

Younis, Asgar and Powers (1975) considered the initiation of cracks through amalgam in relation to microstructural features. The two-phase composite beam first considered by Asgar and Sutfin (1965) was used to test four amalgam alloys, including Dispersalloy, and a Flinn Strain Viewer was used to monitor crack initiation and progress. In addition, these authors tested beams of different ages (2 days and 4 months) to examine the effect, if any, of time on crack initiation. It was observed that, like findings of previous studies, the crack initiated in the gamma 2 phase if this phase was present, and further crack propagation was stopped on encountering a gamma particle. In Dispersalloy, the crack initiated in the Ag-Hg phase or the Ag-Sn phase. The Ag-Cu phase proved to be most resistant to fracture and in all cases, crack initiation force was dependent on specimen age.

Okabe et al (1978) explored the effect of microstructural characteristics of amalgams of mechanical properties including fracture resistance. Specimens made from ten different amalgams were fractured either immediately after condensation or a few days after condensation. Mechanical properties were found to be enhanced by dispersed Cu-Sn crystals and fracture resistant Ag-Cu particles. In high copper ternary amalgams, Cu-Sn crystals ($2\mu\text{m}$) dispersed in the gamma 1 matrix were associated with transgranular (cleavage) fracture or 'round particle' fracture of gamma 1 particles. In Copper Addition amalgams, where the size of such Cu-Sn particles was less than $1\mu\text{m}$, no change in fracture mode was possible.

Vaidyanathan and Schulman (1979) examined microstructural characteristics of a range of amalgams tested to failure under different strain rates using SEM micrographs of the fracture

surface. Possible failure modes under compression in dental amalgams were listed as that occurring :

- (i) Through unreacted gamma particles.
- (ii) Through matrix phases of gamma 1 and gamma 2.
- (iii) Through Ag-Cu eutectic particles in Dispersalloy.

These workers found a significant dependence of amalgam failure mode on composition, particle size and particle shape at low strain rates and two distinct types of failure were observed :

- (i) Catastrophic failure by crack propagation through the gamma/gamma 1 interface. This type of failure occurred in High Copper amalgams at all strain rates but only at higher strain rates (0.05 in/min and 0.02 in/min) for Conventional Ag₃Sn amalgams.
- (ii) No sign of catastrophic failure with continuous plastic deformation, or significant plastic deformation prior to fracture. This was typical of Conventional Ag₃Sn amalgams at low strain rates (0.005 in/min and 0.002 in/min).

The importance of dislocations in amalgam deformation was suggested by the significant amount of plastic deformation evident in many amalgams at slow compressive strain rates and with no resulting catastrophic failure. The authors also suggested that plastic deformation of gamma 2 could not alone account for the extent of plastic flow observed.

The work of Giblin (1979) and Giblin and Wing (1979ab, 1980, 1981, 1982, 1983) encompassed the study of amalgam failure when subjected to static transverse sub-fracture loads over time. Beam specimens were tested using three-point loading at loads equal to defined percentages of the respective transverse strengths of the

individual amalgams. Bending and times to fracture of specimens were correlated to mechanical properties of amalgams, especially creep. Bending of specimens was found to precede crack initiation and subsequent failure by fracture. Amalgams of highest creep were found to bend more than low creep amalgams prior to fracture. Low creep amalgams (of creep less than 0.2%) were found to resist both significant plastic deformation and fracture. Using optical microscopy, etched, mounted specimens were examined for microstructural characteristics of crack initiation and progress.

Fractures were observed to fall into two definite zones - 'fast' and 'slow' - referring to relative speed of crack propagation. The slow zone was the first portion or initiation of the crack at the tensile surface and was formed over almost the whole time required for fracture; the fast zone was a continuation of the slow zone and formed almost instantaneously as the final failure of the specimen. The relative lengths of these zones were found to vary between amalgams with long slow zone and short fast zone characteristic of high creep amalgams.

The slow zone was characteristically wider and more irregular than the fast zone, and was primarily associated with failure at the original alloy - matrix interface. Fracture in the slow zone occurred mainly around spherical original alloy (gamma) particles although very small particles were often fractured. Fast zone fracture was characterized by cleavage through particles of original alloy.

Fracture through Dispersalloy specimens featured a very short slow zone and long fast zone but crack propagation through these respective zones follow paths noted in other alloys. Fracture

lines passed around dispersed Ag-Cu particles and left intact the reaction halo on the surface of the particle. High Copper Ternary amalgams did not undergo discernible bend and failed to fracture at loads applied.

Rapid loading of specimens, as was completed during transverse strength testing of amalgams, produced fractures exhibiting features of fast zone failure namely, cleavage through Ag₃Sn particles (in Conventional amalgams), around dispersed Ag-Cu particles (in Dispersion Modified amalgams) and secondary cracking (in all amalgams). In rapid testing of High Copper Ternary amalgam unique features of fracture progress were discerned. Similar to all other amalgams tested, only fast zone failure was observed which usually initiated at some irregularity on the tensile side of the specimen and not directly opposite load application. Features of around-particle and through-particle failure were seen dependent on particle size, with larger particles proving more fracture resistant.

The division of fracture paths into distinct fast and slow zones by Giblin refines the observations of Asgar and Sutfin (1965) who noted a change in crack propagation from 'around gamma phase particles' to 'through gamma particles' at about half way through the width of the specimen.

The observation of predominantly fast zone failure in rapid testing of all amalgams made by Giblin confirms the conclusions of Vaidyanathan and Schulman (1979) who observed catastrophic failure in High Copper amalgams at all strain rates used. However, the absence of catastrophic failure despite continuous significant plastic deformation exhibited by Conventional amalgams tested at 0.005 in/min and 0.002 in/min as noted by Vaidyanathan and Schulman

contrasts with the findings of Giblin who noted fracture in such amalgams with a faster strain or loading rate of 0.4mm/min (0.016 in/min).

Maoleekulphiroj (1981) followed the methods of Giblin in testing beam specimens of three different thicknesses by subjecting them to static transverse sub-fracture loads equal to percentages of transverse strengths of respective amalgams. Microstructural examination of etched mounted specimens revealed additional information about crack propagation. Largely, the observations of Giblin were confirmed in this study in that characteristics of fast zone and slow zone failure were obvious in specimens tested. In addition, observations about porosity and void content in specimens showed slow zone cracking tended to pass around gamma particles and through gamma 2 and matrix phases. Thickest beam specimens displayed highest creep values, much larger void and gamma 2 content and slightly larger gamma 1 contents which, together, gave rise to stress concentration zones and favoured propagation of the crack through these areas. High creep amalgams were found to bend significantly under sub-modulus of rupture loads and fractures which resulted typically displayed slow zones related in length to creep values. Low creep amalgams exhibited short slow zones and long fast zones. Direct microstructural observations of crack progress tended to confirm findings made by previous studies; slow zone failure in Conventional amalgams featured failure at the gamma/matrix interface and the presence of voids tended to increase likelihood of fracture. Gamma 1 phase particles were most fracture resistant. Fast zone failure of Conventional amalgams tended to be through original alloy (gamma) particles although the stronger gamma/matrix interface bond in spherical alloys reduced this

feature in such alloys. Dispersalloy fractures sometimes progressed through the $\text{Cu}_6\text{Sn}_5/\gamma 1$ interface. In the slow zone, the line of fracture traced around the lathe-cut γ particles and the reacted halo (Cu-Sn) phase. In the fast zone, the fracture line was seen to pass through the original γ particles and no fracturing of dispersed particles was observed.

Mitchell, Henderson and Okabe (1980), using methods similar to previous work of Okabe et al (1978), examined the effect of various strain rates on amalgam fracture. Conventional and High Copper amalgam specimens were fractured at loading rates of 0.5, 0.05, 0.01, 0.991 and 0.0005 in/min, and stereopair micrographs of fractured surfaces were obtained with a scanning electron microscope. Observed crack sites included $\gamma 1/\gamma$ interfaces and $\gamma 2$ grains in Conventional amalgams, the interface between γ and Cu-Sn layer around Ag-Cu particles, $\gamma 1/\text{Cu-Sn}$ interfaces and clusters of Cu-Sn crystals. At high strain rates, short cracks limited the strength of amalgam. At low strain rates, the limiting stress was that needed to cause deformation of the $\gamma 1$ matrix. This stress was high in High Copper amalgams, probably due to hinderance of $\gamma 1$ grain boundary sliding by Cu-Sn crystals.

Along similar lines, Anderson, Zardiackas and Bayne (1983) examined the variation in crack propagation due to different strain rates for three representative amalgams. Specimens were loaded at 0.025, 0.25, 2.5, 25 and 250 mm/min at up to 90% of predetermined compressive strength. Earliest evidence of crack initiation occurred at 30% of compressive strength as a function of dislocation motion and grain boundary sliding.

In the third part of a three-part series on the physical metallurgy of dental amalgams, Cruickshanks-Boyd (1983b) examined fracture behaviour during setting and transverse strength of a selection of Conventional and High Copper amalgams. Transverse strength for the amalgams was determined at four setting times : 1 hour, 3 hours, 24 hours and 7 days. Microstructural examination of specimen fractures showed crack propagation was frequently inter-particular where the crack path circumvented gamma and Ag-Cu particles. Fracture within the matrix was intergranular, revealed by polyhedral facets on the fracture surface. High Copper amalgams were seen to exhibit different fracture characteristics. Fracture tended to be intraparticle, through gamma particles and matrix failure was transgranular rather than intergranular. This differing failure mode of High Copper amalgams, the author suggests, was due to the presence of small rod-shaped precipitates of Cu_6Sn_5 , only evident at very high magnification, in the matrix of such amalgams. Such an observation confirms findings of Okabe et al (1978) and Mitchell, Henderson and Okabe (1980) but conflicts with those of Vaidyanathan and Schulman (1979).

Huh and Um (1984) investigated fracture characteristics of a selection of amalgams subjected to diametral tensile stress testing at a strain rate of 0.05 mm/min. Load was stopped short of fracture and crack development was examined by SEM and analysed by electron probe microanalyser. Findings were similar to those of previous studies for the Conventional, High Copper and Dispersion Modified amalgams tested. High Copper fractures were characterized by cracking through gamma 1 matrix, between Cu-Sn crystals and through unreacted gamma particles.

The characterization of the fracture path in amalgams subjected to extremely slow strain rates was considered in continuing work of Ogura, Miyagawa and Nakamura (1987). Specimens made from Low Copper and High Copper amalgams were subjected to transverse loading at 37°C with a strain rate of $5-7 \times 10^{-7}$ mm/sec. Straining was stopped short of fracture and microstructure was examined by SEM and X-ray energy dispersive analyser. Characteristics of low strain rate fracture of dental amalgam were summarized as :

- (i) Highly localized plastic deformation of gamma 1 grains.
- (ii) Transgranular fracture of gamma 1 grains, often forming long gamma 1 needles.
- (iii) Formation of gamma 2 needles in gamma 2-containing amalgams.
- (iv) Initiation and/or propagation of cracks near the interface between gamma 1 particles and reaction layers.
- (v) Partial adherence of gamma 1 to particles and reaction layers when cracks open at these surfaces.
- (vi) Initiation of cracks at gamma 2 areas in gamma 2-containing amalgams.
- (vii) Absence of elongation of gamma 1 grains 3-5 μ m away from elongated grains or needles.

In an attempt to correlate in vitro microstructural changes to in vivo marginal fracture microstructural changes, Hanawa et al (1987) retrieved Class I amalgam restorations of Dispersalloy from partial dentures at 4 weeks and 48 weeks after placement. These restorations were sectioned normal to the occlusal surface, metallographically polished and examined by scanning electron microscope. Most cracks were found to have propagated

perpendicular to the occlusal surface, perhaps analogous to the compressive side of a beam specimen. The cracks were found to propagate through the gamma 1 matrix and through interfaces between gamma 1 and reaction layers, gamma 1 and gamma particles, and gamma 1 and Cu-Sn particles. Cracks also occasionally passed through reaction layers on Ag-Cu particles, gamma particles, Cu-Sn particles but never penetrated Ag-Cu particles. Some sections of cracks in 48-week old amalgams are relatively straight suggesting transgranular fracture and slow strain rate fracture, however, no evidence of significant plastic deformation was seen in 4-week old amalgam suggesting fast strain rate fracture.

In a subsequent study along similar lines, Hanawa et al (1988) retrieved Class I amalgams at extended periods after placement of 4, 24, 48, 72 and 96 weeks in order to clarify a mechanism of marginal fracture. The procedure used for microstructural examination of specimens was identical to that used previously (Hanawa et al 1987) with the following findings : crack propagation was identical to that noted previously (ibid) and, in addition crack initiation sites were identified at interfaces of gamma 1/reaction layer and gamma 1/Cu-Sn. In High Copper amalgams, cracks passed preferentially through the gamma 1 matrix, alloy particles and occasionally through the gamma 1/Cu₆Sn₅ interface. An interesting finding was that of crack initiation in Tytin at the occlusal surface, whereas no cracks initiated at the occlusal surface in Dispersalloy.

Hanaoka et al (1988) investigated fatigue cracking of notched beam specimens of amalgams subjected to cyclic transverse loading

(3.2 Hz, 0.86 Kg maximum). In all specimens of Tytin¹, Hi-Atomic² and Dispersalloy³ multiple cracks formed in the base of the notch, and needle-like crystals were seen to span the cracks. Gamma 1 and gamma 2 needles were found in Hi-Atomic, but only gamma 1 was found in Tytin and Dispersalloy as a result of stress-assisted crystal growth. Gamma 1 areas exhibited rounded craters suggesting some degree of plastic deformation had occurred prior to failure. For sufficiently small strain rates, it was concluded that plastic deformation could play a role in fatigue crack opening.

Williams and Cahoon (1988) examined marginal breakdown in specimens of Dispersalloy and New True Dentalloy⁴ caused by low intensity cyclic stress. Restorations were placed in cavities prepared in aluminium beams and subjected to three-point cyclical loading (1.7 Hz at 37°C for 21,000 cycles). SEM examinations were conducted at baseline and after each 4,200 cycles and showed that such low intensity cyclic compressive loading resulted in a zone of plastic deformation in the amalgam restoration containing lines of severe plastic deformation, voids and fractures along the margin. Dispersalloy specimens were less significantly deformed than the specimens of New True Dentalloy.

1 S.S. White Manufacturing Co., Philadelphia U.S.A.

2 G.C. Chemical Co., Japan

3 Johnson and Johnson Dental Products Co., New Jersey U.S.A.

4 S.S. White Manufacturing Co., Philadelphia U.S.A.

ORIGINAL
INVESTIGATION

CHAPTER I

SCOPE OF THE INVESTIGATION

In attempts to establish relevant laboratory tests which could serve as guides to clinical performance of dental amalgams, investigators have focused on creep testing as an acceptable alternative to the traditional mechanical tests of compressive strength, tensile strength, dimensional change and flow. Creep testing offers several advantages in terms of ease of specimen production and testing format, use of clinically relevant temperature of testing and results which discriminate between amalgam formulations.

Knowledge about the specific mechanism of amalgam failure may shed light on possible testing formats which may approximate this mechanism better in a controlled and monitored way. To this end, the concept of sub-fracture loading of amalgam specimens may provide useful information about amalgam deformation.

Giblin (1979) considered the effects of static fatigue on amalgam specimens which had been subjected to 'low' transverse loads at 22°C and, in observing microstructural characteristics of fractures which occurred, was able to directly relate amalgam failure to mechanical properties. In that study, amalgam beam specimens were subjected to a range of indirect three-point transverse loads equal to a percentage of the modulus of rupture determined for that amalgam. Many significant observations were made in relation to the factors contributing to amalgam failure, to the correlation of mechanical properties to amalgam failure, and about the macroscopic and microscopic characteristics of fractured amalgam.

A possible mechanism of marginal and bulk fracture of amalgam restorations was proposed.

The Giblin study did not consider sub-(modulus of rupture) loading of specimens at 37°C which may allow observations of temperature related factors to be made. Giblin carried out his studies at 22°C (ambient temperature) because he was studying the effects of sub-fracture loads related to fracture loads observed in normal laboratory testing at ambient temperatures. It must be borne in mind that Giblin did not have the benefit of previous studies of this type.

Giblin postulated that at 37°C bend may be greater and fracture time shorter when amalgam is subjected to sub-(modulus of rupture) loading. He made recommendations for future investigators which may provide worthwhile information. Among the eight recommendations of Giblin were :

- (i) Use of very low transverse loads (10% of Modulus of Rupture).
- (ii) Dynamic versus static loading.
- (iii) Thinner beam specimens for the test.

Giblin did not make a recommendation for testing at 37°C.

The contribution of cyclically applied loading on amalgam to fracture and bending, together with a smaller, more clinically relevant specimen size may yield more information about the mechanism of amalgam failure which may consolidate trends and conclusions made by Giblin.

The aim of this investigation was to examine the behaviour of dental amalgam when subjected to static and cyclic sub-fracture transverse loading at 37°C with the objective of relating this

behaviour to mechanical properties, microstructural characteristics, and possible clinical behaviour.

In attempting to more closely parallel clinical conditions under which amalgam restorations are likely to be subjected, several aspects are worth consideration. A transverse mode of load application was used as it best reflects the clinical situation of amalgam restoration stressing. However, several refinements of this test will be included based on the observations of Giblin.

Transverse, three-point loading was carried out on beam specimens smaller than those of Giblin, approaching the approximate bulk of a typical Class II restoration. Loading was applied directly to the specimen via a testing jig and loading apparatus not used by Giblin. In this way, the entire testing mechanism could be housed in an incubator for testing at 37°C with only the timing mechanism being excluded from the incubator.

Rectangular prism specimens of dimensions 12mm x 4mm x 2mm, were made using three methods of condensation to examine the effect of these methods on fracture characteristics. The mould used by Giblin was modified to allow the production of the smaller specimen and the removal of excess mercury from each specimen. This mould allows condensation of amalgam to proceed in a way similar to the packing of a clinical Class II restoration. Five amalgam alloys representing three basic formulations were used in the study and an additional selection of ten alloys was applied to the model subsequently.

Giblin (1979) and Maoleekulphiroj (1981) noted no direct relationship between conventional mechanical properties of compressive strength and tensile strength and the behaviour of amalgam subjected to sub-fracture transverse loads. However, the

rheologic property of creep was found to be related to fracture and bend behaviour. In view of these findings, testing of compressive strength and tensile strength was not carried out since it is likely no significant relationships would be drawn. However, the modulus of rupture (transverse strength) of each amalgam was determined to allow calculation of an appropriate transverse load to which all specimens will be subjected. Creep values at 3-hours and the extended 72-hours were determined to allow possible correlations to fracture behaviour to be made.

In contrast to Giblin (1979) and Maoleekulphiroj (1981), a constant sub-fracture load was applied to all specimens irrespective of strengths and creep values of the amalgams themselves. This parallels the clinical situation where an individual exerts a specific occlusal load determined by physiological limits of masticatory muscle capability and strength of tooth structure; the individual does not possess the advantage of exerting a large range of loads on his restorations secure in the knowledge that the strength of those amalgams are well in excess of that occlusal load range. The load used was determined as equal to 50% of the average modulus of rupture (transverse strength). The choice of single load also parallels creep testing where a stress of 36 MPa is applied to all amalgams tested irrespective of compressive strength. ✓

Testing was carried out at 37°C which, apart from being clinically relevant, parallels the method of creep testing which is also conducted at this temperature. The new format of testing apparatus allowed testing to be carried out in an incubator set at 37°C. In addition, it was anticipated that higher temperature testing would serve to reduce testing times. 22°C testing was also

carried out to enable comparisons with previous studies as well as comparisons between results at 22°C and 37°C in this study.

The behaviour of specimens tested was monitored by determining bending characteristics and times to failure of specimens. Degree of bend were determined by comparing specimens to a bend scale where representative bends are illustrated and assigned ranks in the range of '1' to '10'. Such a numerical system allowed principles of statistical analysis to be applied to results obtained for relatively subjective data.

Conclusions may be made about relative behaviour of amalgams. Mean times to failure were recorded to the nearest hour and standard deviations calculated. Microstructural analyses were presented in micrographs.

Data and results have been presented in both tabulated and graphic form where possible to clearly illustrate the evidence for trends and conclusions drawn; although this potentially represents duplication of data, where specific tables are compared in the discussion, graphical presentation allows instantaneous visualization and easier interpretation by some readers.

CHAPTER II

EXPERIMENTAL PROCEDURES

Selection of Alloys

Five alloys were selected from three main groups of amalgam alloy formulations. The alloys tested are shown in Table II-1. These groups varied in type of alloy and in particle characteristics. Ag₃Sn-type alloys are represented by Amalcap F and Hi-Atomic. Amalcap Non-Gamma 2 and Dispersalloy represent Dispersion Modified amalgams and Tytin is an example of a Single Melt High Copper Alloy. Also shown in Table II-1 are the codes designating each alloy as used in Chapter III 'Results and Discussion'. The five alloys in Table II-1 represent the alloys studied in detail as the major study. In addition, a further ten alloys as shown in Table II-3 were used in a subsidiary investigation to test the "model". Details of these alloys are presented later in this chapter.

Preparation of Specimens

Two specimen shapes were used for testing mechanical properties. A cylindrical specimen 8mm high and 4mm diameter was used for 3-hour and 72-hour creep determination and a rectangular prism (beam) specimen 12mm x 4mm x 2mm was used for modulus of rupture (transverse strength) determination and static and cyclic sub-modulus of rupture testing.

Cylindrical Specimens

The Mould

This consisted of a cylindrical unit of machined steel containing a central cylindrical space, of dimensions 9mm length

Table II-1 : Amalgam alloys used in the investigation.

Alloy	Manufacturer	Type of Alloy	Particle Characteristics	Code
Amalcap F	Vivadent Liechtenstein	Ag ₃ Sn	Lathe cut Fine	AF
Hi-Atomic	G.C.Chemical Co. Japan	Ag ₃ Sn	Spherical	HA
Amalcap Non-Gamma 2	Vivadent Liechtenstein	Dispersion Modified	Lathe + Spherical	AN
Dispersalloy	Johnson & Johnson USA	Dispersion Modified	Lathe + Spherical	D
Tytin	S.S. White USA	Single Melt High Copper	Spherical	T

and 4mm diameter into which the amalgam mass was packed. The floor of the specimen space was provided by a base and post which closely fit the space and enabled ejection of the specimen after preparation. Two spacer discs which enclosed the post (1mm thick and 8mm thick respectively) together established the dimensions of the specimen. Following condensation, a final layer of one millimetre thickness representing the mercury-rich layer was removed from each specimen by the removal of the 1mm thick spacer and the extrusion of the specimen one millimetre from the mould thereby exposing it to easy removal with a razor blade. The 8mm spacer could then be removed allowing free movement of the steel post and ejection of the specimen (Figure II-1).

Rectangular Prism Specimens

The Mould

Rectangular specimens of final dimensions 12mm x 4mm x 2mm were prepared using a steel mould similar to that used by Peet (1971), ^{Wong} Adair (1971), Giblin (1979) and Maoleekulphiroj (1981). Modifications to the basic mould, to reduce internal dimensions and therefore specimen dimensions, was accomplished with the use of two spacer units : one rectangular prism spacer 12mm x 3mm x 3mm and an L-shaped spacer with horizontal leg 4mm wide and 1 mm high and vertical leg 3mm square. In addition to the spacer units, an enclosing steel jacket unit which fit snugly around the mould and featured a rectangular 'window' 12mm x 4mm on its most superior aspect was incorporated into the mould. The 'jacket' effectively added an additional one millimetre of height to each specimen by virtue of the 'window' being exactly aligned to the opening of the specimen cavity and the thickness of the uppermost 'window' surface

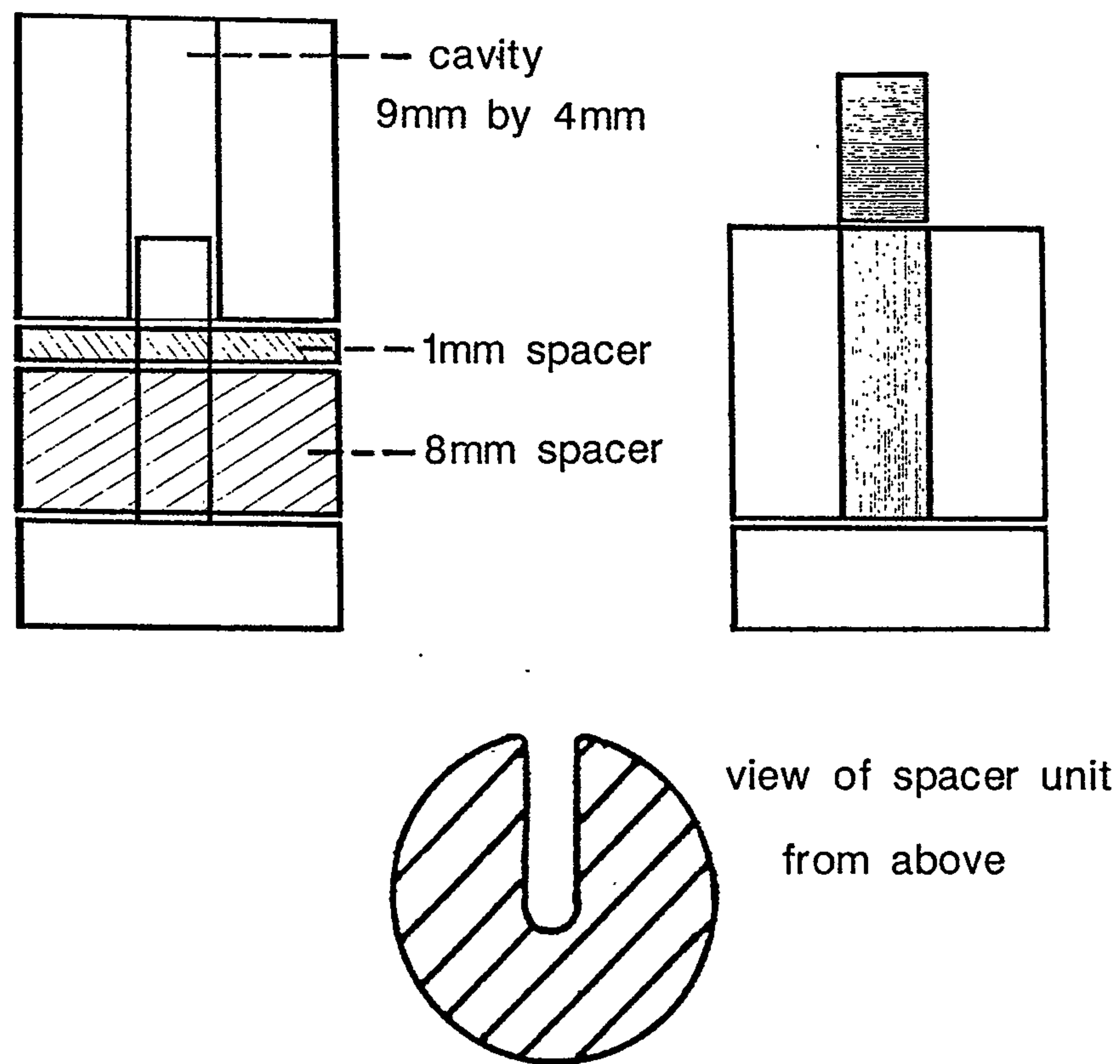


Figure II-1: Diagrammatic representation of mould used for preparation of cylindrical amalgam specimens for 3-hour and 72-hour creep.

being one millimetre thick. A diagrammatic representation of the entire mould, spacer units and enclosing jacket is shown in Figure II-2. A photograph illustrating the specimen used compared with that of Giblin is shown in Figure II-3.

