPLOTS
4150 DISP "PLOT";I;"FILENAME,PLOTNAME,SCALE FACTOR
```

```
RELATIVE TO u/CM AXIS";
4160 INPUT  FILENAME$(I),PLOTNAME$(I),FACTOR(I)
4170 NEXT I
4180 RETURN
4190 ! TO READ DATA FILES
4200 ASSIGN# 1 TO FILENAME$(NO)
4210 READ# 1,2 ; NPTS
4220 READ# 1,3 ; LENGTH,V5MIN
4230 FOR I=1 TO NPTS
4240 READ# 1,I+5 ; TP(I),VP(I)
4250 VF(I)=(VP(I)-V5MIN)*FACTOR(NO)*1.27/LENGTH
4260 TF(I)=TP(I)/3600
4270 NEXT I
4280 ASSIGN# 1 TO *
4290 RETURN
4300 ! TO PLOT THE DIMENSIONAL CHANGE DATA
4310 SETUU
4320 LINE TYPE NO ! CHANGES LINE TYPE FOR EACH PLOT
4330 MOVE TF(1),VF(1)
4340 FOR J=1 TO NPTS
4350 IF TF(J)>=Tmax THEN 4390
4360 DRAW TF(J),VF(J)
4370 IF J=NPTS THEN GOTO 4390
4380 NEXT J
4390 ! TO SET THE LABEL FOR THE PLOT NAME
4400 SETGU @ DEG
4410 WHERE X,Y @ MOVE X+1,Y+1
4420 CSIZE 4
4430 LORG 2
```



```
4440 LDIR 0 @ LABEL PLOTNAME$(NO)
4450 RETURN
4460 ! TO SET UP AXES
4470 GRAPHALL @ CLEAR
4480 FRAME
4490 SETGU
4500 LOCATE L1,L2,L3,L4 ! LOCATES PLOTTING BOUNDARIES
      IN GU
4510 SCALE Tmin,Tmax,Umin,Umax
4520 CSIZE 6
4530 LAXES Ttick,Utick,Tmin,Umin,Tmajor,Umajor
4540 AXES Ttick,0,Tmin,0,Tmajor,0
4550 SETGU @ DEG
4560 MOVE 5,90
4570 LORG 7
4580 LDIR 90 @ LABEL "U/CM"
4590 MOVE 130,5
4600 LORG 7
4610 LDIR 0 @ LABEL "HOURS"
4620 MOVE 100,90
4630 LORG 7
4640 LABEL TITLE$
4650 RETURN
4660 ! TO READ LAST PLOT DATA
4670 ASSIGN# 4 TO "LST PLT D"
4680 READ# 4 ; Tmin,Tmax,Umin,Umax
4690 ASSIGN# 4 TO *
4700 SETGU
4710 GRAPHALL @ CLEAR
```

```
4720 LOCATE L1,L2,L3,L4
4730 SCALE Tmin,Tmax,Umin,Umax
4740 GLOAD "LAST PLOT"
4750 RETURN
4760 ! TO STOP DIMENSIONAL CHANGE PLOT
4770 PLOTTER IS 1
4780 ALPHALL @ CLEAR @ OFF KEY#
4790 ON TIMER# 1,DT1 GOSUB 670 ! RIG1 DVM DATA
      COLLECTION
4800 ON TIMER# 2,DT2 GOSUB 1970 ! G=RIG2 DVM DATA
      COLLECTION
4810 L=1 @ K=1
4820 RETURN
```

APPENDIX 2
COMPUTER PROGRAMME

AVERAGE

```
05 ! PROGRAMME AVERAGE
10 DIM  FILENAME$(15),LENGTH(15),V5MIN(15),V(15),
    LTT(15),LVV(15),NPTS(15)
20 DIM  FT(300),FV(300),S(300),NP(15),DV(180,15),
    DT(180,15),L(15)
30 DISP "Number of runs to be averaged"
40 INPUT N
50 ST=0
60 NT=1
70 FOR I=1 TO N
80 DISP "Filename?"
90 INPUT  FILENAME$(I)
100 ASSIGN# 1 TO FILENAME$(I)
110 READ# 1,3 ; LENGTH(I),V5MIN(I)
120 READ# 1,2 ; NPTS(I)
130 READ# 1,6 ; TT,VV
140 LTT(I)=TT @ LVV(I)=VV
150 IF ST<TT THEN ST=TT @ NT=I
160 NP(I)=1
170 NEXT I
180 DISP "File name of average?NAME MUST START WITH A TO
    USE 'ERROR BARS'PROG"
190 INPUT AVNAME$
200 FOR I=1 TO N
210 ASSIGN# 1 TO FILENAME$(I)
220 TOP=MIN (180,NPTS(I))
230 FOR J=1 TO TOP
240 READ# 1,5+J ; DT(J,I),DV(J,I)
250 NEXT J
```