Manipulation of Amalgam

Proportion of Alloy to Mercury

The ratios of alloy to mercury as recommended by manufacturers and used in the study are shown in Table II-2. This ratio, as can be noted from the table, varied from 1:1 (Amalcap F and Amalcap Non-Gamma 2) to 10:7 (Hi-Atomic).

Trituration

Trituration for all amalgams was accomplished using a Silamat¹ ultra-speed amalgamator. The machine has a rate of oscillation of approximately 4,500 ~~r~~^c.p.m. and when loaded has a throw of 60mm. This amalgamator allowed multiple mixes to be achieved in a short time thereby minimizing the potential for layering of the amalgam to occur. Methods of trituration and trituration time for alloys and applied alloys used in the study are shown in Tables II-2.

Condensation and Mercury Removal

Cylindrical Specimens

Preparation of cylindrical specimens for static creep testing was accomplished using hand condensation and either 1mm, 1.5mm or 2mm diameter pluggers. For lathe-cut and Dispersion Modified amalgams, 1mm and 1.5mm diameter condensers were used. For

1 Vivadent, Schaan-Liechtenstein.

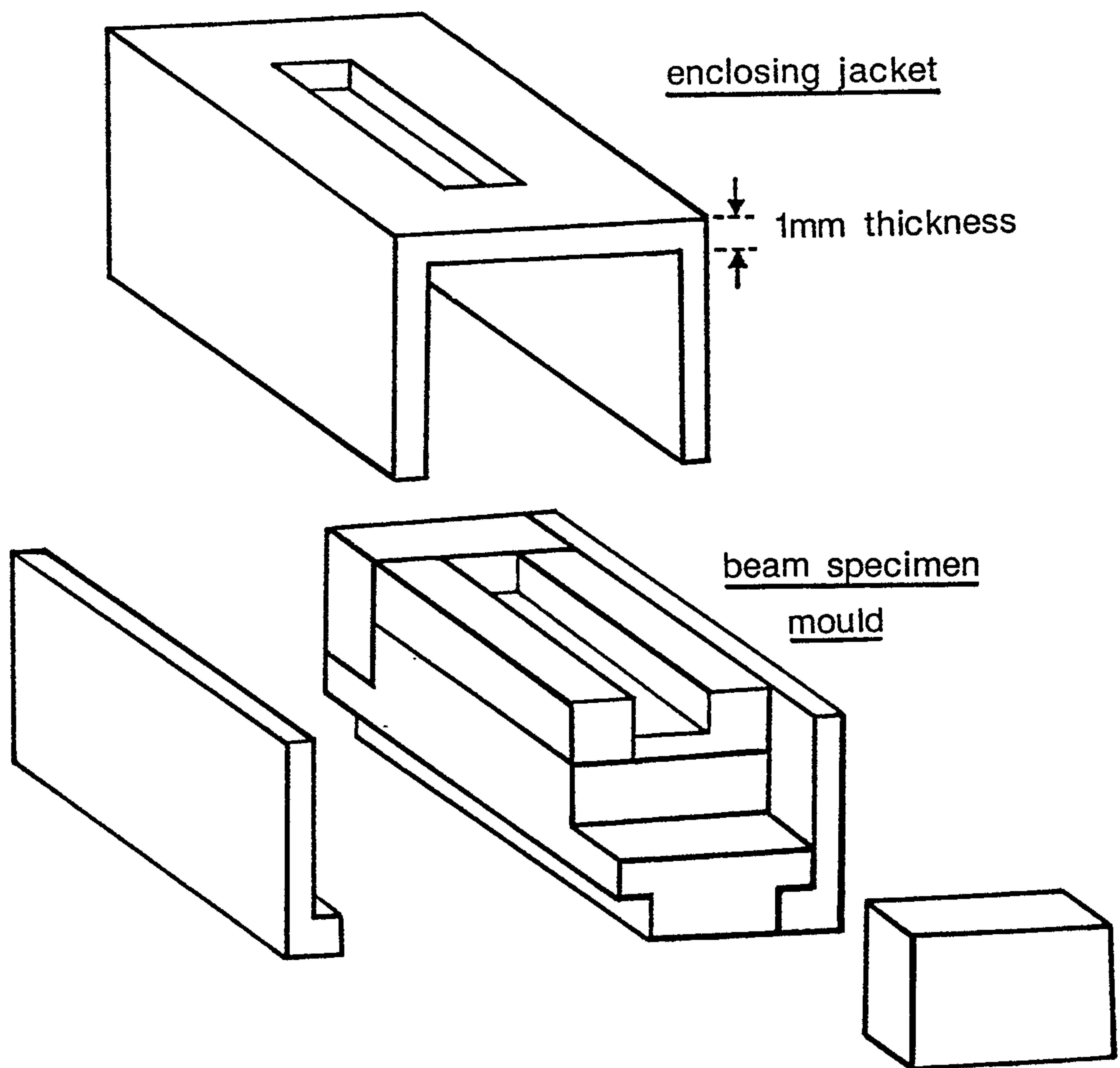


Figure II-2: Diagram depicting mould used to prepare beam specimens.

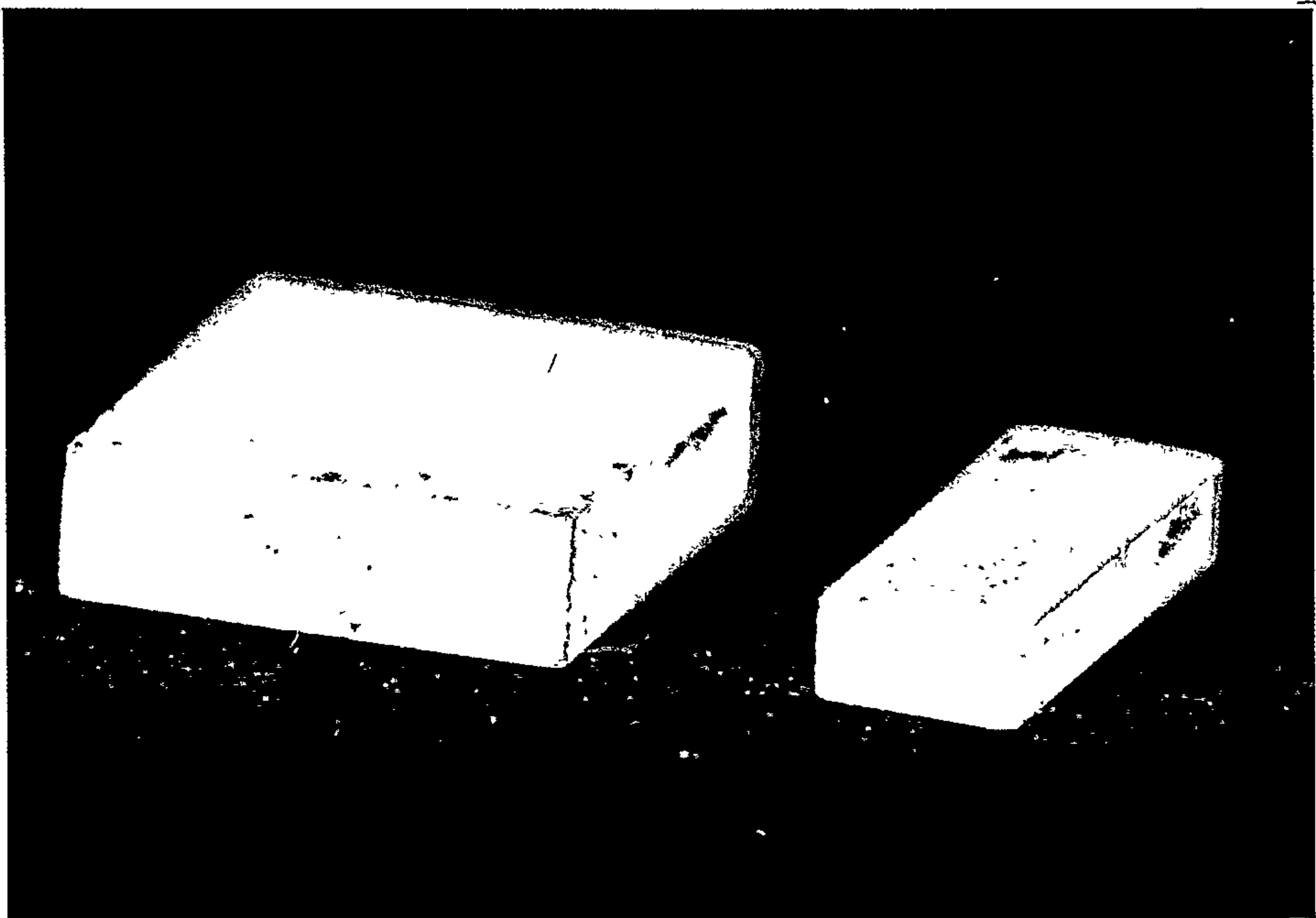


Figure II-3: Actual specimen used (on right)
compared to Giblin specimen.

Table II-2 : Characteristics for manipulation of amalgam alloys.

Alloy	Presentation Form	Alloy : Mercury Ratio	Method of Trituration	Trituration Time (secs)
Amalcap F	Encapsulated Powder	10:10	Plastic capsule - no pestle	7
Hi-Atomic	Bulk Powder	10:7.0	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
Amalcap Non-Gamma 2	Encapsulated Powder	10:10	Plastic capsule - no pestle	7
Dispersalloy	Self-activated encapsulated powder	10:9.8	Plastic capsule - plastic pestle	6
Tytin	Encapsulated powder 600gm	10:7.7	Plastic capsule - no pestle	7

spherical particle alloys, 1.5mm and 2mm condensers were used. Following trituration the amalgam mass was divided into six portions for spherical or eight portions for lathe cut containing alloys and condensed into the mould one portion at a time using 25 thrusts on each increment for a total condensation time of approximately two or three minutes. No mercury was removed prior to condensation. Excess mercury was removed during condensation and the 1mm mercury-rich surface layer was finally removed.

Rectangular Prism (Beam) Specimens

To examine the effect of differing final condensation loads on transverse strength and sub-modulus of rupture loading behaviour, the condensation of amalgam for the beam specimens was accomplished in one of three ways. Following trituration, the mass of amalgam was divided into four portions and condensed one portion at a time with excess mercury removal following the condensation of each portion using a 3.5mm diameter plugger (Ash 3). After satisfactory initial condensation was achieved and the mould assembly was packed with amalgam level to the top of the enclosing jacket, the specimen was further condensed without delay in one of three ways :

- (1) by hand condensation alone; again using a 3.5mm diameter amalgam plugger.
- (2) machine condensed using the Hounsfield Tensometer¹, following hand placement. This method involved the use of a steel beam 3.5mm x 3mm x 11.5mm as a plugger which was positioned directly on top of the newly placed amalgam specimen. The entire assembly was then placed between the

1 Tensometer Limited. Croydon, England.

platens of the tensometer and a positive load of 450N was applied for one minute.

- (3) machine condensation using the Hounsfield Tensometer as for (2) but applying 2250N for one minute.

Following condensation, the enclosing jacket of the mould was removed thus exposing the outer-most layer of each specimen, approximately 1mm thick, representing the mercury rich layer. This layer was then easily shaved away using a razor blade, leaving a specimen 12mm x 4mm x 2mm.

Because of the size of the condensing beam in relation to the overall mould size, excess mercury was removed during load application in the Hounsfield Tensometer.

Mechanical Testing

Creep

Following ageing of the specimens for seven days at 37°C, the cylindrical specimens were prepared for static creep testing at 37°C. To enable positive contact between the specimen and the loader, the ends of each specimen were ground flat with fine abrasive silicon carbide paper. The specimen was placed lengthwise, for testing, under a static load of 36 MPa for four hours as set out in AS 2110-1983. Measurements were taken at one and four hours after the start of the test. Specimens were then immediately reloaded and measurements were again taken at seventy-three hours. The 3-hour creep value was defined as the percentage change in length occurring between one and four hours, the 72-hour creep was the corresponding value difference between one and seventy-three hours. Extended creep testing is an

extension of the usual specification three hour static creep test (Wing 1985, 1986).

Modulus of Rupture (Transverse Strength)

The test was performed by applying a three-point load to a seven-day old beam specimen 12mm x 4mm x 2mm. The testing jig used was that used by Giblin (1979), shown in Figure II-4. The load bearing support block consisted of two hardened steel bars of 2.3mm diameter set, with their centres 6.35mm apart, in a brass block, of 12.1mm x 7.5mm x 25.4mm which in turn was set in another brass block 25.4mm x 50.8mm x 6.3mm. The load was applied through a cylindrical steel bar 2.3mm diameter which was set parallel and equidistant to the supporting bars. This loader was set in a brass plate similar to the base of the supporting section, 25.4mm x 50.8mm x 6.3mm.

Set in the loader plate section were two brass cylinders milled precisely to receive capstan pillars (6.3mm diameter) set into the supporting plate. These cylinders allowed precise, reproducible alignment between loader and load bearers.

The testing jig was used for modulus of rupture testing, static sub-modulus of rupture testing and cyclic sub-modulus of rupture testing. Specimens were positioned in the testing jig so that the first-condensed layer was lowermost.

Modulus of rupture testing took place by positioning the assembled testing jig complete with a specimen between the platens of the Hounsfield Tensometer. The machine was activated at a crosshead speed of 0.4mm/min. The load was increased until fracture of the specimen occurred. The fracture load achieved was applied to a formula to derive the modulus of rupture:

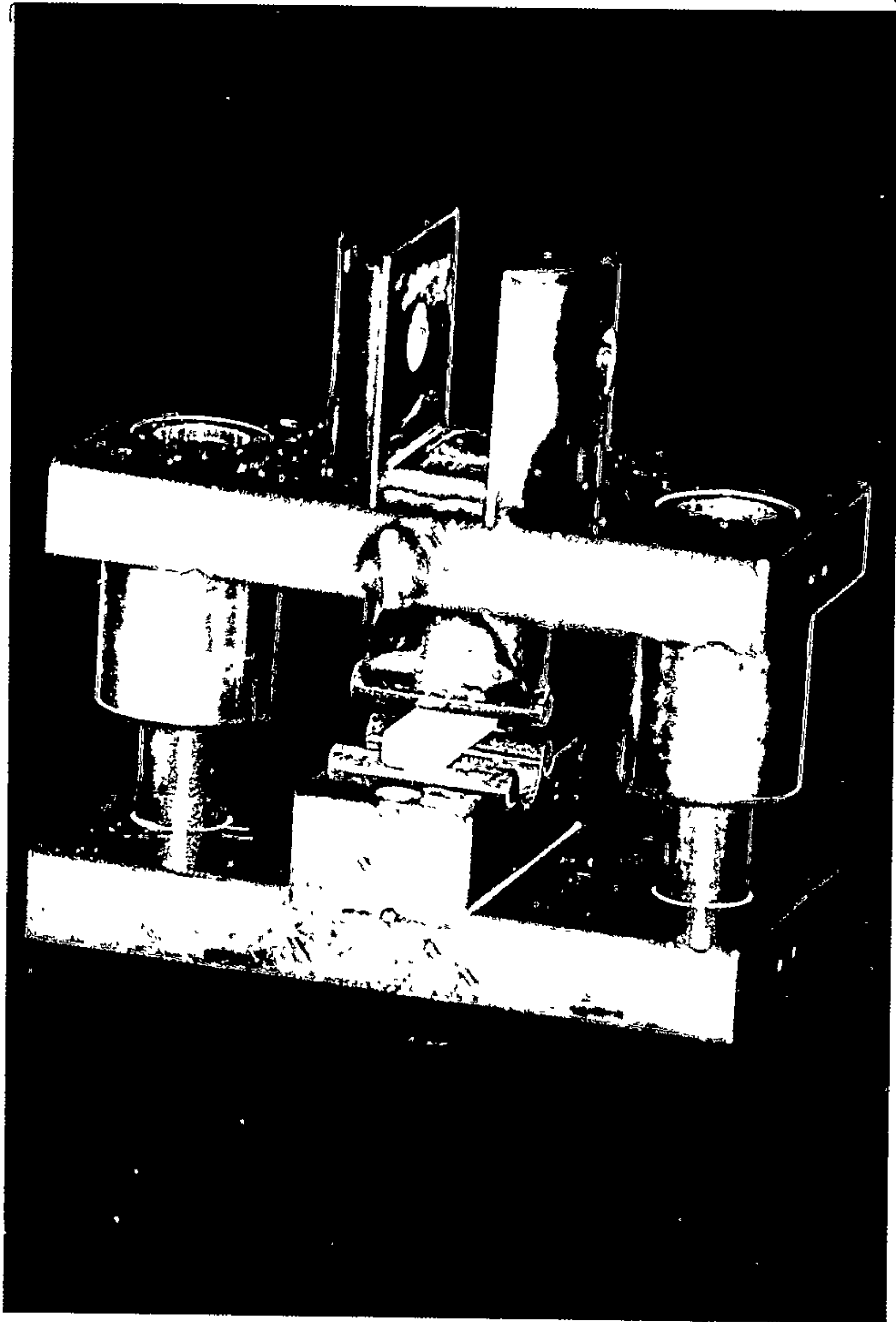


Figure II-4: Modulus of rupture testing jig.

$$M.R. = \frac{3 WL}{2 BD^2}$$

where M.R. = modulus of rupture

W = fracture load.

L = span of supports.

B = width of test piece.

D = thickness of test piece.

A mean and standard deviation of three values for each amalgam produced by the same condensation load was determined.

Sub-Modulus of Rupture)

Static Loading

This test used the same types of beam specimen and testing jig as used for modulus of rupture determination.

Giblin (1981) and Maoleekulphiroj (1981) used the machine designed by Peet (1971) for sub-modulus of rupture loading. This machine accommodated ten support blocks, loaders & specimens simultaneously. Load was applied to the testing jig indirectly via a loading arm 91.44cm long. The framework of the machine was fabricated from 25.4mm x 3.2mm angle steel and had a base area of 0.5m². The framework allowed accurate variable load application which was accomplished using lead shot in bottles attached to a weight carrier. Under each arm was positioned a microswitch to which a digital electric clock was connected. On specimen failure, the arm would fall only a few millimetres before activating the microswitch thereby stopping the clock, recording day and time of fracture. The load necessary to be added to the arm of the static fatigue machine to correspond to that necessary to produce fracture in the Hounsfield Tensometer was calculated taking into account

force exerted by the loading arm at point of load application and mechanical advantage of the added load. Initially, the machine was loaded with ten specimens at one time but the vibration in the machine illicited by the first specimen fracture artificially initiated fracture of other specimens, thus testing was restricted to one specimen at a time. Testing took place at 22°C on bench top.

In order to carry out testing at 37°C a new format for sub-modulus of rupture testing was designed (Figure II-5) which enabled direct loading of specimens within the constricted confines of a 37°C incubator.

The machine¹ consisted of two steel units : a vertically disposed loader platform mounted on a steel column which inserted into a closely fitting channel in the other larger housing unit. The platform was able to move up and down easily within the channel which was lined with ball-bearings. At the base of the column was attached another smaller platform which contacted the superior aspect of the testing jig very positively. Direct loading of the testing jig was achieved by placing loads directly onto the upper platform. Attached to the column was a horizontal arm which protruded approximately 2cm beyond the edge of the platform and 2cm below it. A microswitch mounted onto the main housing unit by a brass bracket was of a sufficiently small distance from the horizontal arm to cause stopping of the clock when the specimen fractured and the loading platform fell. This allowed determination of the time to fracture of the specimen to the

1 A.J. Wilcock; Scientific & Engineering Equipment, Victoria. Australia.

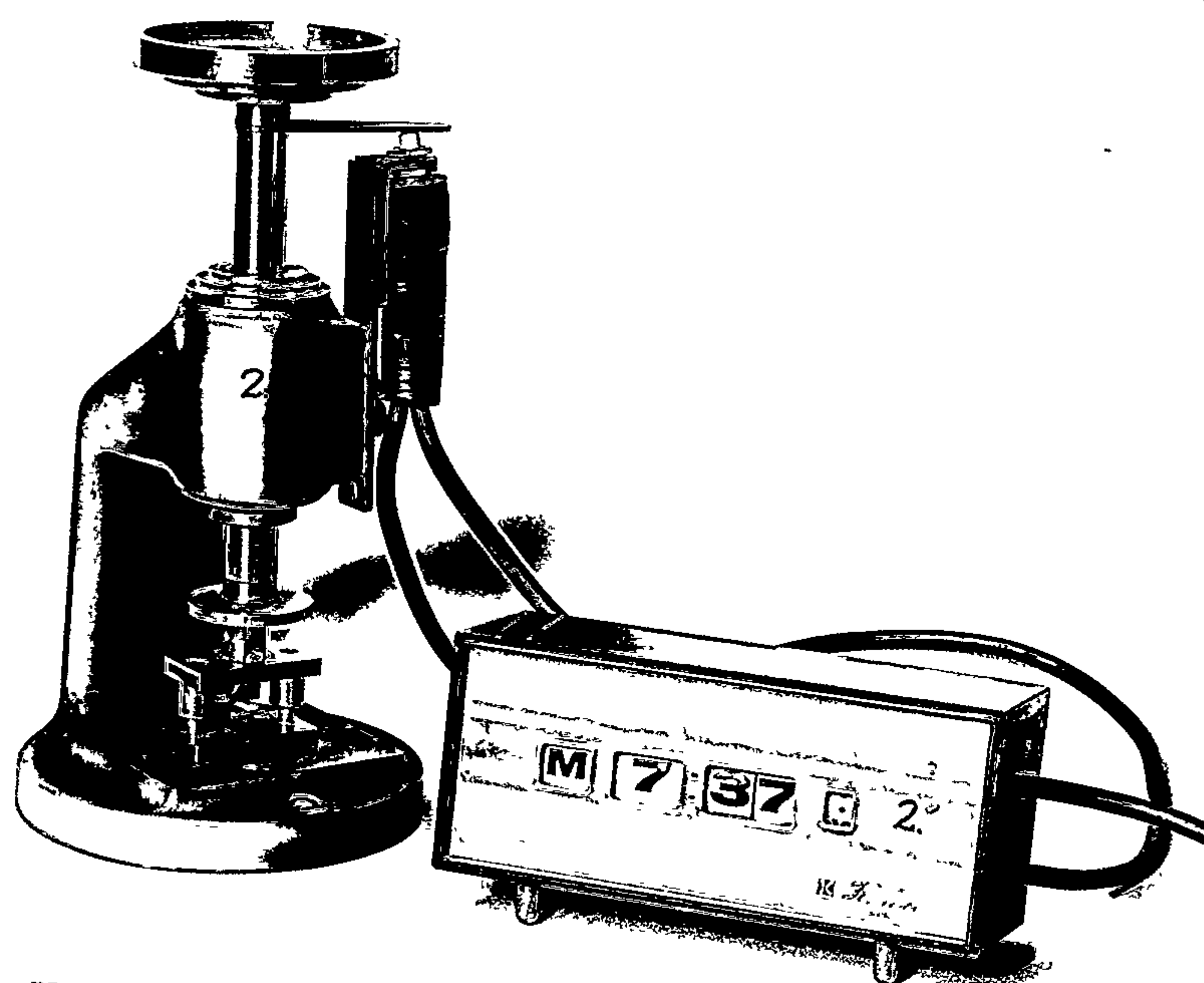


Figure II-5: Loading apparatus used in sub-modulus of rupture testing.

nearest minute without supervision. Standing 28cm from base to load platform and having a base area of approximately 0.02m^2 , the new format transverse loading machine could be easily placed in an incubator¹ allowing testing at 37°C . The clock to which the machine was connected was placed outside the incubator. This enabled observation of fracture time without disturbing the incubator thereby maintaining a constant temperature of testing. Parallel testing of specimens, from each group of alloys was conducted on bench top at 22°C to allow a comparison of results obtained in this investigation at 37°C with a new format beam specimen, with previous investigations at 22°C and a larger format specimen.

Cyclic Loading

To examine the effect of intermittent sub-modulus of rupture loading at 37°C on time to fracture of specimens, an additional test was devised. Using the same equipment as that used for static sub-modulus of rupture loading at 37°C , a time sequence of cyclic loaded and unloaded testing was conducted. The testing jig itself was not removed from the transverse loading apparatus at any time during the cyclic loading test, and specimens were left unloaded over night. To evaluate the effect of the unloaded platform on specimen bending, a specimen of highest creep was subjected to static loading of the unloaded platform for five weeks.

1 Labec Laboratory Equipment Pty. Ltd. Sydney, Australia.

Test Procedure

To obtain a mean modulus of rupture (transverse strength) for each amalgam tested, three specimens were prepared as described earlier and subjected to increasing load in a transverse mode until failure in the Hounsfield Tensometer. Mean modulus of rupture values were then obtained for specimens condensed in each of the three methods of condensation described earlier.

In order to allow direct comparison of the new format specimen to that of Giblin (1979) and Maoleekulphiroj (1981), regarding time to fracture under sub-modulus of rupture loads, specimens were initially tested at 22°C using the new format transverse loading machine and under a load of 90N which represented approximately 50% of the average fracture load of all alloys tested. For the specimen size tested, a load of 90N represents a transverse stress of approximately 67 MPa. Specimens were assembled in the brass testing jig and the timing clock was then set to the time of day. The testing jig was then carefully placed beneath the loading column of the loading machine previously described. Loads were then carefully applied to the testing jig and the microswitch was aligned to the loading machine without further delay. The entire apparatus was then left undisturbed until fracture had occurred or a period of five weeks had passed. No specimens fractured instantly and many, particularly specimens made from Single Melt High Copper amalgams, did not fracture within the period limit of testing.

Following fracture, specimens were re-located and cemented with cyano-acrylate cement¹ to enable evaluation of degree of

1 Selleys Supa Glue Gel - 85% cyanocrylic esters. Japan.

pre-fracture bending. Using the system of ranking devised by Giblin (1979) whereby a numerical value was assigned to the degree of pre-fracture bend achieved by each specimen, a scale was drawn up to which specimens could be matched. A value of '1' represented an untested specimen and '10', a specimen exhibiting greatest bend. Specimens which did not, in fact, fracture but did bend were also ranked. Each specimen was evaluated on three different occasions by the single evaluator. This scale is shown in Figure II-6.

It was then decided to examine the effect of cyclic loading of specimens in contrast to the static loading of specimens conducted in the study thus far. Using the same basic apparatus used in the study ie. three-point loading jig and direct loading apparatus, testing was conducted at 37°C.. Two amalgam alloys from the study were selected for cyclic testing. These were the alloys of highest creep values : Hi-Atomic and Amalcap F.

A testing regime was designed whereby specimens were subjected to the full load (representing 50% of the average modulus of rupture) for a period of one hour at a time after which the load weights were removed from the loading platform. Ideally, to completely unload each specimen each hour, the loading platform would need to have been raised from the transverse testing jig and the upper member of the transverse testing jig would likewise need to be removed from contact with the specimen. Re-centring the loading bar of the transverse testing jig on the specimen each hour, and re-locating the testing jig in the loader proved to be very difficult to achieve reproducibly without premature specimen fracture, and so it was decided that only the applied weights (equal to 80 N) would be removed each hour. A minimum period of

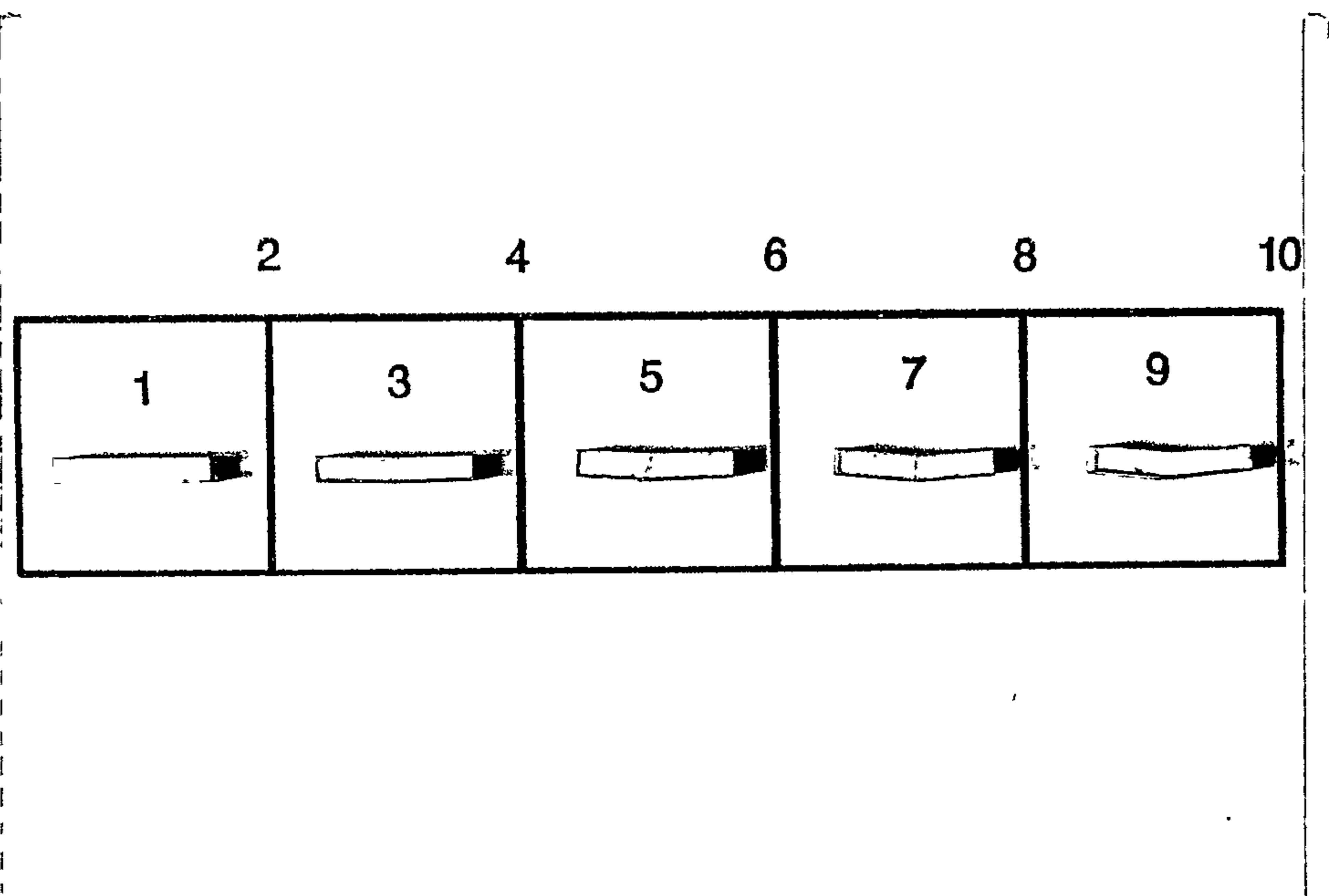


Figure II-6: Scale of bend characteristics.

five minutes was set as the period for which specimens were left 'unloaded'. Thus, on average, each specimen was loaded for one hour, 'unloaded' for five minutes, loaded for another hour, 'unloaded' for five minutes and so on. During the 'unloaded' periods, the loading platform (10 N) was left in contact with the transverse testing jig. To examine the effect, if any, on bending of specimens under the load of the loading platform alone, two specimens made from the amalgam alloy exhibiting highest creep values were set up and left under the 10 N load at 37°C for a period of five weeks.

Selected, re-assembled specimens were mounted in cavities prepared in a lucite rod (25.4mm diameter x 10mm) using cold-curing clear acrylic resin. These specimens were then polished and etched, using the etching technique described by Wing (1961), to allow investigation of the microstructure and method of fracture, as described by Giblin (1979) and Maoleekulphiroj (1981). This was carried out using optical microscopy at magnification of 10x, 50x and 500x.

Data Analysis

In determining static creep, a mean value of four determinations was used. For modulus of rupture (transverse strength) a mean of three determinations was used, for time to fracture at 37°C and 22°C a mean of four and three determinations respectively were used. A mean of three determinations was also used for cyclic loading.

Standard deviations were calculated using the formula :

$$SD = \sqrt{\frac{\sum (x - \bar{x})^2}{(n - 1)}}$$

where x = value for single specimens

n = number of specimens tested.

$\bar{x}$ = mean value

The statistical significance of the difference between two means was determined using the "Student 't' test" (Spiegel, 1961). Differences between means were considered statistically significant when the 't' test indicated that $P \leq 0.02$, i.e. the 98% confidence level.

Microstructural Preparation

The method described by Wing (1961) was chosen for the preparation of specimens for microstructural analysis. Fractured, re-assembled specimens mounted in lucite rods were polished to provide a flat surface with the use of successive grades of silicone carbide paper. The surface of the lucite rod was then polished on a rotating, motor driven pad¹ using a selection of diamond pastes with a mixture of propylene glycol and water as a lubricant.

Following polishing, specimens were etched using a cyanide etch developed by Wing (1961). This etching procedure consisted of the application of two solutions to the polished surface of the

1 Beuhler Ltd., Metallurgical polishing wheel. Evanston, Illinois U.S.A.

specimen with the aim of defining grain boundaries and particle detail. The first solution consisted of 2 grams KCN and 1 gram iodine crystals dissolved in 25 c.c. of water. Specimens were briefly wiped with this solution and rinsed in water. Specimens were then submerged for 4 seconds in a second solution consisting of 2 grams of KFeCN dissolved in 20 c.c. water mixed with a solution of 2 c.c. of ammonia and 20 c.c. of water. Specimens were finally washed in water and dried quickly with alcohol and compressed air.

Prepared specimens were viewed on a Neophot II optical light microscope¹ at magnifications of 10x, 50x and 500x to examine crack morphology.

Additional Alloys

Following completion of the study using the five representative amalgam alloys : Amalcap F, Hi-Atomic, Amalcap Non-Gamma 2, Dispersalloy and Tytin; a group of ten additional amalgam alloys were tested to determine validity of the test method. Table II-3 presents the additional alloys showing type of alloy represented, particle characteristics and code designating the alloy as cited in Chapter III 'Results and Discussion'. Conventional Ag₃Sn-type amalgam is represented by seven alloys varying in particle characteristics; Luxalloy Non-Gamma 2 represents the Dispersion Modified formulation. Single Melt High Copper amalgams are represented by Permite C and ANA 2000. It can be noted from Table II-3 that the alloy Permite C represents a

1 Carl Zeiss, Jena. East Germany.

Table II-3 : Additional alloys used to test the model.

Alloy	Manufacturer	Type of Alloy	Particle Characteristics	Code
F400	S.D. Industries Australia	Ag ₃ Sn	Lathe cut - Superfine	F400
Ultrafine	S.D. Industries Australia	Ag ₃ Sn	Lathe cut - Fine	UF
Caulk Spherical	L.D. Caulk Co. USA	Ag ₃ Sn	Spherical (50 μ m)	CS
Spheraloy	Kerr Mfg. Co. USA	Ag ₃ Sn	Spherical (50 μ m)	S
New True Dentalloy	S.S. White England	Ag ₃ Sn	Lathe Cut - Fine	NTD
Luna Atomic	G.C. Chemical Co. Japan	Ag ₃ Sn	Spherical (100 μ m)	LA
G. & G. Superfine	Englehard Industries Australia	Ag ₃ Sn	Lathe Cut - Fine	GS
Luxalloy Non-Gamma 2	Degussa Dental Germany	Dispersion Modified	Lathe + Spherical	LX
Permite C	S.D. Industries Australia	Single Melt High Copper + Ag ₃ Sn	Spherical (S.M.H.C.) + Lathe-Superfine (Ag ₃ Sn)	P
ANA 2000	AB Nordiska Affineriet Sweden	Single Melt High Copper	Lathe Cut Fine	ANA

blended amalgam featuring lathe-cut particles of Conventional Ag₃Sn formulation and spherical Single Melt High Copper particles.

Table II-4 presents specifications of alloys including presentation form, alloy/mercury ratio, method of trituration and trituration time. Three basic presentation forms characterized these alloys : encapsulated powder, bulk powder and tablet. Proportioning of bulk powder was accomplished using an 'Ash' Alloy Balance (England) and using recommended proportions. Specimens were prepared using only hand condensation for determination of mechanical properties and the three condensation methods for testing.

Specimens made from each of the applied alloys were subjected to three tests :

- (i) Static creep (3-hour and 72-hour).
- (ii) Modulus of rupture testing.
- (iii) Sub-modulus of rupture testing at 37°C with a static load (67MPa).

In addition, bending and fracture characteristics were also ascertained. Results obtained were also examined as were specimens in the main study and subjected to statistical analysis.

Table II-4 : Characteristics for manipulation of additional amalgam alloys.

Alloy	Presentation Form	Alloy : Mercury Ratio	Method of Trituration	Trituration Time (secs)
F400	Encapsulated powder	10:10.5	Plastic capsule with plastic disc pestle	7
Ultrafine	Bulk powder	10:10.5	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
Caulk Spherical	Tablet 300gm	10: 7.5	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
Spheralloy	Tablet 300gm	10: 7.5	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
New True Dentalloy	Tablet 300gm	10:10	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
Luna Atomic	Bulk powder	10:10	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
G. & G. Superfine	Bulk powder	10:10	Re-usable plastic capsule with 1gm cylindrical metal pestle	7
Luxalloy Non-Gamma 2	Encapsulated powder	9:10	Plastic capsule with plastic pestle	8
Permite C	Encapsulated powder(800mg)	10:10	Plastic capsule - no pestle	8
ANA 2000	Bulk powder	10:10	Re-usable plastic capsule with 1gm cylindrical metal pestle	7

CHAPTER III

RESULTS AND DISCUSSION

Mechanical Properties

Table III-1 presents the values for modulus of rupture (transverse strength) determined for alloys tested, related to method of specimen condensation. These data are represented graphically in Figure III-1. Values for 3-hour and 72-hour creep are presented in Table III-2 and graphically in Figure III-2.

From data for modulus of rupture (transverse strength), it may be seen that amalgams prepared from all alloys, with the exception of Tytin, have transverse strengths which fall into the range of 110 MPa to 160 MPa. Tytin, representing the Single Melt High Copper amalgam formulation, exhibited significantly higher transverse strength, a fact not inconsistent with quoted literature. The values for transverse strength derived for the tested amalgams are similar to those values recorded by Giblin (1979), but are somewhat lower than those of Maoleekulphiroj (1981). This observation illustrates that the formula by which transverse strength is derived does indeed take into consideration specimen dimensions, when carrying out the normal rapid determination of modulus of rupture in a testing machine.

When the methods of condensation are compared with respect to transverse strengths achieved, most alloys exhibited comparable strengths for the three methods used, with the exception of Amalcap Non-Gamma 2 specimens which were high machine condensed. A significant improvement in strength was achieved with little scatter in results evidenced by the small standard deviation. In the case of Amalcap Non-Gamma 2, this improvement in strength can be explained if the basic geometry of the specimen cavity is

Table III-1 : Modulus of rupture related to condensation method.

Alloy	Modulus of Rupture (MPa)				
	Amalcap F	Hi-Atomic	Amalcap Non-Gamma 2	Dispers-alloy	Tytin
Method of Condensation:					
Hand	156(25.7)*	135(6.9)	113(12.1)	150(25.5)	219(1.7)
Machine 450N	150(11.8)	130(11.8)	124(16.3)	125(14.0)	196(17.0)
Machine 2250N	168(10.5)	125(11.1)	152(3.7)	156(28.1)	202(9.7)

* Standard Deviation in brackets.

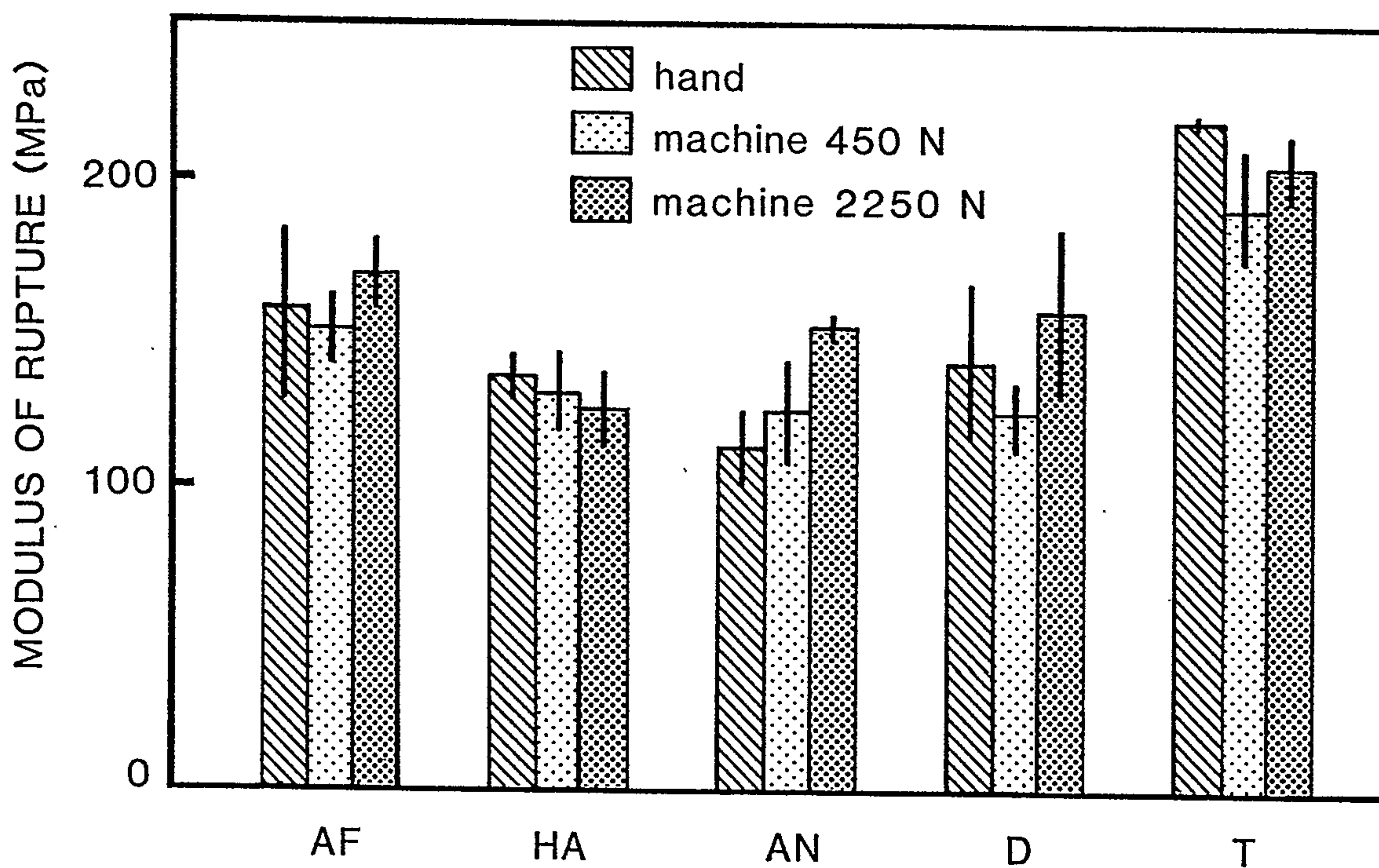


Figure III-1: Modulus of rupture related to condensation method.

* Glossary :

- AF - Amalcap F
- HA - Hi-Atomic
- AN - Amalcap Non-Gamma 2
- D - Dispersalloy
- T - Tytin

Table III-2: Creep values.

Alloy	Creep (%)				
	Amalcap F	Hi-Atomic	Amalcap Non-Gamma 2	Dispers- alloy	Tytin
Test Time:					
3 hr.	3.1 (.04)*	1.2 (.06)	1.0 (.05)	0.4 (.04)	0.11 (.01)
72 hr.	40.0 (2.0)	18.0 (2.4)	11.0 (0.9)	3.1 (.03)	0.50 (.02)

* Standard Deviation in brackets.

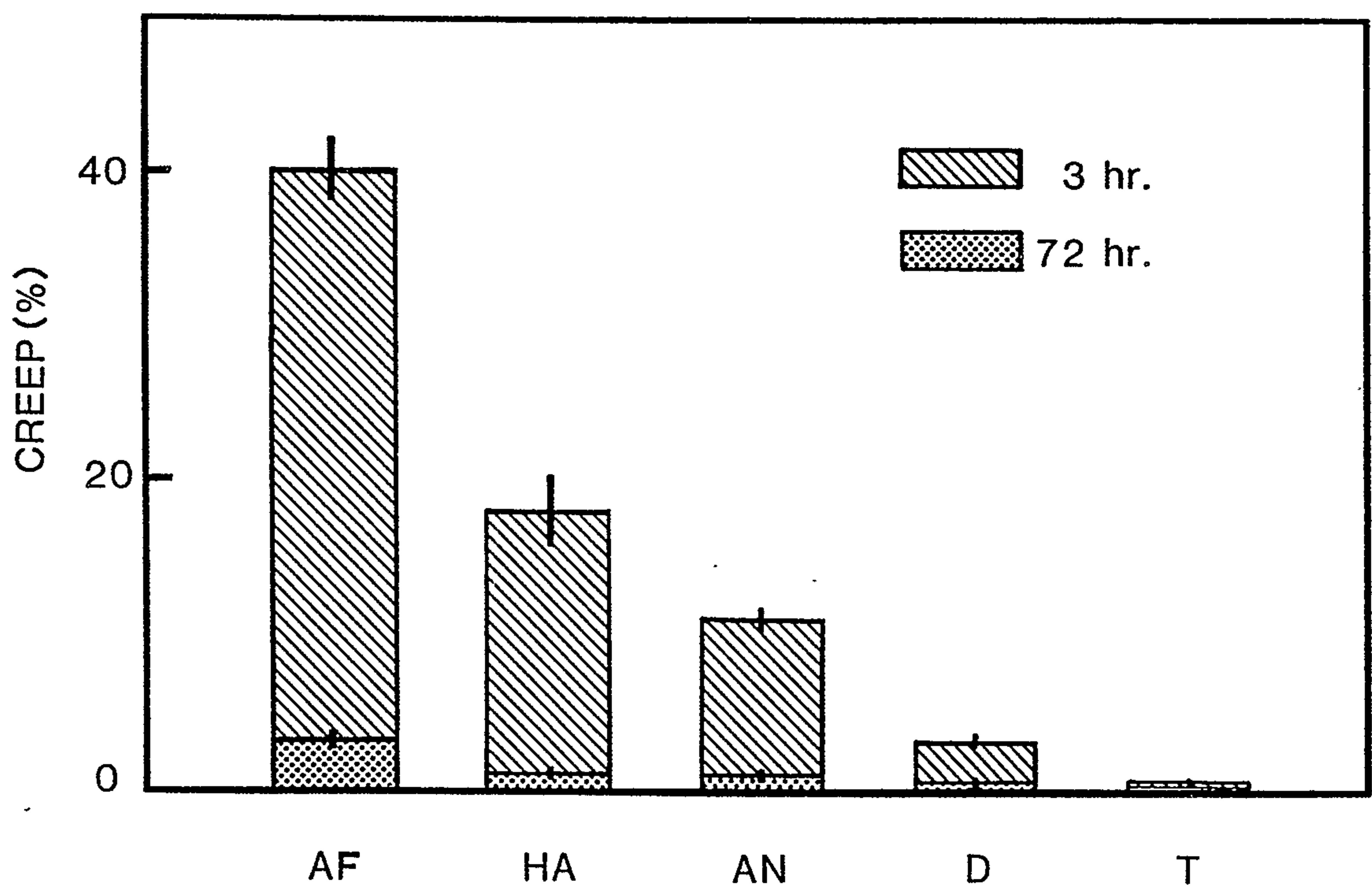


Figure III-2: Creep values.

considered. In contrast to restoration cavities in teeth which are conventionally narrow and deep, the specimen cavity is wide and shallow. The removal of excess mercury is particularly necessary in amalgams such as Amalcap Non-Gamma 2 where the inclusion of excess mercury by the manufacturer has occurred in encapsulated preparations of the alloy. Removal is achieved relatively easier in restoration cavities as multiple condensation thrusts overlap with greater frequency, than in the specimen cavity where a larger surface area must be condensed. Low machine condensing of the Amalcap Non-Gamma 2 specimens was comparable to hand condensing with respect to transverse strength achieved; but with high machine condensing, a greater degree of excess mercury was observed to be extruded so a better quality specimen was produced.

Creep values for the amalgams allow clear discernment between alloy formulations, particularly 72-hour creep values. Values of creep for Ag₃Sn-type formulations and High Copper amalgams showed large differences for 72-hour creep; this contrast was less distinct for 3-hour creep values particularly highlighted in comparing Hi-Atomic and Amalcap Non-Gamma 2. At 72 hours, creep values for these two alloys differ appreciably.

A comparison of creep with modulus of rupture (transverse strength) values indicates no real general correlation apart from the results for Tytin which showed superior results in both tests. Relative ranking of other alloys for creep and transverse strength was interesting - Hi-Atomic showed lower creep values than Amalcap F and also lower transverse strength i.e. a negative relationship, while Dispersalloy with strength values similar to Amalcap F demonstrated significantly lower creep.

Sub-Fracture Loading

Fracture Times

(I) Static Loading

Table III-3 presents the relationship of fracture times of specimens subjected to a static load of 67 MPa at 22°C to method of condensation. Figure III-3 presents this relationship graphically. The temperature of testing (22°C) was used to provide data with which previous studies (Giblin 1979, Maoleekulphiroj 1981), using specimens of different formats, could be compared. Subsequent testing of all amalgams was carried out at 37°C and corresponding results for testing at this temperature are shown in Table III-4 and graphically in Figure III-4. Time to fracture recorded is the mean of seven or eight specimens. The static load of 67MPa used for testing of all amalgams represents approximately 50% of the average modulus of rupture of amalgams tested with the exception of Tytin. This percentage represents a significantly lower static load than that used by Giblin (1979) who employed a range of loads varying from 60% to 95% of modulus of rupture, but compares to that used by Maoleekulphiroj (1981) who used loads varying from 20% to 90% of modulus of rupture.

Results of fracture times at 22°C for specimens for all amalgams machine condensed with 2250N were observed to be significantly long and it was decided to omit these results from Table III-3.

From the data presented in Table III-3, it can be seen that amalgams subjected to a 'sub-fracture' load exhibit a wide range of fracture characteristics. In addition, the behaviour of the amalgam beams appears to relate to the static creep of the amalgam, particularly to the 72-hour determination of this property. An

Table III-3: Fracture time of specimens subjected to a static sub-fracture transverse load (67MPa) at 22°C related to condensation method.

Alloy	Fracture Time (hr.)				
	Amalcap F	Hi-Atomic	Amalcap Non-Gamma 2	Dispersalloy	Tytin
Method of Condensation:					
Hand	17.0 (6.6)*	23.4(12.6)	68.2(19.2)	N.F.†	N.F.
Machine 450N	25.3(10.3)	52.7(14.3)	61.5(14.9)	N.F.	N.F.

* Standard Deviation in brackets.

† No fracture within test period limit.