```
260 L(I)=0
270 NEXT I
280 ! USE TIME INTERVALS FROM FILENAME$(NT)
290 ANPTS=1
300 AML=ANPTS-L(NT)
310 IF AML<= 180 THEN 390
320 ASSIGN# 1 TO FILENAME$(NT)
330 TOP=MIN (180,NPTS(NT)-L(NT)-180)
340 FOR J=0 TO TOP
350 READ# 1,185+J+L(NT) ; DT(J,NT),DV(J,NT)
360 NEXT J
370 L(NT)=L(NT)+180
380 GOTO 300
390 T=DT(AML,NT)
400 FOR I=1 TO N
410 NPML=NP(I)-L(I)
420 IF NPML<= 180 THEN 500
430 ASSIGN# 1 TO FILENAME$(I)
440 TOP=MIN (180,NPTS(I)-L(I)-180)
450 FOR J=0 TO TOP
460 READ# 1,185+J+L(I) ; DT(J,I),DV(J,I)
470 NEXT J
480 L(I)=L(I)+180
490 GOTO 410
500 TT=DT(NPML,I) @ VV=DV(NPML,I)
510 IF TT>= T THEN 560
520 LVV(I)=VV @ LTT(I)=TT
530 NP(I)=NP(I)+1
540 IF NP(I)>NPTS(I) THEN 740
```

```
550 GOTO 410
560 IF T=LTT(I) THEN V(I)=VV @ GOTO 580
570 V(I)=LVV(I)+(T-LTT(I))*(VV-LVV(I))/(T-LTT(I))
580 NEXT I
590 SSQ=0
600 AV=0
610 FOR I=1 TO N
620 V(I)=10*(V(I)-V5MIN(I))/LENGTH(I)
630 AV=V(I)+AV
640 SSQ=V(I)*V(I)+SSQ
650 NEXT I
660 AV=AV/N
670 S(ANPTS)=SQR ((SSQ-N*AV*AV)/(N-1))
680 FT(ANPTS)=T @ FV(ANPTS)=AV
700 DISP ANPTS,T,AV,S(ANPTS)
705 ANPTS=ANPTS+1
710 IF ANPTS>NPTS(NT) THEN 740
720 GOTO 300
740 AGGISN# 1 TO *
750 ANPTS=ANPTS-1
760 DISP "Insert storage disc then press END LINE";
770 INPUT A$
780 CREATE AVNAME$,ANPTS+10,24
790 ASSIGN# 10 TO AVNAME$
800 PRINT# 10,3 ; 10,0
810 PRINT# 10,2 ; ANPTS,0
820 PRINT# 10,1 ; 0,0
830 FOR I=1 TO ANPTS
840 PRINT# 10,5+I ; FT(I),FV(I),S(I)
```