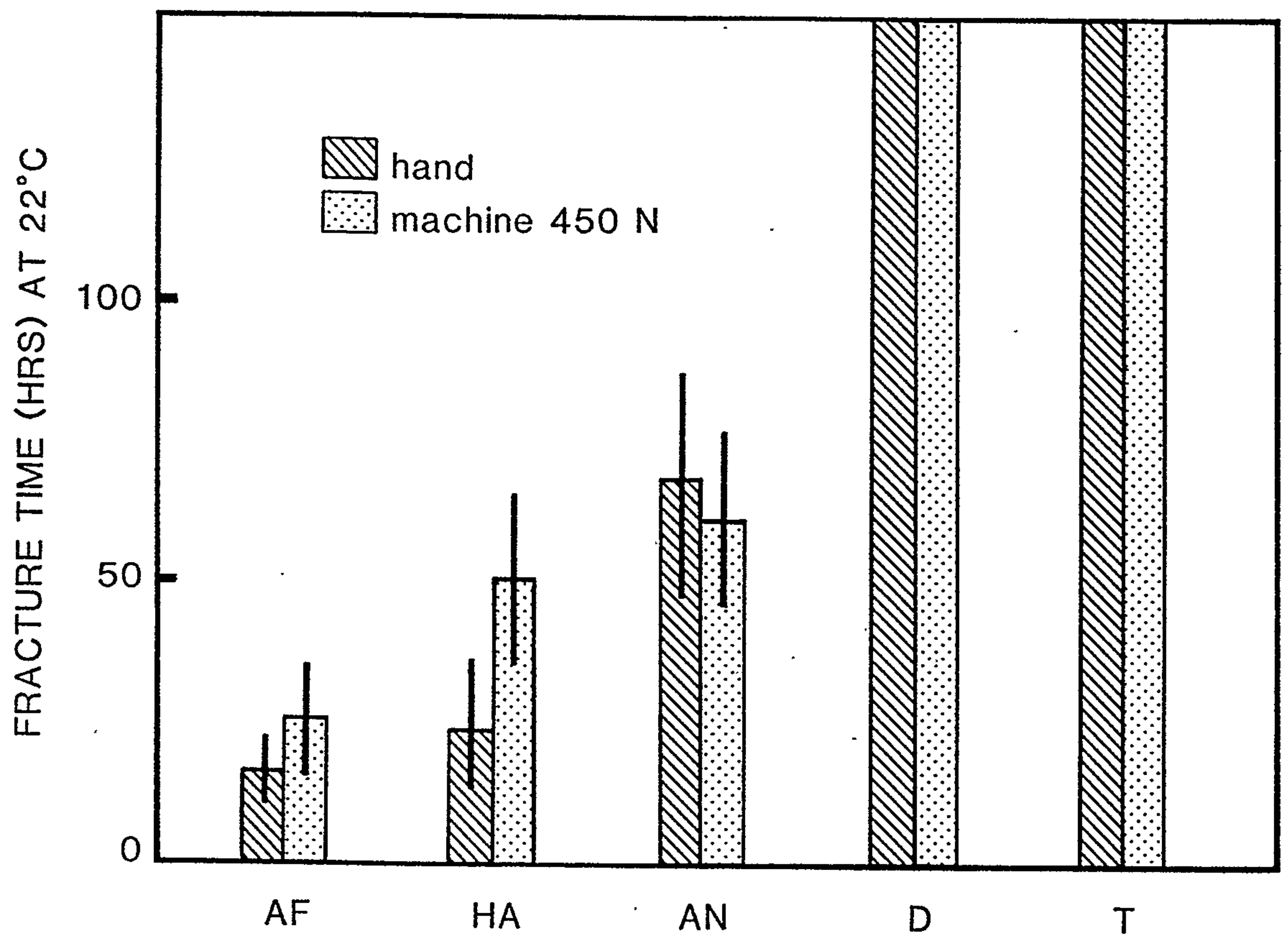


Figure III-3: Fracture time of specimens subjected to a static sub-fracture transverse load (67MPa) at 22°C related to condensation method.

inverse relationship exists between creep and low load fracture. These findings support those of Giblin (1979) and Maoleekulphiroj (1981). All amalgams, except Dispersalloy, and Tytin, exhibited fracture within the test period, within a range of 17 hours to 68 hours. Ag₃Sn-type amalgam specimens fracture at shortest times, around 24 hours. Using the system of categories of fracture times similar to that cited by Giblin (1979)¹ we may similarly divide the performance of various amalgams:

(a) Very short time : $\frac{1}{2}$ hour to 5 hours.

No amalgam specimens tested at 22°C fractured within this period.

(b) Short time : 5 hours to 10 hours.

No amalgam specimens tested at 22°C fractured within this period.

(c) Moderate time : 10 hours to 50 hours.

This group includes hand and low machine condensed (450N) specimens of Amalcap F and hand condensed specimens of Hi-Atomic.

(d) Long time : 50 hours to 100 hours.

This group includes Amalcap Non-Gamma 2 and machine condensed specimens of Hi-Atomic.

(e) Very long time : no fracture within test period limit.

This group includes Dispersalloy and Tytin.

Fracture times for machine condensed specimens were significantly longer for Ag₃Sn-type amalgams than the corresponding times for hand condensed specimens. However, this trend was not observed in the Dispersion Modified amalgam (Amalcap Non-Gamma 2)

1 Giblin, J.P. M.D.S. thesis (1979) Chapter III p.152-153.

suggesting little advantage in machine condensation of specimens over hand condensation in 22°C testing.

In comparing the fracture times of the specimen size used in the current study to those of Giblin (1979) and Maoleekulphiroj (1981), we find that the smaller format specimen has yielded significantly shorter fracture times. Shorter than could be extrapolated from results in the Giblin study which used fracture loads at a minimum of 60% of the respective modulus of rupture and the Maoleekulphiroj study which used a range of loads equal to 20% to 90% of modulus of rupture. This comment is made with the knowledge that only for one alloy - Hi-Atomic - is it possible to make comparisons, Giblin did not quote standard deviations and used an apparatus which was likely to be more effected by external forces such as vibration than the currently developed test equipment.

Maoleekulphiroj used three specimen sizes varying in thickness : 1mm, 2mm and 3mm. Despite the identical thickness used and the comparable load (approximately 50% of modulus of rupture) the current study exhibited fracture times significantly shorter than those of Maoleekulphiroj. This may be explained in terms of method of specimen condensation which in the Maoleekulphiroj study was accomplished using a Hounsfield Tensometer under a force of 1,500 pounds producing a packing pressure of 56.5 MPa which was considerably higher than the condensation pressures used in this study.

The inverse relationship of creep to beam behaviour and the relative order of the amalgams in results made in the Giblin study were supported in the current study. In addition, fracture times

were observed to relate closely to 72-hour creep, suggesting an advantage in extended creep testing.

Table III-4 presents the relationship of fracture times of specimens subjected to a static load (67MPa) at 37°C to method of specimen condensation. Specimens which were high machine condensed yielded fracture times within the test period for all amalgams except Dispersalloy and Tytin and thus these results are included. Figure III-4 presents this relationship graphically.

A wide range of static fracture behaviour was observed for all amalgams with the exception of Tytin which exhibited no fractures within the test period limit. Shortest times to fracture were exhibited by Ag₃Sn-type amalgams and an inverse relationship of fracture behaviour to 3-hour and especially 72-hour creep values was observed. In comparing the advantage of different methods of condensation on the fracture times it can be seen that machine condensation at higher forces offers little improvement in the performance of Ag₃Sn-type amalgams. However, Dispersion Modified amalgams were affected by differing condensation loads. Fracture times of high machine condensed specimens of Amalcap Non-Gamma 2 were significantly greater than comparable results for the other condensation methods. Low machine condensation yielded Dispersalloy specimens which were less able to withstand sub-fracture loading compared with hand condensed specimens. Fracture times of hand condensed Dispersalloy specimens were significantly higher than all other hand condensed results with the exception of Tytin. Generally, a constant trend was noted for low load mechanical condensation to be worse than hand or high load mechanical condensation.

Table III-4: Fracture time of specimens subjected to a static sub-fracture transverse load (67MPa) at 37°C related to condensation method.

Alloy	Fracture Time (hr.)				
	Amalcap F	Hi-Atomic	Amalcap Non-Gamma 2	Dispersalloy	Tytin
Method of Condensation:					
Hand	4.7 (3.4)*	7.6 (2.6)	20.3 (4.4)	130.0(28.8)	N.F.†
Machine 450N	2.9 (2.6)	5.0 (2.7)	18.2 (4.7)	48.2(11.1)	N.F.
Machine 2250N	5.6 (2.4)	13.9 (7.4)	100.9(10.4)	N.F.	N.F.

* Standard Deviation in brackets.

† No fracture within test period limit.

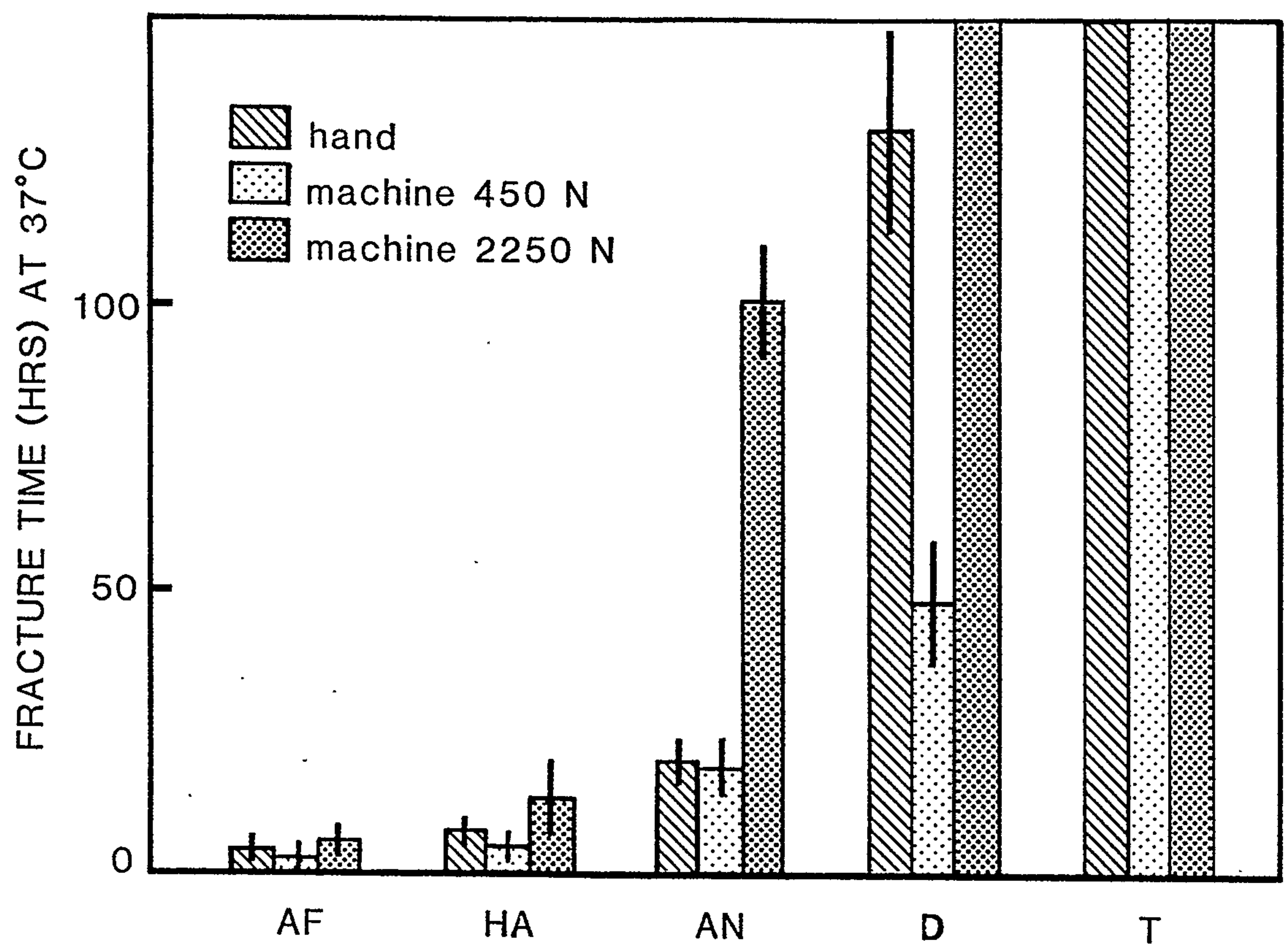


Figure III-4: Fracture time of specimens subjected to a static sub-fracture transverse load (67MPa) at 37°C related to condensation method.

In categorizing fracture times in the broad groups suggested by Giblin (1979) and already applied to 22°C testing, we find that a closer correlation to the relative positions of the amalgams as determined by Giblin are seen in the current study for specimens tested at 37°C:

- (a) Very short time : ½ hour to 5 hours.

This group includes hand and low machine specimens of Amalcap F and low machine specimens of Hi-Atomic.

- (b) Short time : 5 hours to 10 hours.

This group includes high machine specimens of Amalcap F and hand condensed specimens of Hi-Atomic.

- (c) Moderate time : 10 hours to 50 hours.

Included here are high machine specimens of Hi-Atomic, hand and low machine specimens of Amalcap Non-Gamma 2 and low machine specimens of Dispersalloy.

- (d) Long time : 50 hours to 100+ hours.

Hand condensed specimens of Dispersalloy and high machine specimens of Amalcap Non-Gamma 2 are included here.

- (e) Very long time : no fracture within test period limit.

All Tytin specimens and high machine specimens of Dispersalloy are included here.

Fracture times at 37°C for all amalgams irrespective of method of condensation, with the exception of Tytin, were significantly shorter than corresponding values at 22°C. Table III-5 illustrates this observation in a comparison of 37°C and 22°C results for all amalgams specimens which were low machine condensed. A graphic representation of this is found in Figure III-5. The advantage of 37°C testing is thus a practical one in the significant reduction

Table III-5 : Comparison of fracture times of specimens tested at 37°C and 22°C.

Alloy	Fracture Time (hr.)				
	Amalcap F	Hi-Atomic	Amalcap Non-Gamma 2	Dispers-alloy	Tytin
Machine Packed 450N					
Temperature of Testing:					
37°C	2.9 (2.6)*	5.0 (2.7)	18.2 (4.7)	48.2(11.1)	N.F.†
22°C	25.3(10.3)	52.7(14.3)	61.5(14.9)	N.F.	N.F.

* Standard Deviation in brackets.

† No fracture within test period limit.

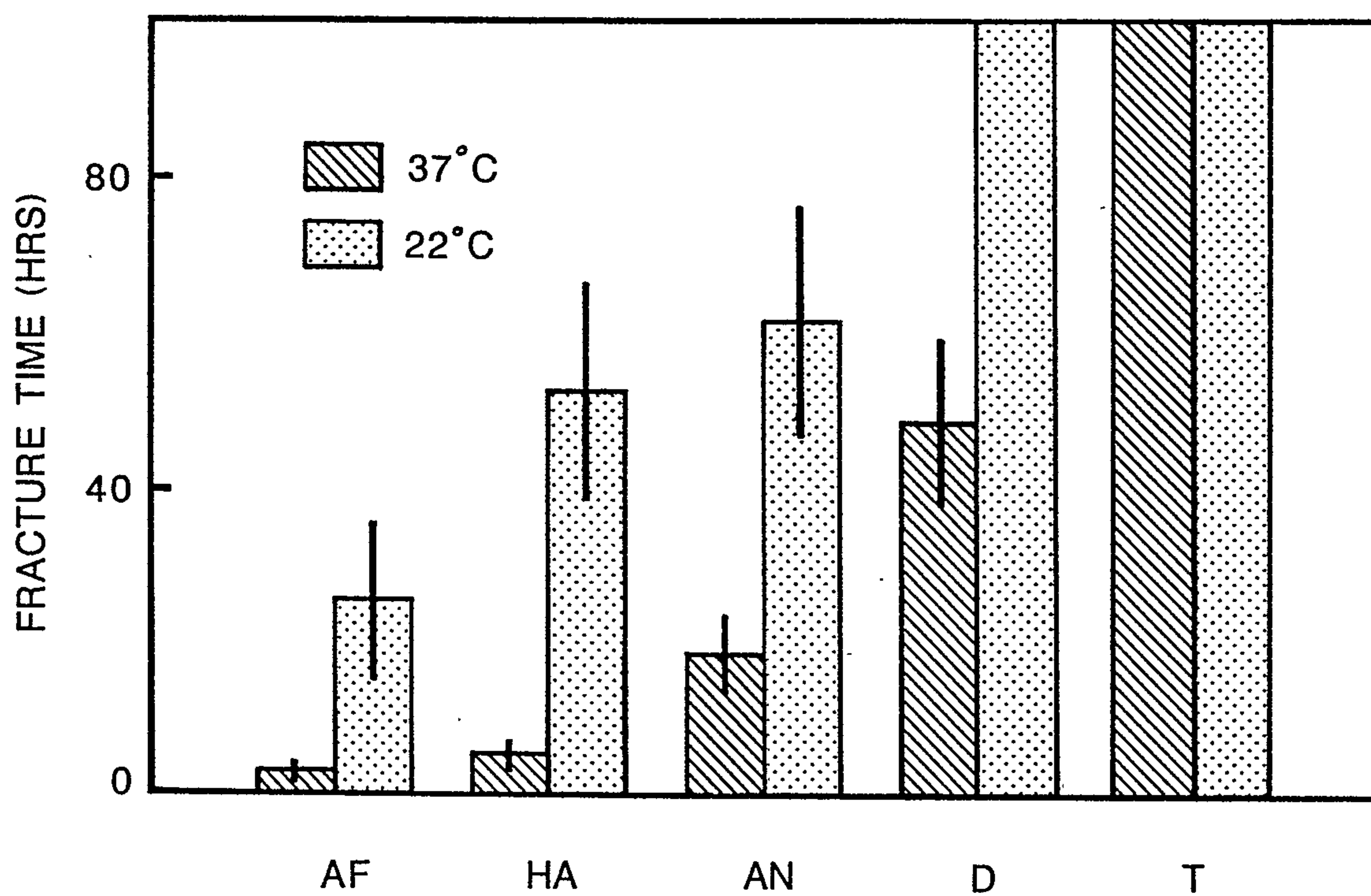


Figure III-5: Comparison of fracture times of specimens condensed by low machine load and tested at 37°C and 22°C.

of fracture times, the correlation to mouth temperature and especially relevance to creep testing which is also conducted at 37°C. A further advantage is the bringing of "resistant to fracture" alloys such as Dispersalloy at 22°C to a fracture stage in a significantly longer but finite time, compared with other fracturing amalgams.

(II) Cyclic loading

Table III-6 presents the fracture times of specimens subjected to a cyclic sub-fracture load at 37°C related to method of condensation. The exact procedure followed for this testing has been outlined in Chapter II 'Experimental Procedures'. Figure III-6 presents this data graphically. The times presented are total times of load application and do not include "time off". Cyclic testing was restricted to amalgams exhibiting highest creep, namely Hi-Atomic and Amalcap F and results represent the mean of three specimens.

Hi-Atomic specimens withstood sub-fracture loading for longer periods than Amalcap F specimens; this was most significant for low machine condensed specimens.

A comparison of fracture times for Hi-Atomic and Amalcap F specimens subjected to cyclic and static loads is presented in Table III-7 and graphically in Figure III-7. Respective results for the two amalgams tested show no significant difference in fracture times with the exception of Hi-Atomic specimens manufactured by low machine condensation which achieved superior results in cyclic loading.

These results suggest that the deformation taking place in the specimens during sub-fracture loading does not undergo any

Table III-6: Fracture time of specimens subjected to a cyclic sub-fracture transverse load (67MPa) at 37°C related to condensation method.

Alloy	Fracture Time (hr.)	
	Amalcap F	Hi-Atomic
Method of Condensation:		
Hand	4.7 (1.0)*	7.7 (0.1)
Machine 450N	5.0 (2.0)	23.4(11.9)
Machine 2250N	12.9 (8.4)	14.5 (6.3)

* Standard Deviation in brackets.

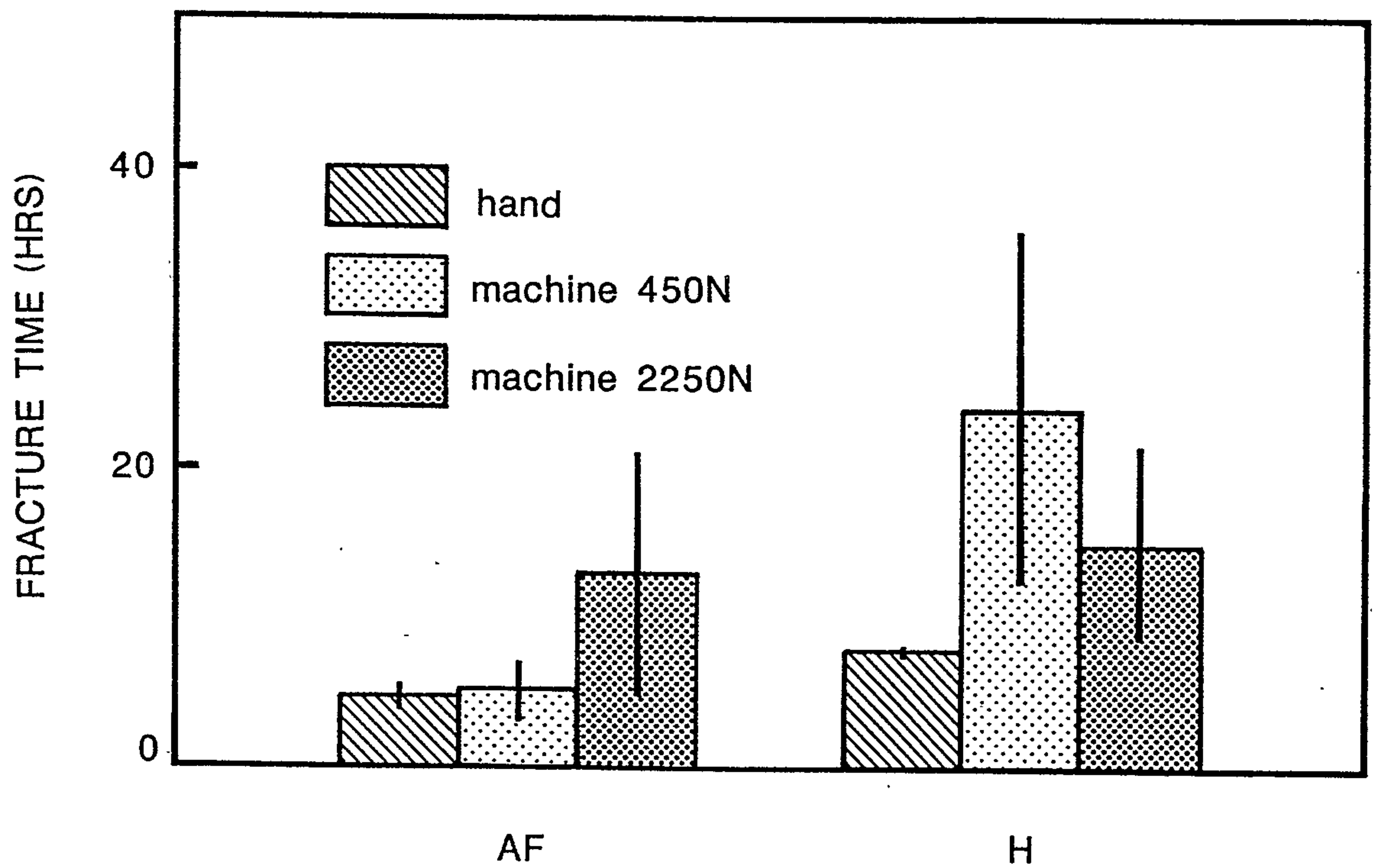


Figure III-6: Fracture time of specimens subjected to a cyclic sub-fracture transverse load (67MPa) at 37°C related to condensation method.

Table III-7: Comparison of fracture times for specimens subjected to cyclic and static sub-fracture transverse loads (67MPa) at 37°C related to condensation method.

Alloy	Fracture Time (hr.)			
	Amalcap F		Hi-Atomic	
	Static	Cyclic	Static	Cyclic
Method of Condensation:				
Hand	4.7 (3.4)*	4.7 (1.0)	7.6 (2.6)	7.7 (0.1)
Machine 450N	2.9 (2.6)	5.0 (2.0)	5.0 (2.7)	23.4(11.9)
Machine 2250N	5.6 (2.4)	12.9 (8.4)	13.9 (7.4)	14.5 (6.3)

* Standard Deviation in brackets.

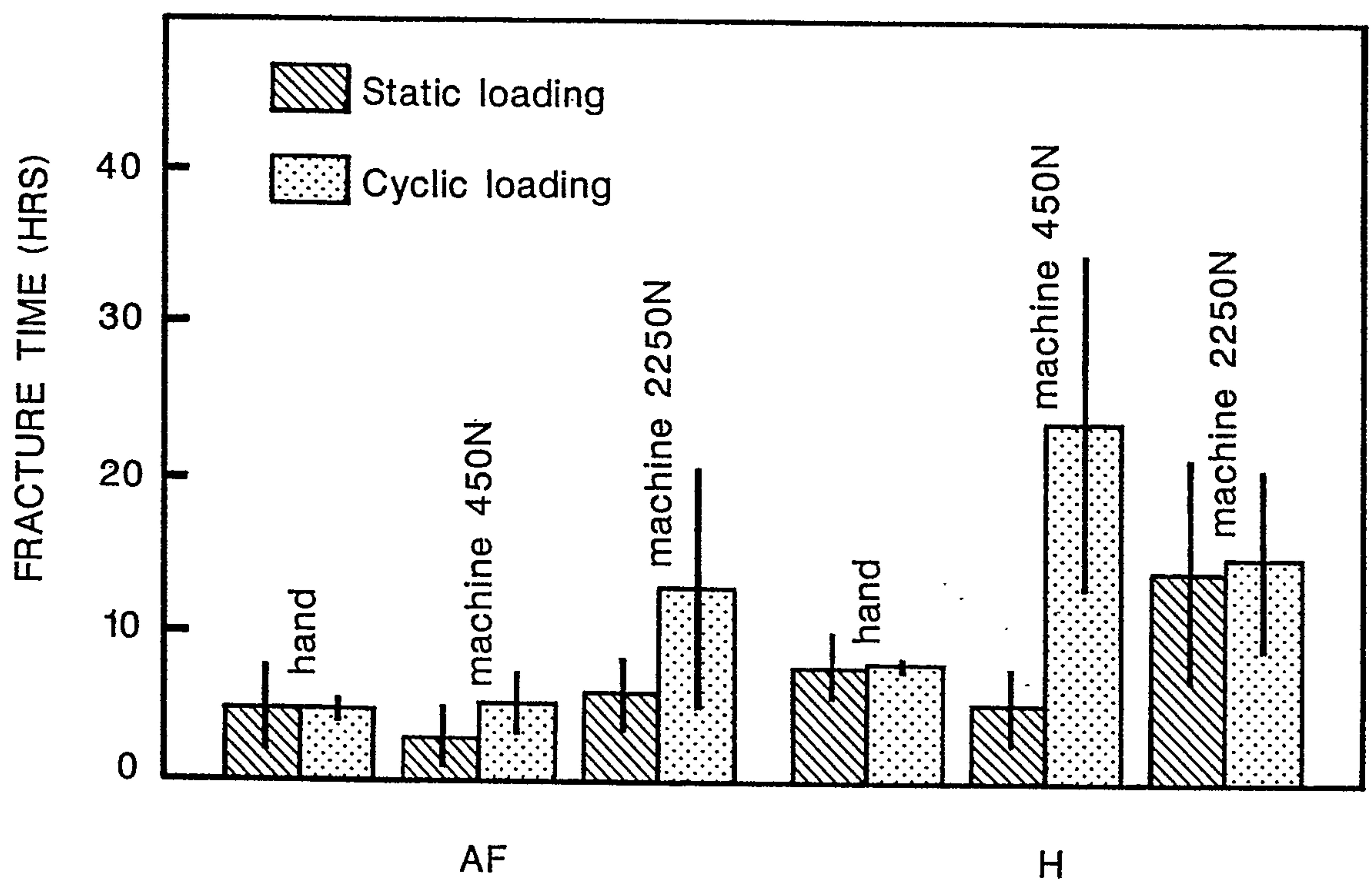


Figure III-7: Comparison of fracture times for specimens subjected to cyclic and static sub-fracture transverse loads (67MPa) at 37°C, related to condensation method.

significant degree of recovery if the loading is relieved as during cyclic loading. This observation is not unexpected in view of the parallel observed by Mahler between static and dynamic creep testing (Mahler, 1969).

Bending Characteristics

(I) Static Loading

As outlined in Chapter II 'Experimental Procedures', fracture behaviour of every specimen subjected to sub-fracture loading, both cyclic and static, was assessed by virtue of assigning an arbitrary value to the degree of bend as determined from the scale in Figure II-5. Specimens which fractured were re-assembled and graded as were all unfractured specimens. A grading system devised by Giblin and Wing (1979) was used. It assigned a ranking of '1' to untested specimens and '10' to specimens exhibiting greatest relative bend.

Table III-8 presents the mean bend and standard deviation of each category of specimen which had been subjected to static loading. In addition, method of condensation and temperature of testing are also considered as these factors affect bend. Also included in the table for all specimens is an indication of whether or not fracture actually occurred. In this table, 'F' denotes fracture during the test period (preceded by bend ranking) and 'NF' denotes no fracture had occurred (also preceded by bend ranking). In some cases, a ranking of degree of bend for all specimens within the group was uniform and no scatter of results was recorded. From Table III-8, several observations can be made :

(i) Bend behaviour of all specimens tested fell into two main groups; specimens which did not fracture but exhibited bend and specimens which exhibited bend and ultimately fractured. Specimens

Table III-8: Bend characteristics of specimens subjected to a static sub-fracture transverse load (67 MPa) related to condensation method and temperature of testing.

Alloy	Bend Characteristics				
	Amalcap F	Hi-Atomic	Amalcap Non-Gamma 2	Dispers-alloy	Tytin
Method of Condensation:					
Hand 37°C	8.8 (0.8)*F†	5.0 (1.9)F	9.0 (1.6)F	8.0 (2.8)F	2 NF§
22°C	9.6 (1.0)F	5.4 (1.5)F	9.7 (0.8)F	4 NF	2 NF
Machine 37°C	8.0 (1.2)F	5.0 (1.3)F	9.2 (1.1) F	8.8(1.8)F	2 NF
450N 22°C	9.0 (1.0)F	6.3 (1.7)F	10 F	3 NF	2 NF
Machine 37°C	8.3 (1.5)F	5.2 (0.5)F	6.6 (4.8)F	3 NF	2 NF
2250N					

* Standard Deviation in brackets.

† Bend prior to fracture.

§ No fracture, bend at end of test period limit.

which fell into the first group did not attain a bend greater than '4' and included all Tytin specimens and some Dispersalloy specimens. The second group encompassed the majority of specimens which, irrespective of alloy formulation, method of condensation and temperature of testing, suffered bend greater than '5' and ultimately fracture. In this second group are all Ag₃Sn-type amalgams and Dispersion Modified amalgams except Dispersalloy specimens tested at 22°C. From this observation, we may consider a bend ranking of '5' to be a 'critical' bend which, if attained, commits the specimen to ultimate fracture. If bend is maintained below '5', fracture will not occur within the test period. This identification of a 'critical' bend differs from the findings of Giblin (1979) and Maoleekulphiroj (1981) where fractured specimens featured bends of all ranks. This difference can be explained in terms of the times to fracture exhibited by specimens in both the previous studies compared to the times to fracture found here. The bulkier specimens used by Giblin were more resistant to loading hence fracture times were extended allowing time for bending to occur. Fracture ultimately took place as a result of physical changes occurring within the amalgam. The degree of bend noted by Maoleekulphiroj (1981) for Hi-Atomic specimens 2mm thick and subjected to a load equal to 40% of modulus of rupture was comparable to Hi-Atomic specimens in the current study although fracture did not occur. Above 40% modulus of rupture loading, Maoleekulphiroj found cracking in Hi-Atomic specimens suggesting a critical degree of bend had been achieved.

(ii) Bends achieved by specimens prepared by the three methods of condensation were not significantly different within the specific

amalgam groups. This was particularly evident in the bend behaviour of Hi-Atomic specimens where the bends recorded at 37°C for specimens made by hand condensation, low machine condensation and high machine condensation were 5.0(1.9), 5.0(1.3) and 5.2(0.5) respectively.

(iii) The temperature of testing did not significantly affect bend characteristics for all amalgams tested with the exception of Dispersalloy where very little bending was noted in specimens tested at 22°C but significant bend was observed at 37°C.

(iv) In applying data of modulus of rupture (Table III-1) and fracture times (Table III-3 and Table III-4) to bend characteristics it could be seen that the amalgam of highest transverse strength (Tytin) best withstood bend testing despite extended times of testing. Conversely, Amalcap Non-Gamma 2 with the lowest modulus of rupture showed a time related resistance to bending and fracture approximately four times that of Amalcap F with a higher modulus of rupture.

(v) When bend characteristics were compared to creep values (Table III-2) it was noted that the amalgam exhibiting least bend was the amalgam of least creep. However, the clear distinction between amalgams afforded by 72-hour creep values cannot be re-affirmed purely by a consideration of bend characteristics alone. Bend appears to be related to many factors including time and creep.

(II) Cyclic Loading

Table III-9 presents the mean bend and standard deviation of specimens of Hi-Atomic and Amalcap F which were subjected to cyclic sub-fracture loading at 37°C. From data presented in Table III-9 it can be noted that little variation in degree of bend was

Table III-9: Bend characteristics of specimens subjected to a cyclic sub-fracture transverse load (67MPa) at 37°C related to condensation method.

Alloy	Bend Characteristics	
	Amalcap F	Hi-Atomic
Method of Condensation:		
Hand	9.3 (1.2)F	7.7 (2.5)*F†
Machine 450N	9.7 (0.6)F	7.5 (3.3)F
Machine 2250N	9.3 (1.2)F	8.0 (2.0)F

* Standard Deviation in brackets.

† Bend prior to fracture.

observed when specimens of both alloys made by different methods of condensation were compared. All specimens fractured and achieved bends greater than '7'. No significant differences were present at the 98% confidence level.

Table III-10 compares the bend characteristics of specimens prepared from Hi-Atomic and Amalcap F subjected to static and cyclic sub-fracture loads. The consistent degree of bend exhibited for each alloy irrespective of method of specimen condensation is seen in data for both cyclic and static loading. Cyclically loaded specimens of both amalgams exhibited degrees of bend uniformly but marginally greater than corresponding results for static loaded specimens. These results may be compared to fracture times for these amalgams shown in Table III-7 which showed no significant difference for these times in the static and cyclic tests.

Structural Observations

(I) Macroscopic Examinations

As noted previously, when amalgam beam specimens are subjected to sub-modulus of rupture loading they undergo varying degrees of bend and may ultimately fracture. Bend of both unfractured and re-assembled specimens prior to fracture was obvious macroscopically. The majority of specimens which fractured did so by simple cleavage of the specimen into two parts of approximately equal proportions with the fracture line commencing at the inferior surface of the specimen opposite the point of load application.

Where the loader and bearer rods made contact with the superior and inferior surfaces of the specimen respectively, discrete grooves traversing the width of the specimen could be discerned macroscopically. In all cases of reassembled fractured

Table III-10: Comparison of bend characteristics of specimens subjected to cyclic and static sub-fracture transverse loads (67MPa) at 37°C related to condensation method.

Alloy	Bend Characteristics			
	Amalcap F		Hi-Atomic	
	Static	Cyclic	Static	Cyclic
Method of Condensation:				
Hand	5.0 (1.9)*F†	7.7 (2.5)F	8.8 (0.8)F	9.3 (1.2)F
Machine 450N	5.0 (1.3)F	7.5 (3.3)F	8.0 (1.2)F	9.7 (0.6)F
Machine 2250N	5.2 (0.5)F	8.0 (2.0)F	8.3 (1.5)F	9.3 (1.2)F

* Standard Deviation in brackets.

† Bend prior to fracture.

specimens, the main crack through the specimen originating at the inferior surface ended in the groove on the superior surface of the specimen made by the contact of the loader with the specimen.

In some cases, additional minor cracking was seen in the lower half of the specimens. Such cracks generally ran parallel to the main crack but ended approximately half-way through the thickness of the specimen and did not ultimately result in specimen fracture.

(II) Microscopic Examinations

Mounted, etched specimens were viewed at magnifications of 10X, 50X, and 500X. Photographs taken at these magnifications are presented.

The appearance of a typical fractured specimen of Amalcap F tested at 37°C is shown at magnifications of 10X and 50X in Figure III-8(a) and Figure III-8(b) respectively. Such a specimen fractured in approximately 5 hours and attained a bend of '8'. From Figure III-8(a) we may see the substantial bend attained by the specimen and the crack path through the specimen. At a higher magnification of 50X as shown in Figure III-8(b), the 'fast zone' and 'slow zone' of a typical crack path cited by Giblin (1979) can be seen. The 'slow zone' is characterized by an irregular, wide crack path located at the lower or tensile region of the specimen and is the first stage of specimen failure. Continuing from the 'slow zone' is a portion of the crack which ultimately results in specimen failure, the 'fast zone'. This zone is straighter, narrower and more uniform than the slow zone and is located in the uppermost or compressive half of the specimen. Also in Figure III-8(b) some minor secondary cracks originating at the tensile surface can be observed.

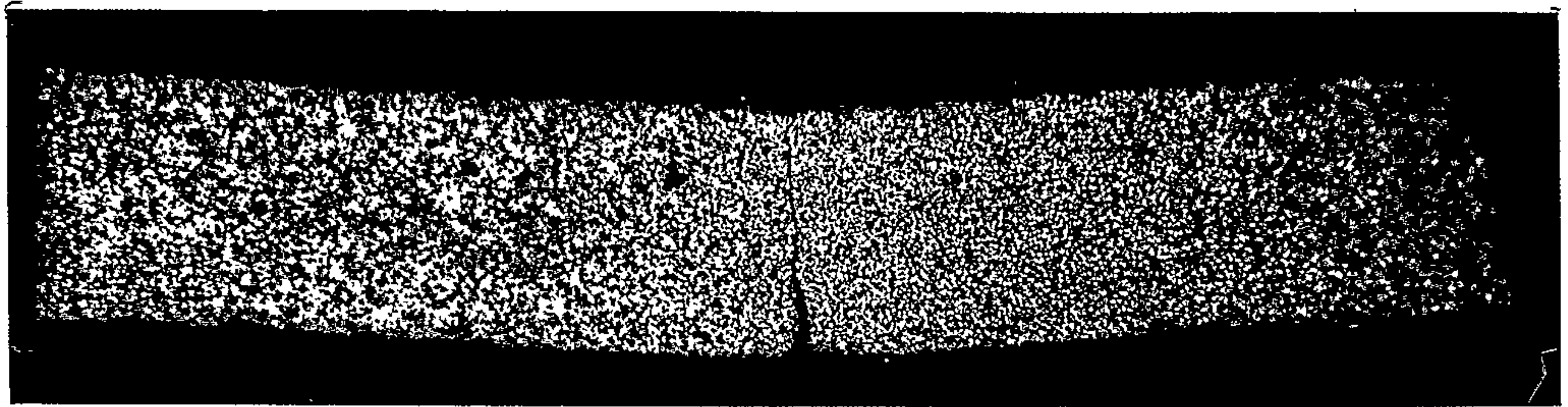


Figure III-8(a): A specimen of Amalcap F tested at 37°C
at a magnification of 10X.

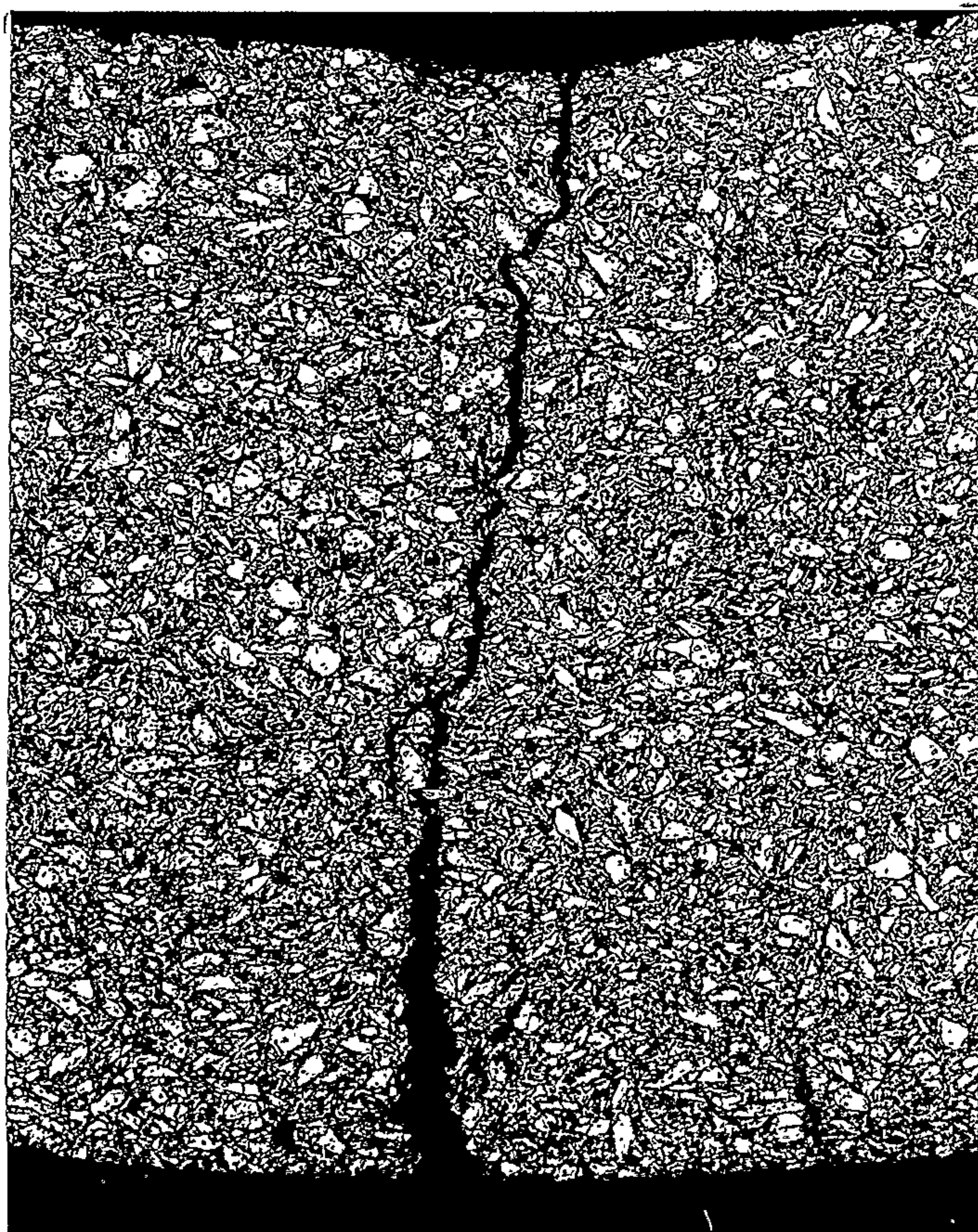


Figure III-8(b): A 50X micrograph of the specimen shown
in Figure III-8(a).

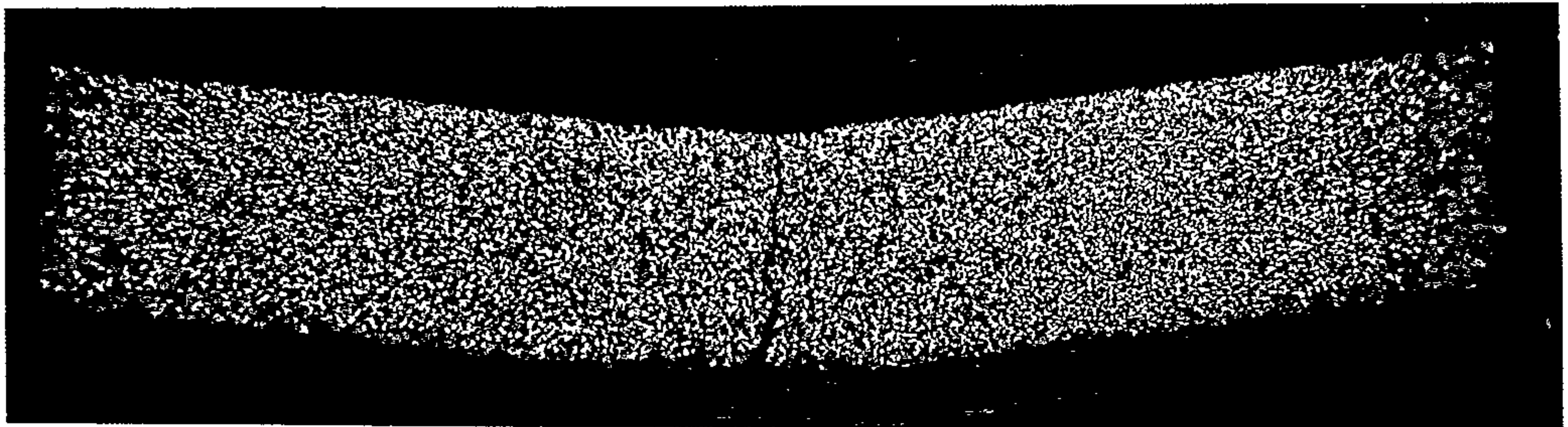


Figure III-9(a): A specimen of Amalcap F tested at 22°C
at a magnification of 10X.

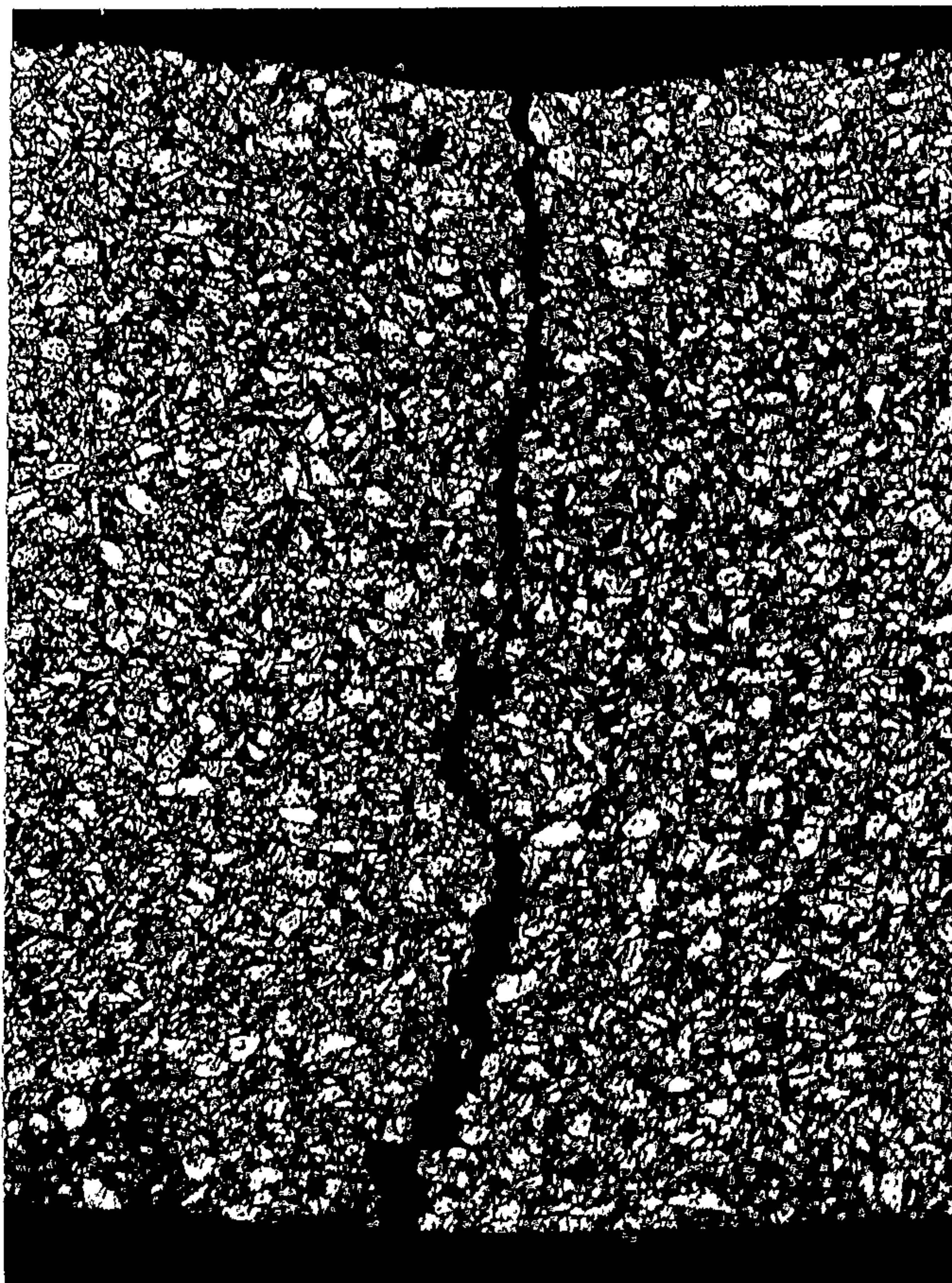


Figure III-9(b): A 50X micrograph of the specimen shown
in Figure III-9(a).

Figure III-9(a) presents a 10X micrograph of a typical Amalcap F specimen tested at 22°C. Figure III-9(b) shows this specimen at 50X magnification. This specimen failed at 17 hours and attained a bend of '9'. Distinct fast zone and slow zone failure may be observed. In comparing failure of specimens of Amalcap F tested at 37°C and 22°C it can be seen that despite the differing times to fracture, a similar degree of bend was attained with similar microstructural features of crack propagation in both tests.

A typical failed specimen of Hi-Atomic tested at 37°C is shown at magnifications of 10X and 50X in Figures III-10(a) and III-10(b) respectively. Distinct slow and fast zones of the failure path can be observed. An exceptionally uniform fast zone may be seen in Figure III-10(b). The slow zone is an irregular path, much wider than the fast zone. This specimen fractured in 7 hours and attained a bend of '5'.

Figures III-11(a) and III-11(b) present micrographs of a fractured specimen of Hi-Atomic tested at 22°C at magnifications of 10X and 50X respectively. A bend of '8' was attained by this specimen with failure occurring in 23 hours. This specimen illustrates several interesting features of unusual fracture path progression. From Figure III-11(a) we may note that at least two slow zone cracks have originated at the tensile surface. Neither of these cracks is located directly opposite the loader bar contact on the compressive surface of the specimen and so the point on the tensile surface of maximum tensile stress has avoided failure. Crack progression has developed, in one of these secondary cracks, into fast zone failure and the path chosen by this crack has deviated through the specimen to converge at the point of greatest compressive stress. Figure III-11(b) illustrates this crack progression at a

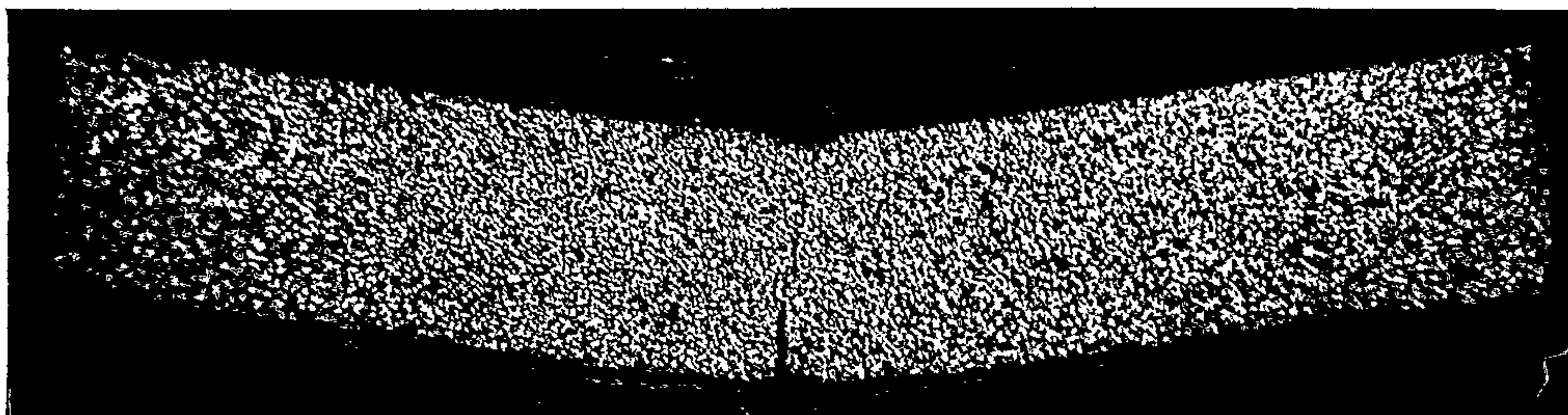


Figure III-10(a): A specimen of Hi-Atomic tested at 37°C
at a magnification of 10X.

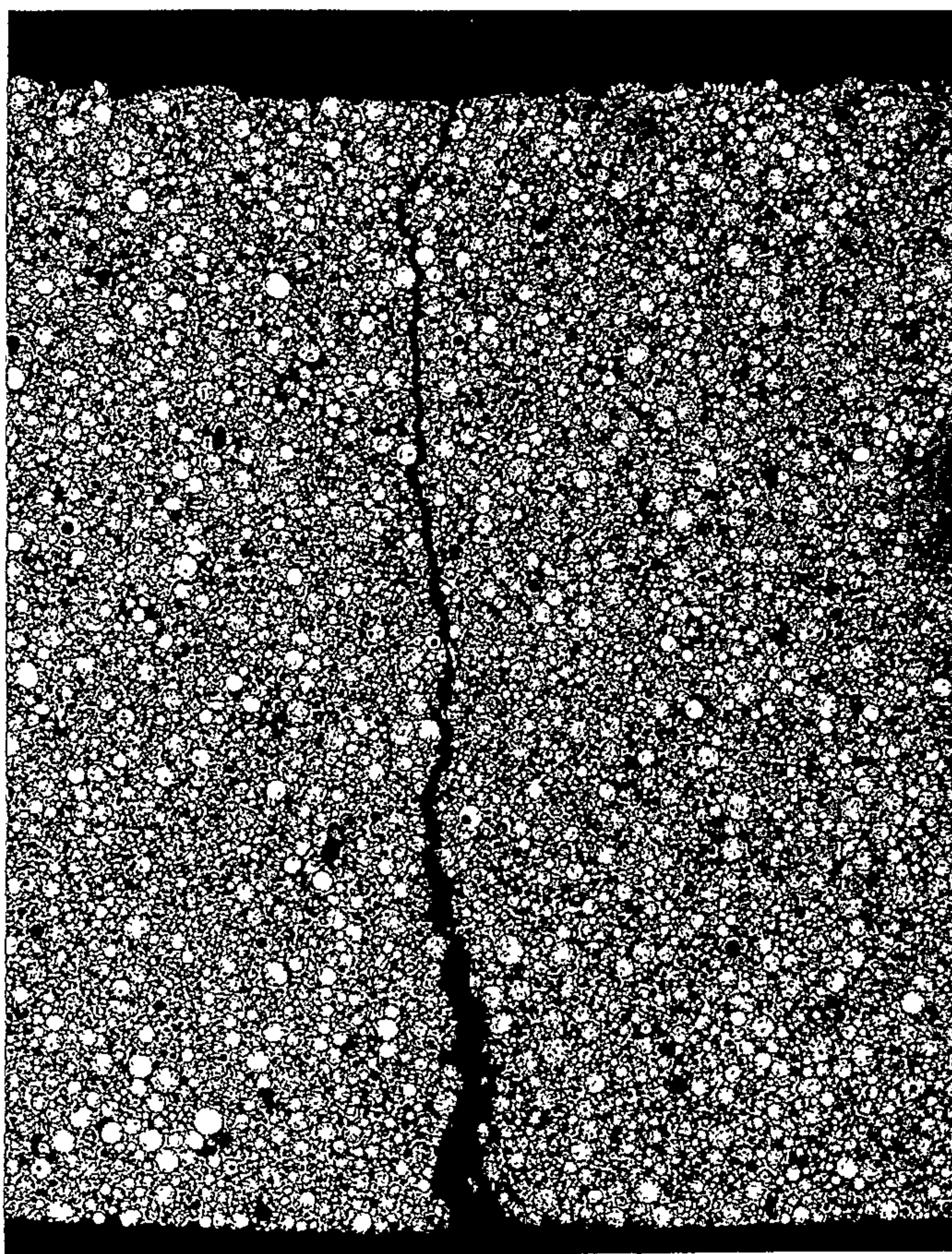


Figure III-10(b): A 50X micrograph of the specimen shown
in Figure III-10(a).