```
850 NEXT I
860 ASSIGN# 10 TO *
870 DISP "finished"
880 STOP
```

APPENDIX 3

PUBLISHED PAPER

A DILATOMETER FOR MEASURING THE DIMENSIONAL
CHANGE IN DENTAL AMALGAMS

APPENDIX 3PUBLISHED PAPERA DILATOMETER FOR MEASURING THE DIMENSIONAL
CHANGE IN DENTAL AMALGAMS

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ABSTRACT

A counterbalanced dilatometer suitable for the determination of small dimensional changes which occur during the hardening of dental amalgam is described. The instrument can operate without loss of sensitivity or accuracy with the specimen under loads as low as 20mg. The instrument has been designed to enable measurements of dimensional change to be commenced 45 seconds after the preparation of the specimen of amalgam.

INTRODUCTION

The amalgam used by dentists to fill teeth is prepared by mixing or "trituration" carefully proportioned quantities of liquid mercury and finely divided powder of a silver-tin alloy. The components are usually contained in a small plastic capsule which is vibrated for a period of some 8-10 seconds in a high frequency (70hz) mechanical vibrator.

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What emerges from the capsule at the end of this period is a soft pliable ball of material which can be inserted into the prepared cavity in the patient's tooth. But the amalgam rapidly hardens. After two or three minutes it is no longer pliable and after 5 minutes it is hard enough to be carved with a sharp tool to make the surface of the restoration conform to the tooth anatomy. Ten or fifteen minutes later this is no longer possible and any further modifications to the surface must be carried out by grinding. Four or five hours after trituration the strength and hardness are sufficient to permit the restoration to withstand normal masticatory stresses but full hardness appears not to be achieved for some days.

As the hardening reaction proceeds the amalgam may undergo a series of small but significant dimensional changes which may be either negative (i.e. the material contracts) or positive (i.e. the material expands). These changes in dimension may have clinical importance and indeed, commercial dental amalgams are required to conform to standards of dimensional change which are normally measured using a test cylinder of amalgam approximately 10mm long prepared by compaction (or "condensation") in accordance with widely accepted standardised procedures. Typical standards (Aust. Standard Spec. AS2110-1977, I.S.O./D.I.S. 1559) require that the preparation of the test cylinder should be completed and the cylinder loaded into the dilatometer in times such that the first measurements of dimensional change are made five minutes after the end of trituration and that the dimensional change after seven days should not exceed $\pm 20\mu\text{m}$ per centimetre. This imposes some limitations on the design of the equipment used to monitor dimensional change.

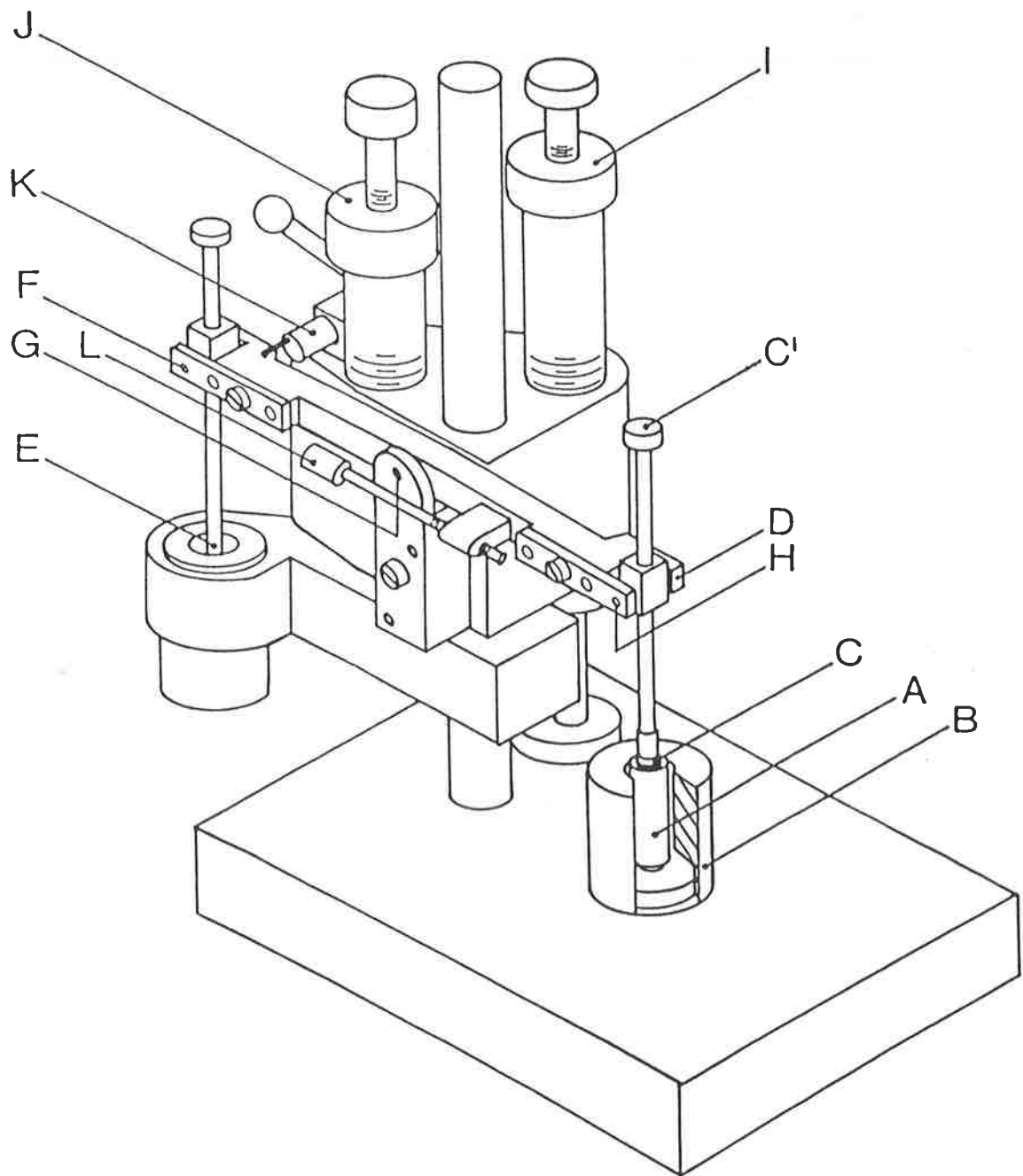
There is now considerable interest in the reaction mechanism occurring during the first few minutes of hardening. Accurate monitoring of dimensional change yields valuable information on these early mechanisms, and thus monitoring of dimensional change as soon as possible after the end of trituration is being called for.