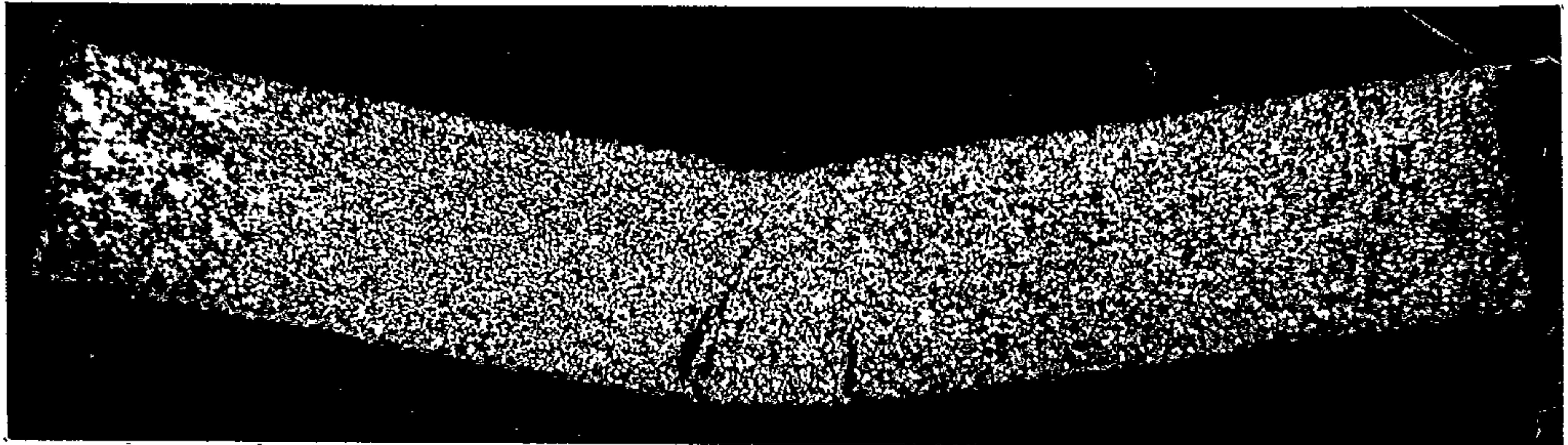


Figure III-11(a): A specimen of Hi-Atomic tested at 22°C
at a magnification of 10X.

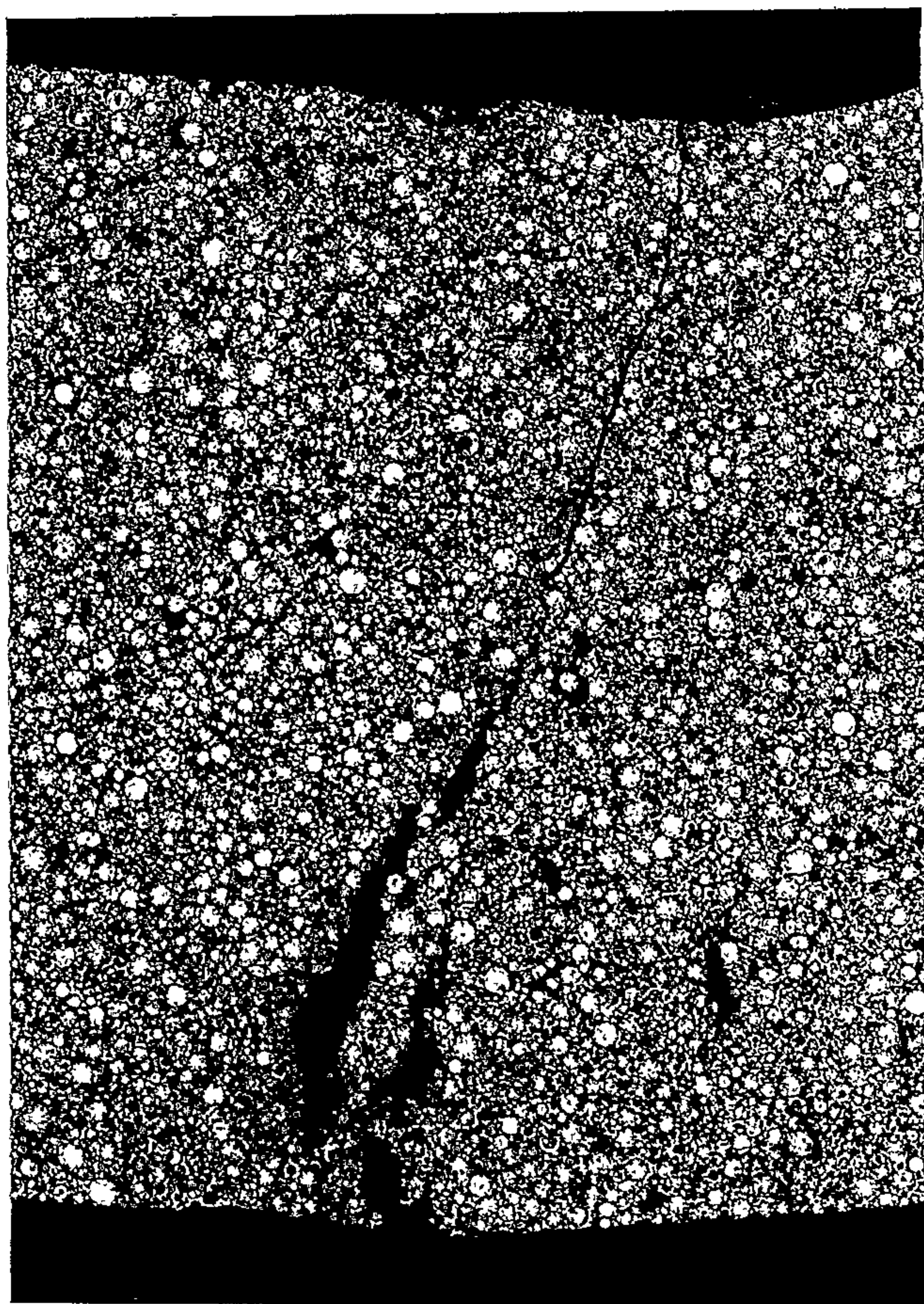


Figure III-11(b): A 50X micrograph of the specimen shown
in Figure III-11(a).

higher magnification. Crack initiation is nevertheless opposite the loader although not at the base of the indentation convexity. Initiation may have been influenced by structural elements such as voids or gamma 2 inclusions. Micrographs of this specimen at a magnification of 500X will be presented in later figures.

In comparing the microstructural features of fractured specimens of Amalcap F and Hi-Atomic thus far presented, several observations about failure of Ag₃Sn gamma 2-containing amalgams may be made:

- (i) The fracture path contains features of slow zone and fast zone failure as described by Giblin (1979).
- (ii) The fracture usually originates ^{in the region} at a point of greatest tensile stress on the tensile surface but may also originate from secondary cracks nearby related to the point of greatest tensile stress.
- (iii) The crack usually terminates on the compressive surface at a point of greatest compressive stress.
- (iv) Features of crack progression appear to relate to particle morphology and distribution but are independent of temperature of testing and loading time.

Figure III-12(a) and III-12(b) present micrographs of a typical failed specimen of Dispersalloy tested at 37°C at magnifications of 10X and 50X respectively, representative of Dispersion Modified amalgam failure. This specimen failed in 70 hours having attained a bend of '8'. In examining Figure III-12(b) distinct fast zone and slow zone features are obvious but the relative lengths of these zones differs to that observed in previous specimens thus far considered. The irregular, disjointed slow zone extends to only approximately one third of the specimen

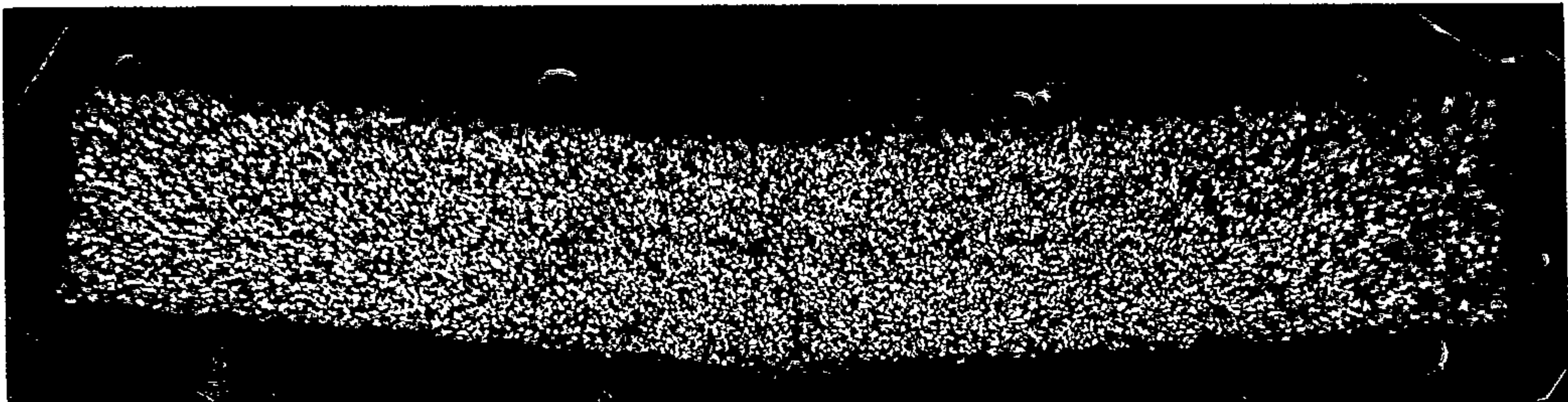


Figure III-12(a): A fractured specimen of Dispersalloy tested at 37°C at a magnification of 10X.

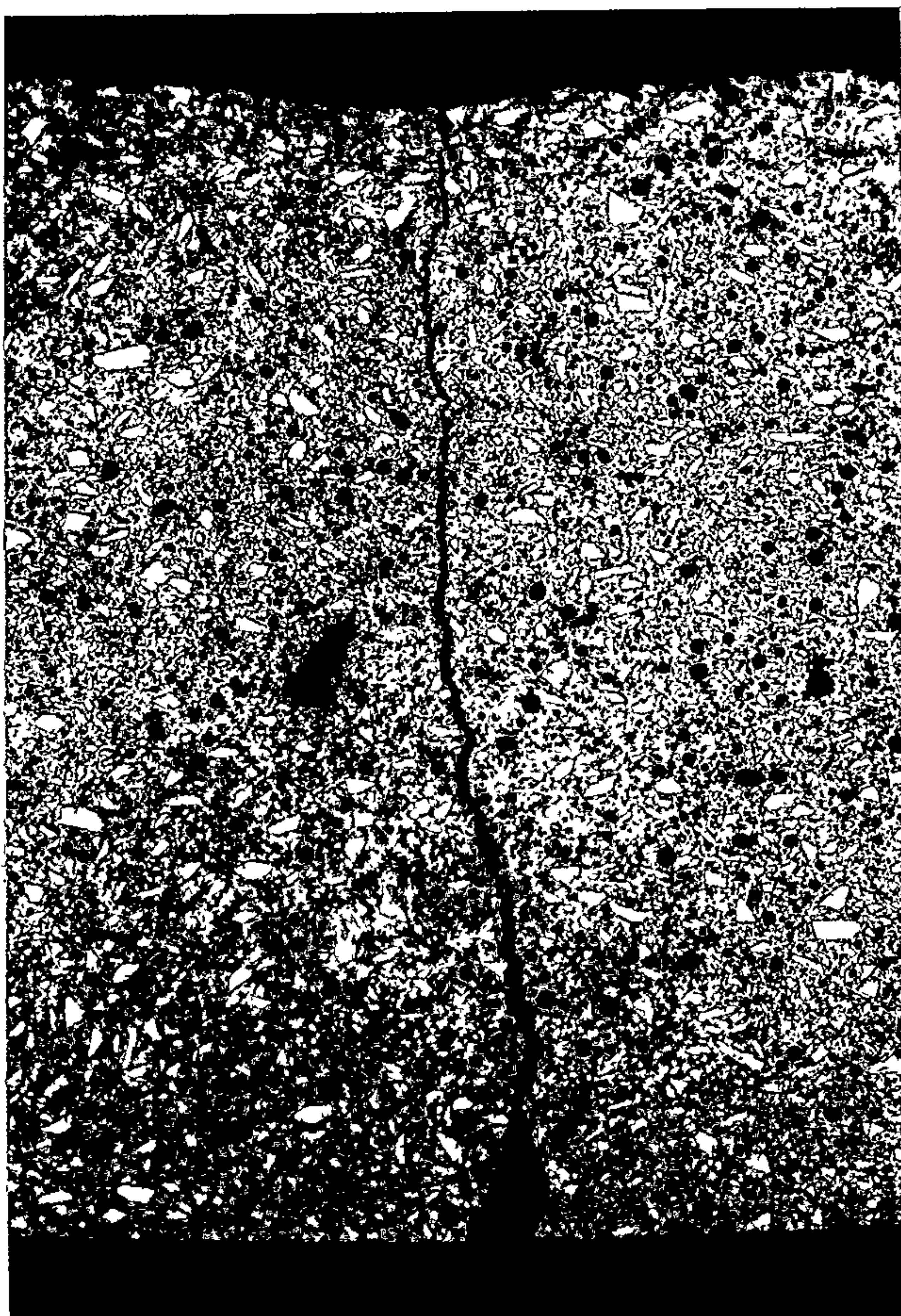


Figure III-12(b): A 50X micrograph of the specimen shown in Figure III-12(a).

thickness. A uniform, narrow fast zone continues from this slow zone to termination at the compressive surface.

A typical, unfractured, deformed specimen of Dispersalloy tested at 22°C at magnifications of 10X and 50X is shown in Figures III-13(a) and III-13(b) respectively. This specimen failed to fracture within the test period limit of 5 weeks. The bend which occurred was a '4'. This specimen appears to have predominantly suffered a very irregular slow zone cleavage with secondary cracking which extends to one third of the specimen thickness. The tip of the slow zone crack may be seen. In comparing the fractured and unfractured specimens of Dispersalloy, several observations may be made:

- (i) Fracture of Dispersion Modified amalgams is characterized by short slow zone and long fast zone failure.
- (ii) Specimens which do not fracture exhibit a lesser degree of bend but a slow zone similar in length and morphology to that exhibited in fractured specimens.
- (iii) Secondary cracking is a frequently observed feature of both fractured and unfractured specimens and may contribute to failure.

Figures III-14(a) and III-14(b) present micrographs of the slow zone and fast zone, respectively, of Hi-Atomic at a magnification of 500X tested at 37°C. Figures III-15(a) and III-15(b) present corresponding micrographs of Hi-Atomic tested at 22°C. These higher magnification micrographs are presented to clearly illustrate features of typical specimen failure with regard to morphological characteristics of crack propagation in various zones.

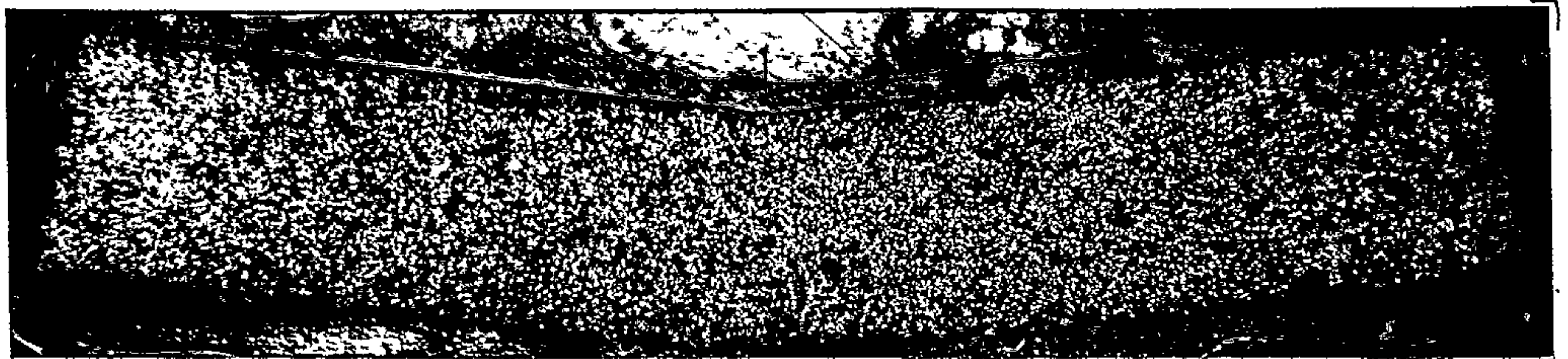


Figure III-13(a): Unfractured specimen of Dispersalloy tested at 22°C at a magnification of 10X.

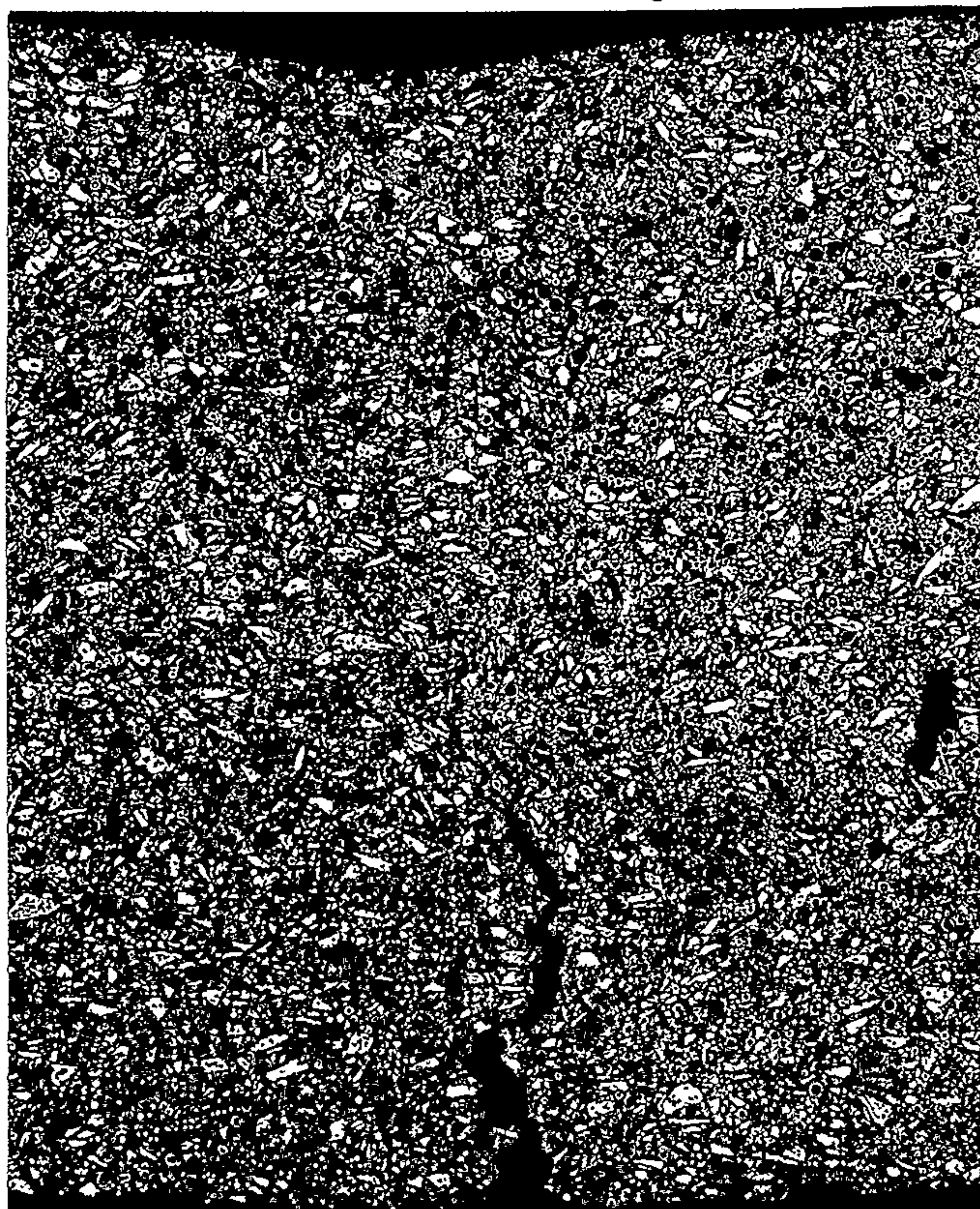


Figure III-13(b): Specimen shown in Figure III-13(a), at a magnification of 50X.

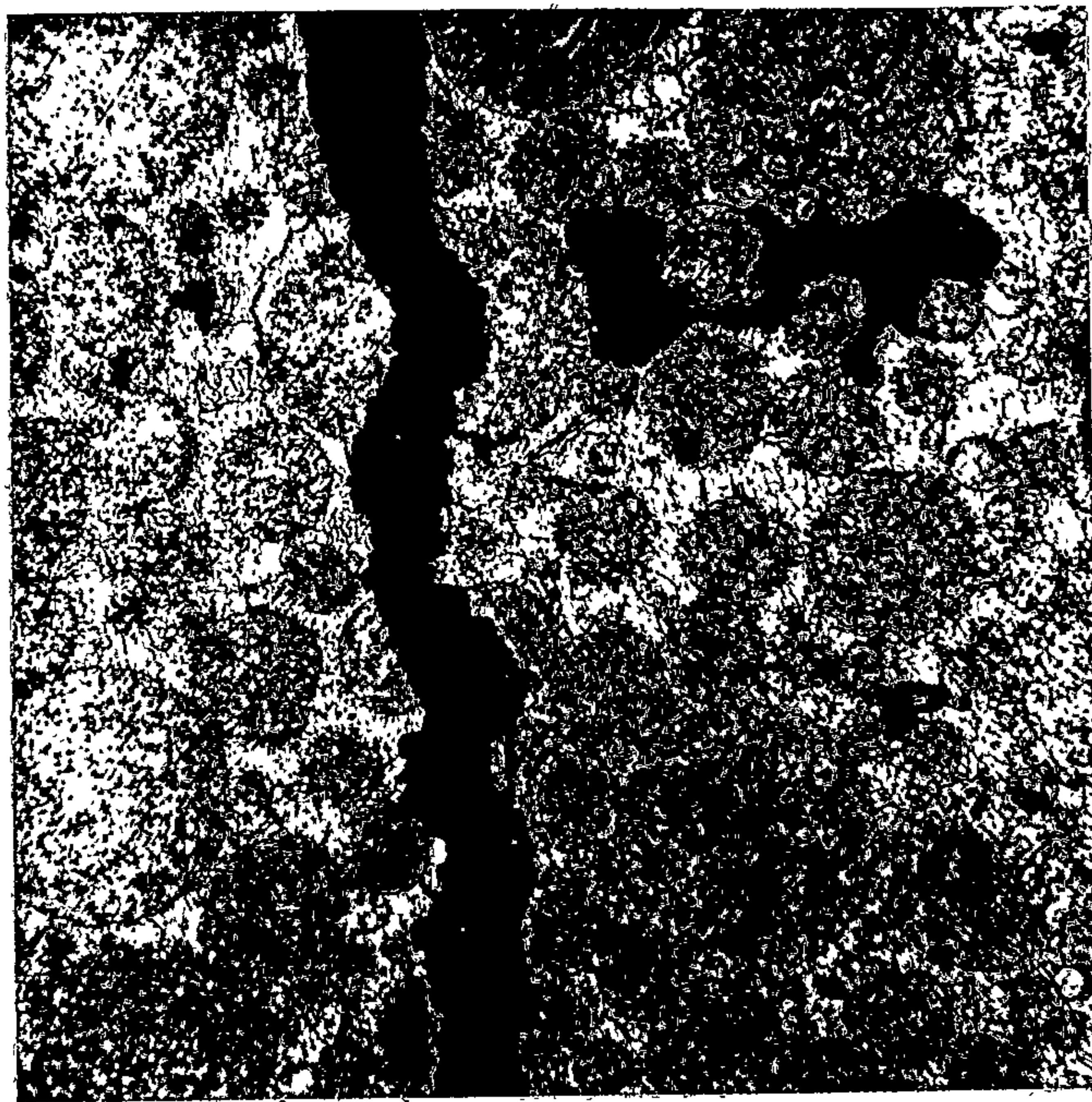


Figure III-14(b): 500X magnification of the fast zone of
a Hi-Atomic specimen tested at 37°C.

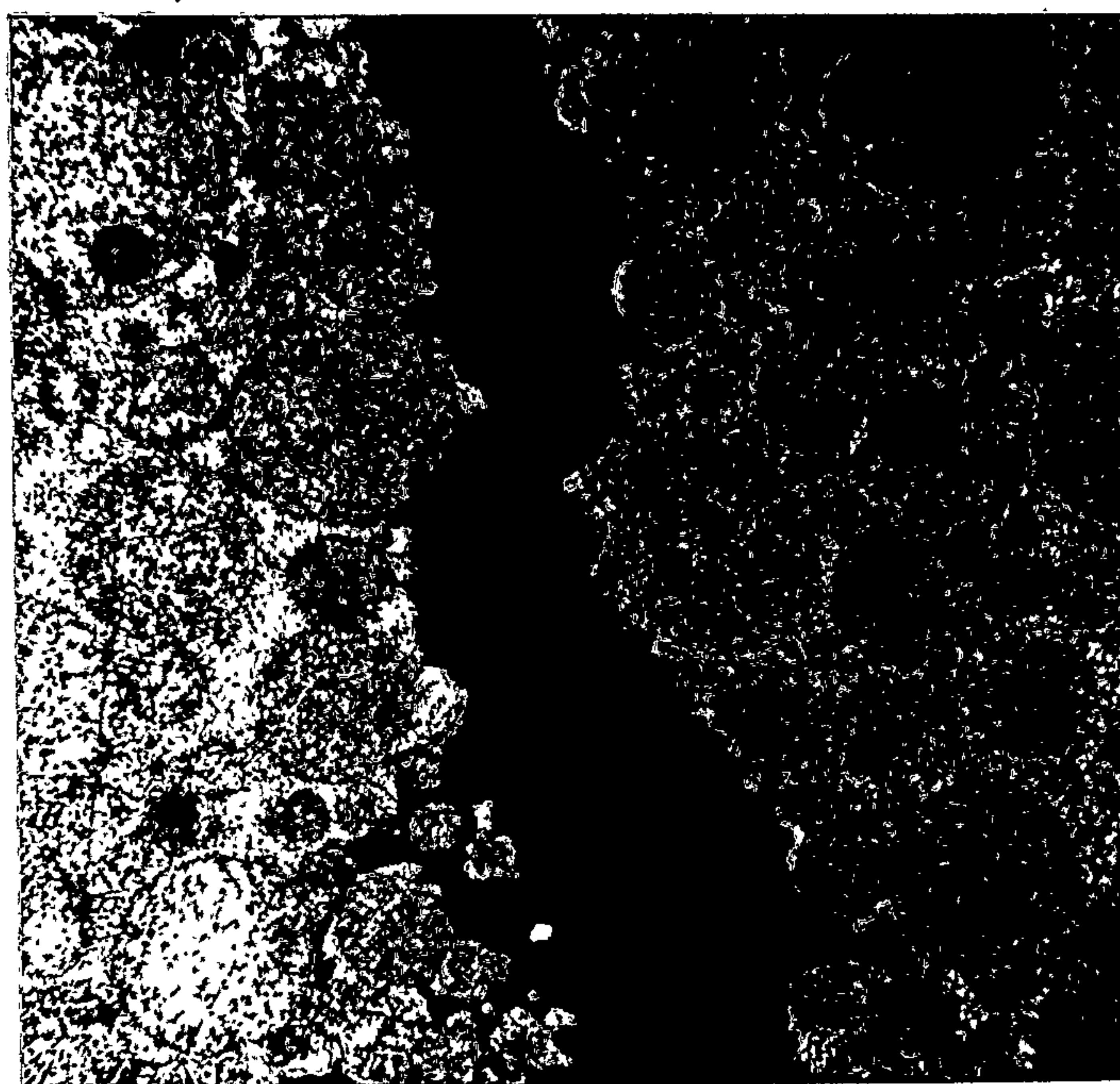


Figure III-14(a): 500X magnification of the slow zone of
a Hi-Atomic specimen tested at 37°C.

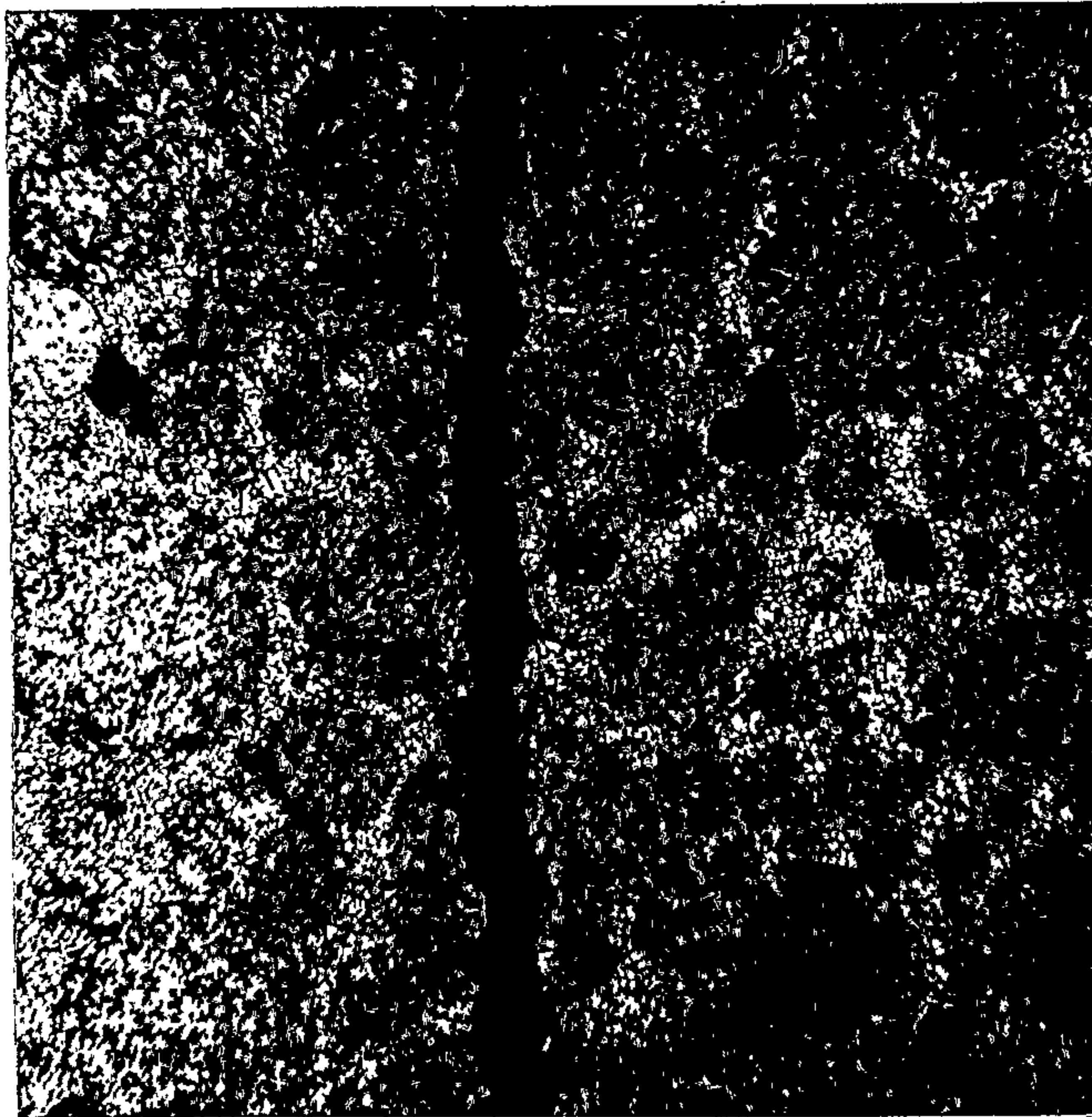


Figure III-15(b): 500X magnification of the fast zone of
a Hi-Atomic specimen tested at 22°C.



Figure III-15(a): 500X magnification of the slow zone of
a Hi-Atomic specimen tested at 22°C.

The features of irregular 'around-particle' slow zone failure are obvious in Figure III-14(a). Here, the progress of the crack has followed a path frequently around spherical particles. Figure III-14(b) illustrates the uniform 'through-particle' mode of fast zone failure. Unobstructed progress of the crack through the specimen can be easily observed. Figure III-15(a) presents the slow zone in a specimen tested at 22°C. A more uniform, thinner fast zone is seen at 22°C but the failure can still be seen to progress through particles predominantly. However, slow zone failure at 22°C, as shown in Figure III-15(b), appears much more irregular and wide than at 37°C but the 'around-particle' mode persists.

Study of 500X magnifications of all types of amalgams examined confirmed that no difference occurred in the mode of fractures for slow and fast zones at 22°C and 37°C.

Slow zones for all types of amalgams were characterized by a high proportion of around particle failure and fast zones by a high proportion of through particle failure, at both temperatures.

The same basic characteristics as described by Giblin (1979) for 3mm thick specimens at 22°C have been observed for the thinner 2mm specimens at both 22°C and 37°C.

Application of Additional Alloys

In order to evaluate the model and the trends and conclusions drawn, an additional group of ten amalgam alloys was selected and tested. The alloys chosen encompassed a wide range of formulations; specific details of these alloys are given in Chapter II, 'Experimental Procedures', Table II-3.

Mechanical Properties

In Table III-11 the values of modulus of rupture (transverse strength) of the applied alloys are presented. Figure III-16 represents this data graphically. Also found below Figure III-16 is a glossary explaining the code used in all graphs pertaining to results of applied alloys. It was decided that for the determination of modulus of rupture values for the additional alloys, all specimens would be prepared with final condensation applied by hand. Results reflect the mean and standard deviation of three specimens.

From Table III-11 it can be noted that the alloys selected reflect a range of transverse strengths with the lowest strength being that of Spheraloy and the highest, that of G. & G. Superfine. Generally, the transverse strengths derived fall into the range of transverse strengths noted for amalgams in the study. Significantly, the strength of the Single Melt High Copper Ternary alloys (ANA 2000 and Permite C) ^{BASIS OF} did not reach the high value expected for such alloys, and seen in the study exhibited by Tytin. Giblin (1979) noted a similar low value for Sybraloy¹, a Single Melt High Copper alloy.

Table III-12 presents 3-hour and 72-hour creep values determined for the applied alloys. These values are presented graphically in Figure III-17. It can be seen from Table III-12 that 3-hour creep values range from 3.0% for F400 (an Ag₃Sn-type alloy) to 0.1% for ANA 2000 (a Single Melt High Copper Ternary alloy). In comparing the creep values of applied alloys to those values of alloys used in the study, several similarities between

1 Kerr-Sybron. Romulus, Michigan. U.S.A.

Table III-11: Modulus of rupture of alloys applied to the model.

Alloy	Modulus of Rupture (MPa)
F400	121 (27.6)*
Ultrafine	112 (0.8)
Caulk Spherical	121 (8.7)
Spheraloy	90 (5.0)
New True Dentalloy	111 (1.4)
Luna Atomic	113 (8.8)
G. & G. Superfine	141 (8.4)
Luxalloy Non-Gamma 2	115 (4.4)
Permite C	128 (7.5)
ANA 2000	139 (1.9)

* Standard Deviation in brackets

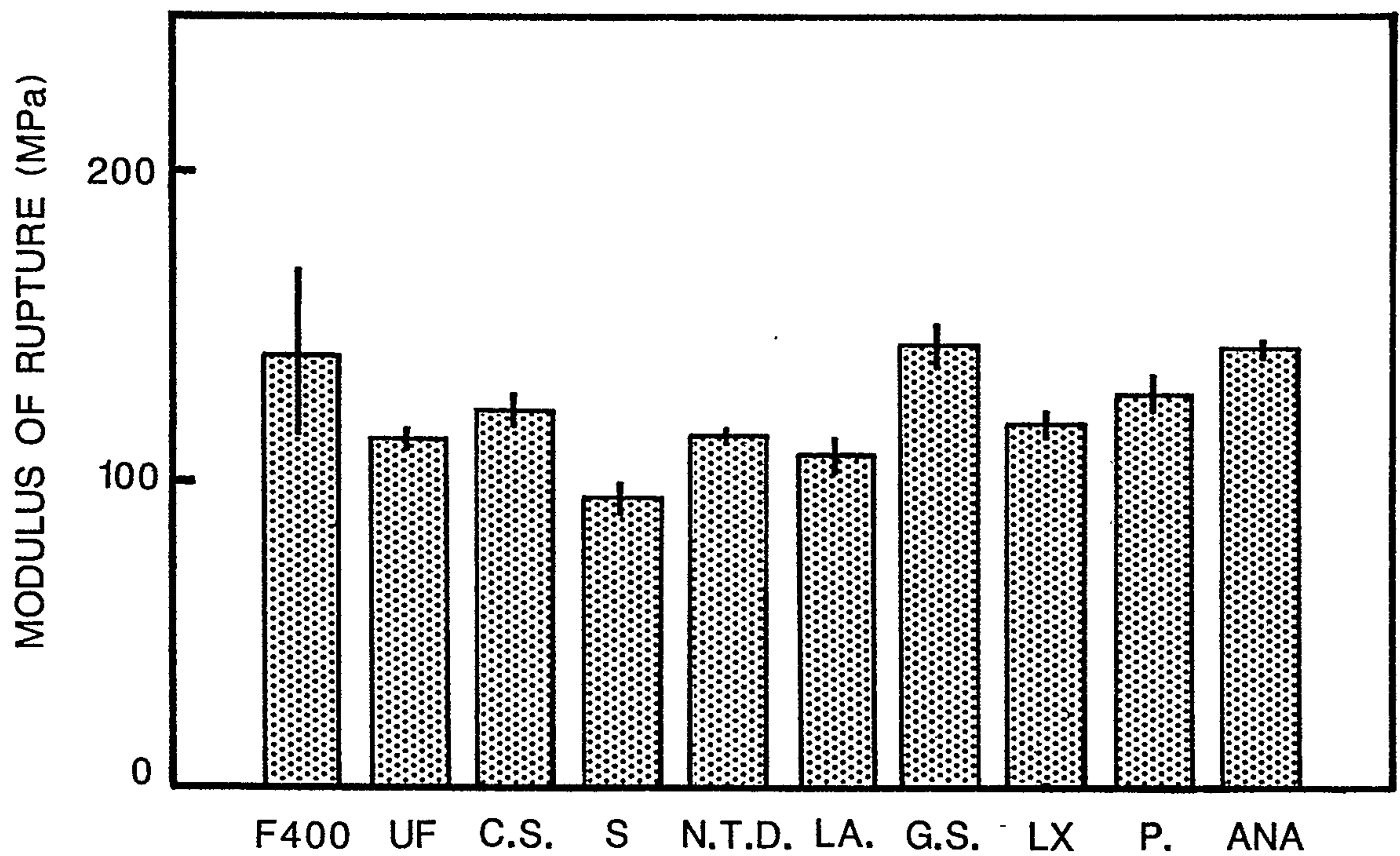


Figure III-16: Modulus of rupture of alloys applied to the model.

* Glossary :

F400	-	F400
UF	-	Ultrafine
C.S.	-	Caulk Spherical
S	-	Spheraloy
N.T.D.	-	New True Dentalloy
LA	-	Luna Atomic
G.S.	-	G. & G. Superfine
LX	-	Luxalloy Non-Gamma 2
P	-	Permite C
ANA	-	ANA 2000

Table III-12: Creep values of applied alloys.

Alloy	Creep (%)	
	3-hour	72-hour
F400	3.2	40
Ultrafine	1.9	28
Caulk Spherical	2.0	30
Spheraloy	1.8	30
New True Dentalloy	1.2	18
Luna Atomic	0.8	25
G. & G. Superfine	1.5	25
Luxalloy Non-Gamma 2	1.0	12
Permite C	0.2	2
ANA 2000	0.1	0.3

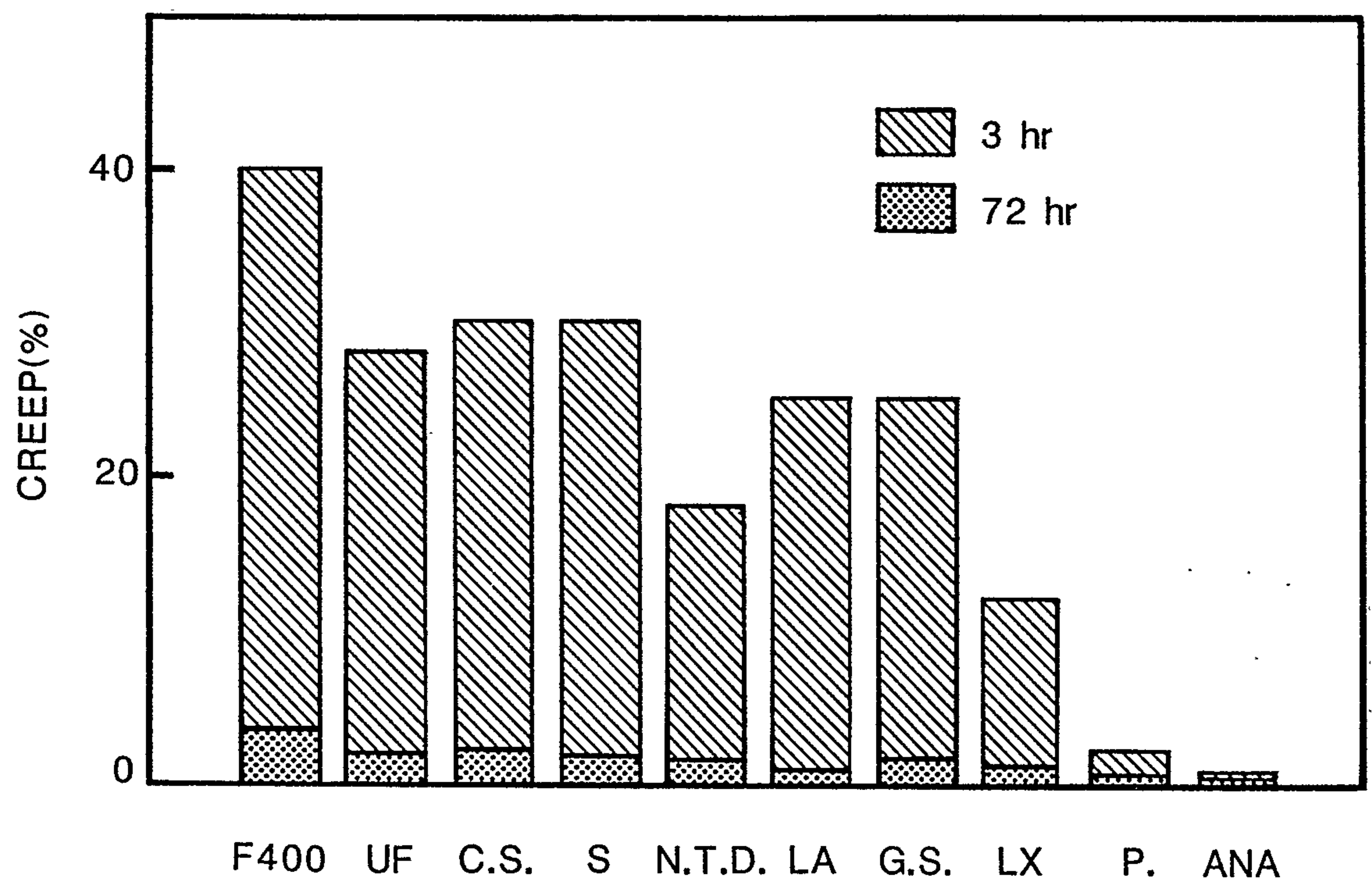
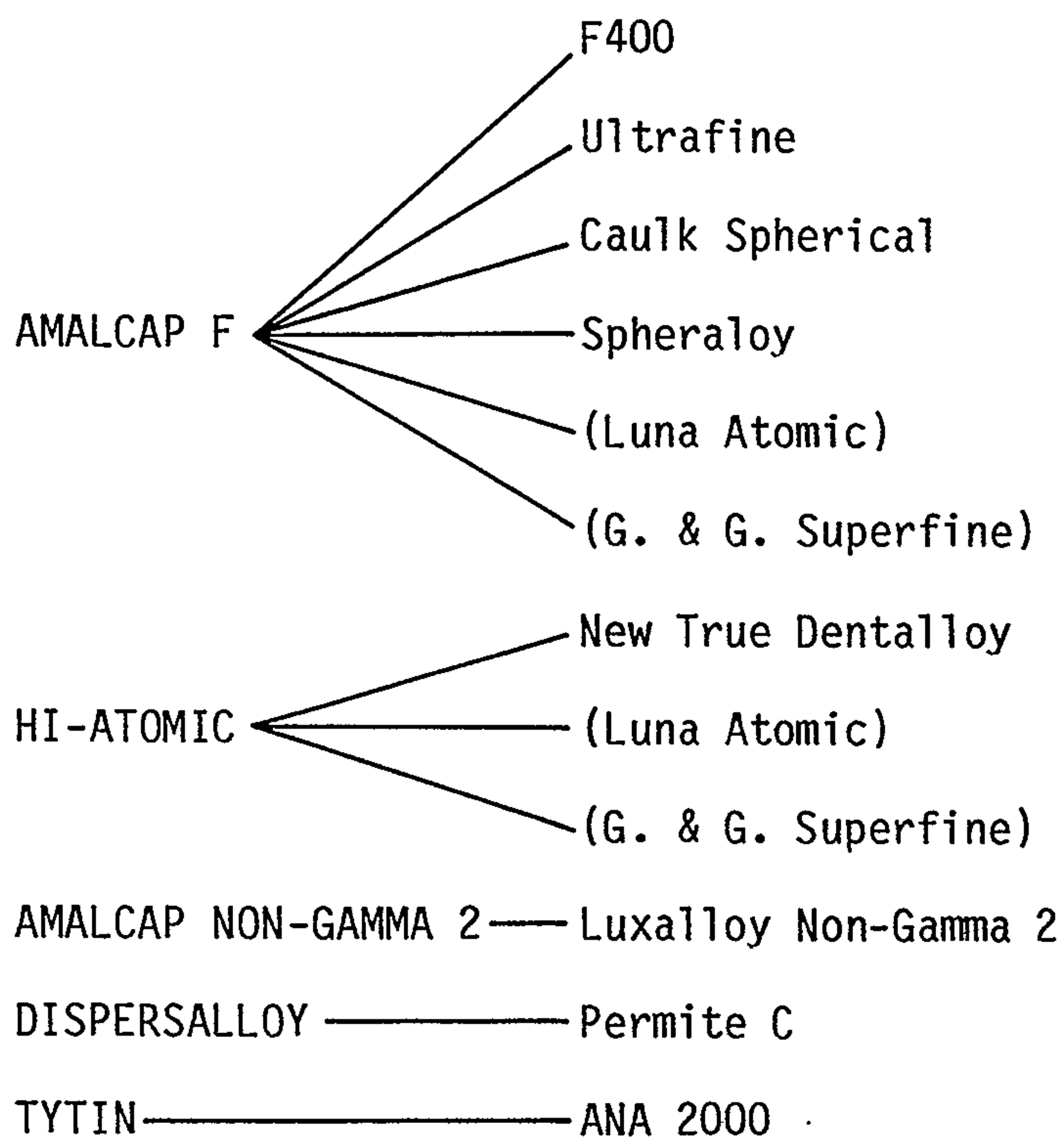


Figure III-17: Creep values of applied alloys.

specific alloys can be noted within the broad formulations. For example, Amalcap F, with 3-hour and 72-hour creep values of 3.1% and 40.0% respectively encompasses the alloys F400, Ultrafine, Caulk Spherical and Spheraloy closely. Hi-Atomic, with creep of 1.2% and 18.0% for 3-hour and 72-hour values respectively mirrors values derived for New True Dentalloy. Amalcap Non-Gamma 2 is 'similar' to Luxalloy Non-Gamma 2, Dispersalloy relates well to Permitem C, and Tytin exhibits creep rates similar to ANA 2000. Less similar to the tested alloys in terms of creep are the alloys Luna Atomic and G. & G. Superfine which have 3-hour creep similar to Hi-Atomic and Amalcap Non-Gamma 2 but 72-hour values which rise sharply to be similar to Amalcap F and Hi-Atomic. The performance of applied alloys in testing will be directly compared to the performance of their closely related alloys in the main study. The particular coupling of the applied alloys to the test alloys in terms of creep can thus be shown:



Fracture Times

Table III-13 presents the fracture times of the additional alloys subjected to a static sub-fracture load at 37°C, related to method of condensation. These results are presented graphically in Figure III-18. Specimens which did not fracture within the test period are marked 'N.F.'

In comparing fracture times of applied alloys to the results in the main study (Table III-4) it can be seen that the performance achieved by alloy formulation groups have been proven by the applied alloys with few exceptions. Fracture times of Ag₃Sn-type alloys, in the range of 3 to 14 hours set by Amalcap F and Hi-Atomic, have been affirmed by the performance of most applied Ag₃Sn-type alloys. Dispersion Modified alloys yielded results similar to those of Amalcap Non-Gamma 2 and Dispersalloy.

It can be noted that longest times to fracture were achieved by all specimens machine condensed with a force of 2250N. The alloy ANA 2000 yielded specimens which did not fracture, similar to Tytin. Similarly machine condensed (2250N) specimens of Permite C (a Single Melt High Copper alloy) recorded no fracture. High machine condensed specimens of Luxalloy Non-Gamma 2 improved on the performance of Amalcap Non-Gamma 2 and also did not fracture. An unexpected superior performance of machine condensed (450N) specimens of Luxalloy Non-Gamma 2 and Permite C, in excess of 200 hours, was observed.

In the graphic representation of fracture times attained by the applied alloys shown in Figure III-18, the inverse relationship of fracture times to creep (particularly 72-hour creep) found in the main study was again observed with the applied alloys.

Table III-13: Fracture time of specimens of additional alloys subjected to a static sub-fracture transverse load (67MPa) at 37°C related to condensation method.

Alloy	Fracture Time (hr.)									
	F400	UF	C.S.	S	N.T.D.	LA	G.S.	LX	P	ANA
Method of Condensation:										
Hand	6.2	8.8	4.6	0.7	2.6	8.2	17.2	70.8	N.F.*	N.F.
Machine 450N	5.9	8.2	4.9	1.1	4.7	1.9	18.5	202.4	270.5	N.F.
Machine 2250N	14.5	28.9	8.5	1.3	6.6	17.2	36.6	N.F.	N.F.	N.F.

* No fracture within test period.