This makes more stringent demands on the design of the dilatometer. Moreover, since it has been established (G.Wing, Aust.Dent.J., 1964, 9, 426) that the load applied to the cylindrical test specimen by the measuring instrument itself may have an influence on the magnitude of the dimensional change, it was necessary to make provision for operation of the instrument under specimen loads down to 20mg while maintaining the sensitivity such that changes in length of the order $\pm 0.01\mu\text{m}$ could be readily detected.

Construction and Operation

The general arrangement of the dilatometer is illustrated in Figure 1. The cylindrical specimen A of condensed amalgam, the dimensional change of which is to be monitored, is placed in a stainless steel stand B. The stand is then centrally located beneath the sensing probe C attached to one end of the beam D. The other end of the beam carries the core of the LVDT E. The beam D is symmetrical, and is suspended by a carbon steel pivot-shaft pressed into its centre. The pivots fit into 0.19mm synthetic ruby watch jewels. Similar jewelled bearings are used at F and H. The design eliminates the need for extremely close tolerances between the pivots and the jewelled bearings as the pivots are always at the lowest position in the bearings due to gravity. Height

Figure 1. The general arrangement
of the dilatometer.



adjustments of the LVDT and the beam are made by means of differential pitch lead screws I and J to facilitate the initial alignment and zeroing of the apparatus. When correctly zeroed the beam is horizontal. The sensing probe can be lowered onto the surface of the specimen or lifted clear of it by means of the lever operated cam K. The balancing screw L, attached to the beam permits the nett load applied to the specimen by the measuring system to be accurately controlled over wide limits. The magnitude of this nett load can be determined directly by placing a pan balance under the sensing probe C. Calibration of the dilatometer, the LVDT and the electronic/microprocessor data logging system can be readily carried out with the aid of a micrometer head mounted so that it makes contact with the upper end C' of the rod carrying the specimen probe. Calibration tests carried out in this way indicated the LVDT was linear over its entire range of $\pm 1.25\text{mm}$ and the displacement could be readily resolved to an accuracy of $\pm 0.01\mu\text{m}$. The dilatometer was housed in a constant temperature enclosure and to eliminate the influence of extraneous vibrations, this was placed on a concrete slab which was mechanically isolated from its surroundings by air-bag supports.

The mechanical and electrical stability of the whole system was such that measurements of the length of an invar test specimen over a period of 7 days showed variations less than $\pm 0.01\mu\text{m}$.

The equipment has proved to be completely reliable and it is sufficiently quick and convenient to operate that measurements of dimensional change in standard test specimens of dental amalgam can be commenced on a routine bases, within 45 seconds of their

preparation.

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