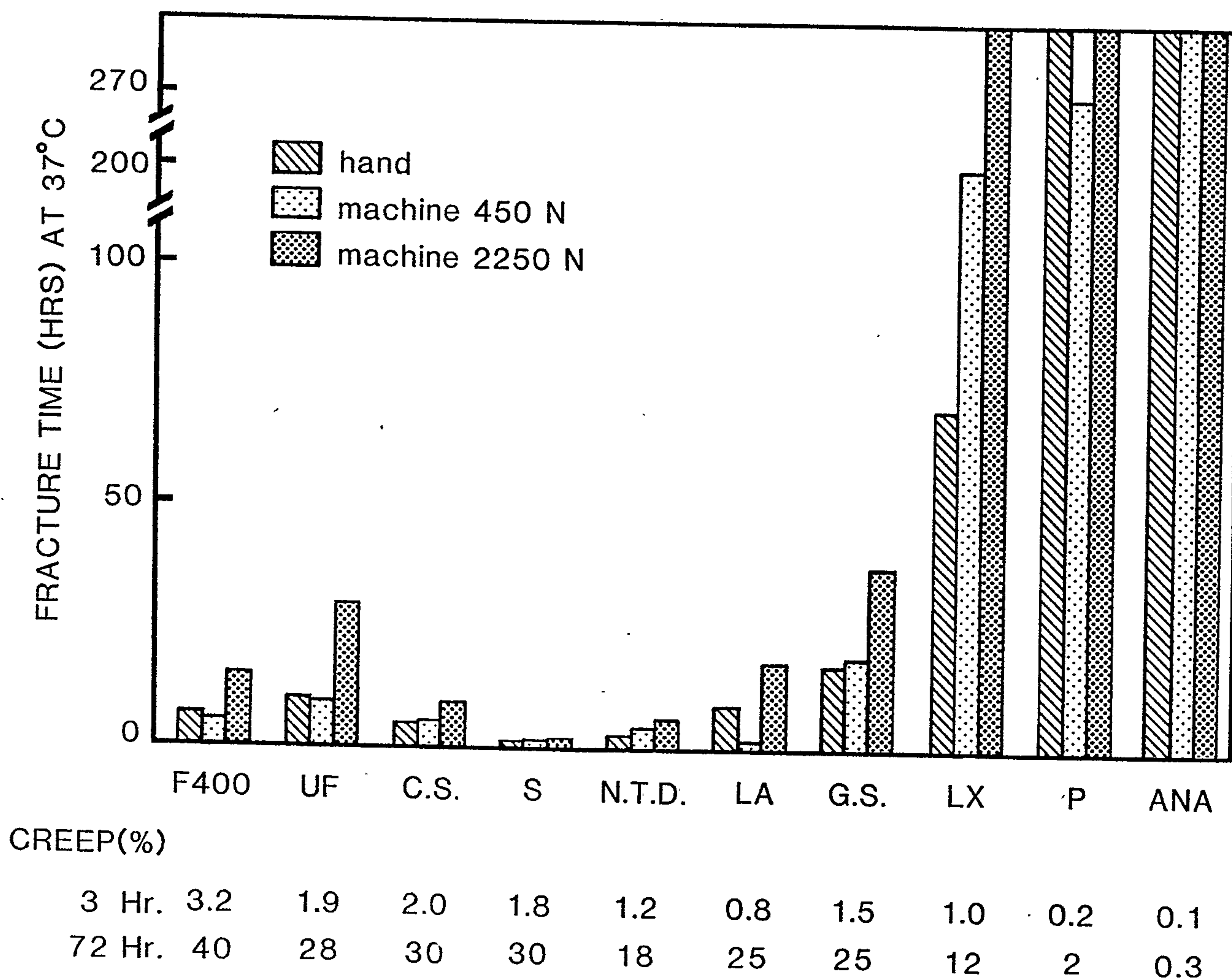


Figure III-18: Fracture time of specimens of additional alloys subjected to a static sub-fracture transverse load (67MPa) at 37°C, related to method of condensation. Values for 3-hour and 72-hour creep shown below the graph.

Bending Characteristics

Bend of specimens of additional alloys subjected to a static sub-fracture transverse load was determined using the system of ranking devised by Giblin and Wing (1979) and already applied to specimens in the main study. Table III-14 presents the bend results of each specimen according to method of condensation. In addition, included in the table is an indication of whether or not fracture took place and a ranking of bend prior to fracture or at the end of the test period.

From Table III-14, several observations can be made:

(i) Bend characteristics of all specimens fall into the two main groups noted in the main study; one group containing specimens which bend slightly but do not fracture and a second group of specimens which bend appreciably and ultimately fracture. The 'critical' bend ranking of '5' described in the main study was again observed. The poor performance of Spheralloy specimens noted in short fracture times was achieved with little prior bend. Alloys of the Ag₃Sn-type formulations exhibited bend characteristics comparable to those of Amalcap F and Hi-Atomic. A most obvious example of the comparison was the bend achieved by New True Dentalloy which duplicated results of Hi-Atomic. Related amalgams Amalcap Non-Gamma 2 and Luxalloy Non-Gamma 2, representing the Dispersion Modified formulation, likewise exhibited comparable bend. Maximum bend prior to fracture was exhibited uniformly by three amalgams: F400, Ultrafine and G. & G. Superfine which suggested close parity to Amalcap F. This parity is also seen in their respective creep values, particularly the 72-hour determination of this property. ANA 2000, representing a Single Melt High Copper amalgam, resisted fracture and exhibited

Table III-14: Bend characteristics of specimens of additional alloys subjected to a static sub-fracture transverse load (67MPa), related to condensation method.

Alloy	Bend characteristics.									
	F400	UF	C.S.	S	N.T.D.	LA	G.S.	LX	P	ANA
Method of Condensation:										
Hand	10F*	10F	5F	2F	5F	5F	10F	10F	4NF†	2NF
Machine 450N	10F	10F	5F	2F	5F	5F	10F	10F	6F	1NF
Machine 2250N	10F	10F	5F	2F	6F	6F	10F	3NF	1NF	1NF.

* Bend prior to fracture.

† No fracture, bend at end of test period limit.

very little bend, thereby echoing the performance of Tytin. The bend achieved by the respective alloys Permite C and Dispersalloy more closely reflects the effect of increased available time for bend than the effect of intrinsic amalgam characteristics. In comparing hand condensed specimens Dispersalloy in an average fracture time of 130 hours, achieved bends of '8'; Permite C specimens resisted fracture. Machine condensed (450N) specimens of Permite C achieved bends of '6' in 270 hours whereas Dispersalloy specimens took, on average, only 48 hours to achieve bends of '8'.

(ii) Bends achieved by specimens prepared by the three methods of condensation were not significantly different within the specific amalgam groups. This finding supports that noted in the main study. An exception however was the performance of machine condensed (2250N) specimens of Luxalloy Non-Gamma 2 which attained little bend and resisted fracture. This effect, though not as significant, was also seen in Amalcap Non-Gamma 2.

(iii) In applying modulus of rupture values attained by the applied alloys (Table III-11) and fracture times (Table III-13) to bend characteristics it can be seen that the superiority of Single Melt High Copper amalgams in resisting bend noted in the study has been proved by the performance of the applied alloy ANA 2000. However, the lack of direct correlation between modulus of rupture and characteristics of bend and fracture seen in the main study has been confirmed by the applied alloys. An example of this is a comparison of alloys Ultrafine and Luna Atomic which exhibited comparable transverse strengths and fracture times but significantly different bend characteristics. Similarly, the transverse strengths attained by G. & G. Superfine and ANA 2000

are comparable but both fracture times and bend characteristics differ significantly.

(iv) When bend characteristics of applied alloys are compared to creep values (Table III-12) it can be noted that amalgams exhibiting lowest creep (Permite C, ANA 2000) also exhibited least bend and optimal resistance to fracture. If we compare bend characteristics of other applied alloys to their 3-hour creep we find few obvious trends; however, if 72-hour creep is considered, several trends emerge. For example, alloys of highest and comparable 72-hour creep such as F400, Ultrafine and G. & G. Superfine also suffer greatest bend. Similarly, but with less strict correlation, Caulk Spherical, New True Dentalloy and Luna Atomic show comparable 72-hour creep, fracture times and bend characteristics. Thus, the relationship between bend characteristics and 72-hour creep suggested by the study has been supported by the performance of the applied alloys.

CHAPTER IV

CONCLUSIONS

This investigation aimed to compare the behaviour of amalgam subjected to a direct continuous or intermittent sub-modulus of rupture load at 37°C with that at 22°C and sought to relate this behaviour to mechanical properties of the amalgams. A group of five amalgam alloys of three basic formulations was tested, and a subsequent group of ten additional alloys was applied to the model. Specimen behaviour was examined in terms of bend and fracture characteristics, times to fracture and microstructural features of crack morphology. Mechanical properties investigated were modulus of rupture (transverse strength), 3-hour creep and 72-hour creep. Microstructural examination was carried out at magnifications of 10X, 50X and 500X. Specimens prepared from the test alloys were condensed in one of three methods and the effect of this on results was examined.

From the various trends which emerged in the study, several conclusions may be made with respect to the relevance of mechanical tests to predicting fracture behaviour and the relative ability of amalgam formulations to resist deformation. In view of these conclusions, further information may be added to that in other studies which have similarly sought to explain factors involved in isthmus and marginal fracture of amalgam restorations.

Modulus of Rupture

Values determined for modulus of rupture (transverse strength) did not differentiate between the amalgam formulations. The High Copper amalgams and in particular the Single Melt High

Copper amalgams with the exception of Tytin do not possess significantly higher moduli of rupture than other amalgams. This characteristic in the transverse mode differs from the very high compressive and tensile strengths demonstrated by the Single Melt High Copper amalgams, in studies by numerous workers, frequently quoted and personally observed in studies at the University of Sydney, Department of Operative Dentistry. With the exception of Tytin and Spheraloy the transverse strengths of the fifteen alloys tested fall between 110 and 160 MPa. Tytin is an exception being significantly higher than the range and Spheraloy is significantly lower than the range. Sybraloy¹ falls within the range (Giblin 1979).

The three methods of condensation used in specimen preparation of the test alloys were observed to not significantly affect modulus of rupture values or fracture times although a tendency for hand condensation values to be similar to high machine load condensation values - both higher than low machine load condensation values - was noted. This may be explained in terms of efficiency of excess mercury removal during condensation which was similar in hand and high machine load condensation but particularly poor in low machine load condensation. Few results for modulus of rupture values are quoted in the literature but the values derived in this study agree with those quoted by Giblin (1979). The differing geometry of the specimen used in the two studies, had no significant effect on results for modulus of rupture confirming the fact that the modulus of rupture formula theoretically could take account of specimen shape.

1 Kerr-Sybron. Romulus, Michigan U.S.A.

Sub-Modulus of Rupture Loading

The ability to remove the "mercury rich surface layer" in the redesigned specimen mould used in the present study did not produce significant improvements either in hand condensed or machine condensed specimens. Almost certainly the explanation for this is that in transverse testing the highest tensile stress areas will be at the inferior surface of the amalgam opposite the point of loading and largely independent of the quality of the uppermost layers of the specimen.

Fracture times of specimens tested at 37°C were significantly shorter than times at 22°C for the test alloys. This finding was not unexpected since, like creep, sub-modulus of rupture fracture susceptibility is a rheologic property and may be expected to be temperature dependent. Single Melt High Copper amalgams were most resistant to fracture at both temperatures, not failing in the test limit. Ag₃Sn-type amalgams failed in the shortest times. The use of 37°C for testing as opposed to 22°C reduced the fracture time of Ag₃Sn-type amalgams by a factor of approximately ten. For the Dispersion Modified amalgams the effect was to allow bending to occur in the non-fracturing (at 22°C) Dispersalloy and to reduce the time of fracture for Amalcap Non-Gamma 2 by a factor of approximately three.

Leading to fracture, amalgams undergo bend. The degree of bend where fracture occurred was similar at 22°C and 37°C although a great difference in time was required to achieve the same bend. A notable exception to this was Dispersalloy where at the end of the arbitrary test time at 22°C a lesser degree bend without fracture had occurred. At the time of fracture a similar degree of bend is likely to have been achieved even for alloys with differing

creep values. An exception was G.C.'s Hi Atomic which fractured in a time longer than the other Ag₃Sn-type amalgam, Amalcap F. The degree of bend is very dependent on creep as well as time. Hi Atomic with a lower creep than Amalcap F but containing gamma 2 as discrete clumps, fractures in a statistically similar time but with less bend than Amalcap F. Amalcap Non-Gamma 2 which fractures in a longer time than the Ag₃Sn-type amalgams and with a lower creep achieves a similar degree of bend.

At a constant sub-fracture stress, amalgam may continue to deform for an indefinite time and with sufficient time will eventually fracture. Very low creep amalgams require times exceeding several months before fracture, even at a temperature of 37°C. Some physical change occurs and it may be assumed that eventually even lowest creep Single Melt High Copper amalgams would fracture with sub-fracture loading. This deformation like creep, is highly temperature dependent. It is evident that the two most important factors contributing to fracture are the time and the temperature at which the load is applied.

In comparing the creep values, of all amalgams tested, to bend and fracture at 37°C, many interesting observations may be made. Bend achieved more closely parallels 72-hour creep values than 3-hour creep values with the noted exception of Dispersalloy and the anomalous behaviour of Spheralloy. Amalgams having 72-hour creep of less than 10% resisted fracture - this identification of a "critical" creep value cannot be made from 3-hour creep values. Amalgams having similar 72-hour creep can be expected to achieve comparable bend in comparable times - for example Amalcap F and F400, New True Dentalloy and Hi-Atomic, and Amalcap Non-Gamma 2 and Luxalloy Non-Gamma 2. Spheralloy was distinguished in its extremely

low fracture time and related low bend achieved compared to Caulk Spherical which exhibited better results despite having comparable creep to Spheralloy. Spheralloy exhibited a significantly lower modulus of rupture than all other alloys and as a result the load applied represented a significantly higher percentage of the modulus of rupture of approximately 65%. In the case of Dispersalloy, however, its performance may be explained in light of the metallurgical and morphological characteristics this unique amalgam represents. In this study, Dispersalloy exhibited unique behaviour : at 22°C specimens underwent little bend and resisted fracture, at 37°C specimens underwent appreciable bend and did fracture. No other alloy exhibited such behaviour. Such behaviour may be explained by considering solid state deformation. It is known that increased solid state deformation takes place as the temperature of testing approaches solidus temperature. The solidus temperature of gamma 2 - the most creep-prone phase of Ag₃Sn-type alloys - is 96°C. 22°C testing and 37°C testing represent testing within approximately 60°C of solidus from this temperature and appreciable deformation might be expected. In Dispersalloy, gamma 1 solidus temperature is 122°C; 22°C testing is sufficiently remote from this temperature to prevent deformation taking place but 37°C is closer to this solidus temperature and this results in perceptible solid state deformation being exhibited.

The results for fracture time and bend give a possible clue to a fundamental mechanism for the rheology of dental amalgam. As previously pointed out in comparing fracture times for 37°C and 22°C different factors influence times to fracture at 22°C and 37°C for Ag₃Sn, gamma 2 containing alloys and Dispersion Modified alloys. It is likely that the elevation of temperature from 22°C

to 37°C i.e. 15°C closer to the gamma 2 solidus temperature of 96°C is sufficient to produce the very rapid solid state deformation of amalgams containing this phase - hence a reduced time to fracture of one-tenth at 37°C of that at 22°C. For Amalcap Non-Gamma 2, which does not contain the gamma 2 phase, the reduction in time to fracture following bend is likely to be related to the gamma 1 phase with a higher solidus temperature, and a reduced time to one third at 37°C of that at 22°C. Similar dependence on the gamma 1 solidus temperature is applicable to both Dispersalloy and Amalcap Non-Gamma 2. No linear relation can be established but a possible mechanism for non-gamma 2-containing alloys emerges.

Modulus of rupture values did not correlate well with bend behaviour or fracture times. Thus these values cannot be confidently used to predict likely clinical behaviour of the amalgams, assuming that clinical fracture does not occur as a result of application of stresses greater than the strength of the amalgam but as a result of repeated low load applications resulting in bend and fracture.

Cyclic or intermittent testing at 37°C was carried out using specimens prepared from amalgams of highest creep. No significant difference in fracture times or bend achieved was observed in comparing cyclic and static testing. Under intra-oral conditions, occlusal loading is fluctuating rather than continuous and causes "dynamic creep". A close correlation has been reported between results of dynamic and static creep testing in current literature which has led to universal acceptance of the simpler static creep test. Similarly, the close parity of static and cyclic sub-fracture loading suggests no real advantage in cyclic testing.

There is little doubt that the repeated application of low loads in compressive or transverse modes causes permanent plastic deformation which is cumulative.

Additional alloys applied to the model confirmed trends exhibited by the test alloys. Since the model confirmed the close correlation of cyclic and static testing, the uniform extended fracture times at 22°C and the greater bend at 22°C for all alloys, testing of the additional alloys was restricted to the determination of modulus of rupture values, creep values, fracture times at 37°C and bend characteristics at 37°C. Modulus of rupture values fell into the range seen in the test alloys. Creep values, like those of the tested alloys, allow differentiation between alloy formulations particularly the 72-hour creep values. Since the times to fracture of the applied alloys reflect single tested specimens, only an indication of a tendency to follow trends made by the tested alloys may be suggested. Alloys of respective formulations represented in the study have followed general trends of bend characteristics made in the study. Specimens condensed by hand and by low machine load exhibited comparable fracture times for all amalgams with the exception of Luxalloy Non-Gamma 2 and Permite C which both showed superior times for machine condensed specimens. For most alloys, superior times to fracture were exhibited by high load machine condensed specimens which probably reflects efficient excess mercury removal.

In consideration of the microstructural observations of amalgam fractures this study has supported the fracture mechanisms for clinical amalgams as postulated by Giblin and Wing (1983). Actual fracture of amalgam under low loads is a gradual process initiated by the formation of a crack usually from an imperfection

on the tensile surface. Under the influence of repeated or constant stressing, the crack spreads progressively through the width of the specimen. Crack propagation continues through the tensile half until eventually the stress in the remaining section of sound material or compressive half rises to a point where ordinary breaking stress has been exceeded and almost instantaneous fracture occurs. Giblin and Wing noted distinct features of slow zone or "around" particle failure and fast zone or "through" particle failure. This study has largely confirmed those features although respective lengths of these zones have differed slightly. These same features have been seen in specimens tested at both 37°C and 22°C. If different features at the two temperatures had been observed, this finding would have pointed to the need for 37°C testing of all mechanical properties.

The present study has pointed to the importance of inherent characteristics of amalgam particles, efficiency of condensation in reducing void content, relative amounts of creep-prone phases present, temperature of testing and loading mode and magnitude of load in crack initiation and propagation in a laboratory situation. It presents a mechanism of failure. In the oral environment failure will be effected by moisture and corrosive conditions and if corrosion takes place while the amalgam is repeatedly stressed, failure may occur sooner. The same mechanism seen in the study may be enhanced by the hostile oral environment. Resistance to fatigue stresses is reduced. The presence of "stress raisers" such as surface irregularities or sharp internal angles which increase local stresses resulting from repeated loads also greatly reduce the resistance to fatigue. In clinical conditions no recovery occurs during unloaded periods and so damage is

cumulative. All restorations which undergo occlusal loading are subjected to cyclic bending fatigue stressing which fluctuates between zero and maximum. The ability of a restoration to bend depends on its supporting structures to a significant degree whether these structures are a lining or base material, dentine or residual or recurrent caries. Soft linings or caries may allow bending and the established mechanism to apply.

This study has utilized a specimen size, loading format and temperature of testing which are more closely related to clinical amalgams. At a constant sub-fracture stress, amalgam can continue to deform for an indefinite time, and will eventually fracture. This deformation, is highly temperature dependent and the most important factors which contribute to fracture are time, temperature and creep. The close relationship found between extended creep values and sub-modulus of rupture loading failure suggests no justification for the inclusion of a low load test or a rapid transverse strength test into specifications because this information is given by the 72-hour creep test. In addition, fracture toughness tests as reviewed using 80% loading probably do not directly relate to clinical conditions and have a cumbersome and complex test format using an artificial means of crack initiation which must give rise to artificial failure propagation.

The present study has pointed to the importance of the use of 37°C testing in low load stressing of dental amalgams. The study has confirmed the nature of failure of amalgam related to its rheologic properties at the temperature at which amalgam functions clinically. The use of 37°C significantly reduced the times of fracture where this occurs, from in excess of one day to several hours. It is an excellent test because it generally establishes a

mechanism of failure but is cumbersome because the test must be extended for several weeks in the case of High Copper amalgams to determine their long term behaviour.

Having established the close relationship between the behaviour of a wide range of alloys in the sub-modulus of rupture beam test and extended creep a firm conclusion is that the simplest and most relevant test which reflects the mechanism of isthmus and marginal fracture of restorations may be an extended 72-hour static creep test.

Suggestions for possible future research may include:

- (1) a consideration of dynamic testing of specimens at 37°C.
- (2) an examination of the fracture toughness of amalgam alloys.
- (3) the testing of repaired amalgam beam specimens in a sub-fracture transverse mode.
- (4) testing amalgam beam specimens supported by base materials like zinc-oxide eugenol or glass ionomer cements.

APPENDIX

APPENDIX

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APPENDIX

The following summary tables have been included to provide a clear representation of the findings of the study as set out in conclusions drawn in Chapter IV for all alloys tested including applied alloys. In addition, a news article reproduced with permission from the Sydney Morning Herald from the edition of that paper of June 6, 1988 is also included.

Table A-1 presents the relationship between modulus of rupture and fracture time of amalgams subjected to a static sub-modulus of rupture load at 37°C. Amalgam alloys have been ranked in increasing order of modulus of rupture values irrespective of basic alloy composition or particle characteristics. Modulus of rupture values have also been segregated to ranges where the upper-most range encompasses 90-125 MPa, the second range 125-150 MPa, the third range 150-156 MPa and the final range above 156 MPa. From Table A-1, no direct correlation between these ranges of modulus of rupture values and fracture times can be seen.

Table A-2 presents the relationship between fracture times and 3-hour creep values. Amalgams are presented in order of increasing creep values determined, which does not provide any correlation to the order of modulus of rupture values presented in Table A-1. An inverse relationship between fracture times and 3-hour creep may be seen to be emerging. Amalgams of 3-hour creep below 0.4% are seen to best resist sub-modulus of rupture loading. Creep values of 1% and above are related to short fracture times usually within 24 hours. The correlation of 3-hour creep to fracture times is a closer and more faithful correlation than that of modulus of rupture values and fracture times.

Table A-1 : Relationship between modulus of rupture and fracture time under sub-modulus of rupture loading, for all alloys.

Alloy	Modulus of Rupture (MPa)	Fracture Time (hr.)
Spheraloy	90	0.7
Ultrafine	111	2.6
New True Dentalloy	112	8.8
Luna Atomic	113	8.2
Amalcap Non-Gamma 2	113	20.3
Luxalloy Non-Gamma 2	115	70.8
F400	121	6.2
Caulk Spherical	121	4.6
Permite C	128	N.F.*
Hi-Atomic	135	7.6
ANA 2000	139	N.F.
G. & G. Superfine	141	17.2
Dispersalloy	150	130.0
Amalcap F	156	4.7
Tytin	219	N.F.

* No fracture within test period limit.

Table A-2 : Relationship between 3-hour creep values and fracture time under sub-modulus of rupture loading, for all alloys.

Alloy	Creep (%)	Fracture Time (hr.)
ANA 2000	0.1	N.F.*
Tytin	0.1	N.F.
Permite C	0.2	N.F.
Dispersalloy	0.4	130.0
Luna Atomic	0.8	8.2
Amalcap Non-Gamma 2	1.0	20.3
Luxalloy Non-Gamma 2	1.0	70.8
Hi-Atomic	1.2	7.6
New True Dentalloy	1.2	8.8
G. & G. Superfine	1.5	17.2
Spheralloy	1.8	0.7
Ultrafine	1.9	2.6
Caulk Spherical	2.0	4.6
Amalcap F	3.1	4.7
F400	3.2	6.2

* No fracture within test period limit.

Table A-3 relates extended 72-hour creep values to fracture times. The inverse relationship between these factors seen in Table A-2 is again exhibited but an even closer relationship of creep to fracture times is afforded by the extended determination of creep. Luna Atomic, for example, exhibiting high 72-hour creep and relatively low 3-hour creep assumes a more 'appropriate' position in the ranking of fracture times of alloys when 72-hour creep values are considered. Alloys of 72-hour creep below 3% can be expected to resist fracture and an amalgam of 72-hour creep above 15% can be expected to fracture in a few hours.

Table A-4 relates fracture time to the three properties tested and enables understanding of the closer relationship between fracture time and extended creep testing compared with the specification 3-hour creep test and the lack of relationship between fracture time and modulus of rupture.

An excellent demonstration of the rheologic property of flow manifested by a solid material is presented in the news article reproduced in Figure A-1. The article reports of an experiment conducted in the Physics Department of the University of Queensland over a period of approximately 60 years whereby 'loading' has been applied to an 'apparently' solid material, pitch. The deformation taking place as a result of air pressure and gravitational force (weight) exerted on the quantity of pitch is observed as the formation of drops which separate from the main body of pitch at exceptionally far spaced periods of time. The experiment provides an analogy to the sub-fracture loading of dental amalgam. Like pitch, amalgam is a solid, composite material which can deform under certain conditions. These conditions must include sufficient time to allow intrinsic mechanisms of crystal movement to occur and

Table A-3 : Relationship between 72-hour creep values and fracture time under sub-modulus of rupture loading, for all alloys.

Alloy	Creep (%)	Fracture Time (hr.)
ANA 2000	0.3	N.F.*
Tytin	0.5	N.F.
Permite C	2.0	N.F.
Dispersalloy	3.1	130.0
Amalcap Non-Gamma 2	11.0	20.3
Luxalloy Non-Gamma 2	12.0	70.8
Hi-Atomic	18.0	7.6
New True Dentalloy	18.0	8.8
G. & G. Superfine	25.0	17.2
Luna Atomic	25.0	8.2
Ultrafine	28.0	2.6
Caulk Spherical	30.0	4.6
Spheralloy	30.0	0.7
F400	40.0	6.2
Amalcap F	40.0	4.7

* No fracture within test period limit.

Table A-4 : Relationship between fracture time and modulus of rupture, 3-hour creep and 72-hour creep , for all alloys.

Alloy	Fracture Time (hr.)	Creep (%)		Modulus of Rupture (MPa)
		72-hour	3-hour	
Spheralloy	0.7	30.0	1.8	90
Ultrafine	2.6	28.0	1.9	111
Caulk Spherical	4.6	30.0	2.0	121
Amalcap F	4.7	40.0	3.1	156
F400	6.2	40.0	3.2	121
Hi-Atomic	7.6	18.0	1.2	135
Luna Atomic	8.2	25.0	0.8	113
New True Dentalloy	8.8	18.0	1.2	112
G. & G. Superfine	17.2	25.0	1.5	141
Amalcap Non-Gamma 2	20.3	11.0	1.0	113
Luxalloy Non-Gamma 2	70.8	12.0	1.0	115
Dispersalloy	130.0	3.1	0.4	150
Permite C	N.F.*	2.0	0.2	128
Tytin	N.F.	0.5	0.1	219
ANA 2000	N.F.	0.3	0.1	139

* No fracture within test period limit.

Not many drips in Que



The glass funnel.

By BOB BEALE,
Science Writer

Patience is a virtue, but a strange experiment at the University of Queensland demands almost saintly forbearance.

Since 1927, successive members of the Physics Department have been monitoring a glass funnel full of pitch which drips unbelievably slowly.

In all that time, only six drops of the dense black substance have fallen from the funnel into a beaker below.

While you could not say the

researchers are holding their breaths in anticipation, the drip is expected to fall after a nine-year journey down the neck of the funnel.

The perfectly formed drop probably elongate, then several threads before finally falling into the beaker, said Professor John Mainstone.

Watching the experiment while watching grass grow, he said.

It was set up by the university's first physics professor, Thomas Parnell, to show

some form of loading to provide deformation impetus. In pitch, the deformation which has occurred is a direct product of the interaction of time, load and flow and has resulted in the formation of droplets. Amalgam deformation, as it occurs in the laboratory and in the mouth, is a direct result of the interplay of time, load and creep resulting in bending and subsequent fracture.

This article on the need for extended research, 60 years and ongoing, may parallel the situation with ultimate time to fracture of Single Melt High Copper amalgams such as Tytin. It may be useful to set up such an experiment with a sub-fracture load Tytin specimen to be observed to fracture. This may take many years and occupy several generations of tutors, lecturers and researchers. It would seem to be inappropriate in a single investigation.

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