THE RELATIONSHIP OF BEAM THICKNESS

TO

TRANSVERSE LOADING OF DENTAL AMALGAM

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INTRODUCTION

Dental amalgam has been the subject of a great deal of research in the fields of Operative Dentistry and Dental Materials. This material has saved and continues to save more teeth than all other restorative materials.

Basic research on dental amalgams has been largely concerned with their mechanical properties and with the development of specifications. The first scientific appraisals of dental amalgam were carried out by Black (1896) who developed a formulation for amalgam alloys to be mixed with mercury. Black's formulation is still the basis for the composition of dental amalgam alloys today. The development of improved techniques for polishing and etching amalgam specimens (by Wing, 1961) and the use of the electron probe microanalyser for positive identification of the phases within amalgam led to accelerated interest in the relationship of the basic nature of amalgam to its clinical behaviour. The microstructure of amalgam is also of value when considered in conjunction with manipulative variables and physical properties. As a result of these research advances, the dispersion-modified alloy and, more recently, the high copper ternary amalgams have been introduced to the dental profession. These new alloys have been developed to improve mechanical properties, particularly creep;

they contain little or no γ_2 phase and therefore should be more resistant to corrosion in oral cavities, and so come closer to the ideal clinical restorative. In the last decade a great deal of interest has centred on the reduction of creep values and the elimination of the γ_2 phase, which may act as predictors of clinical behaviour.

Forsten (1969, 1971, 1972) has conducted numerous tests on the modulus of rupture (transverse strength) of amalgams. The work of Giblin (1979), and Giblin and Wing (1979*a*, *b*, 1980, 1981) involving both modulus of rupture and "sub-modulus of rupture" loading of amalgam specimens, has increased interest in these tests. The sub-modulus of rupture testing appears to reveal factors determining the mechanism of failure of clinical amalgams and relates to their creep values. Little research has been conducted on the "sub-modulus of rupture" transverse loading behaviour of amalgams.

The present study was undertaken in an attempt to find an explanation for the behaviour of beams of dental amalgam of varying thicknesses which have been subjected to "sub-modulus of rupture" transverse loading. The microstructures of amalgams in beams of various thicknesses have been compared with the microstructure of cylindrical specimens on which creep tests have been carried out. The effects of variation of condensation effectiveness on creep values and on the microstructure

of amalgam have been observed. An attempt has been made to assess the true creep value of amalgam, so that its probable behaviour may be predicted, either in an experimental specimen or in a clinical restoration.

A review of the literature pertinent to modulus of rupture and "sub-modulus of rupture" testing is included before detailing the experimental work carried out. A description of the elementary metallurgy necessary in this work may be found in Chalmers (1959), Fenner (1965), Forrest (1962), Smallman (1970), Bunshah (1971), and Schlenker (1971).

REVIEW OF THE LITERATURE

CHAPTER 1

MECHANICAL PROPERTIES OF DENTAL AMALGAM

Dental amalgam has a characteristic quality of tending to creep when subjected to compressive forces. Under a continued mild application of force in compression, an amalgam will show a continued deformation even after the mass has completely set. When subjected to a rapid application of stress either in tension or compression, however, a dental amalgam behaves as a brittle material. Mechanical tests of various kinds are very widely employed as tools by research workers. They give a detailed knowledge of the material as affected by the size, shape, method of finishing, etc., of components under actual service conditions.

Compressive Strength

Traditionally for many years the strength of dental amalgam has been measured under compressive stress, using a cylindrical specimen of dimensions approximately comparable with the volume of a typical amalgam restoration.

Taylor *et al.* (1949) found that compressive strength testing is dependent on the size of the specimen and the rate of loading. He emphasized that the specimen should maintain a ratio between height and diameter of 2:1.

The compressive strength decreases as the specimen increases in size, while the compressive strength increases with the rate of loading. These findings were in agreement with the later results of Caul, Longton, Sweeney, and Paffenbarger (1963), and Mahler (1972). They noted that the effect of the rate of loading is more significant in materials with high creep.

Miller (1959) found that the strength of an amalgam in the first hour was a factor of clinical importance. It is during this period that a restoration can easily be fractured. It is therefore important that an amalgam which has a high one-hour compressive strength should be used. Wing (1975) suggests that a better measure of the quality of amalgam, if compressive strengths are to be used, is the one-hour compressive strength, which tends to give a greater spread of results and to be influenced by manipulative variables more than the fully set compressive strength.

Many investigators have studied the effect of manipulative variables and temperature on compressive strength (Wing, 1961, and Caul *et al.*, 1963).

Wing (1961) and Phillips (1973) investigated the effect of trituration on the compressive strength of amalgam. They found that under-trituration produced weaker amalgam, and over-trituration produced stronger amalgam. However, it is apparent that after a certain

optimal period, continuation of trituration has no marked effect on the strength. Spherical amalgams are not as strongly influenced by reduction in trituration as lathe-cut alloys. Spherical amalgam alloys triturate to form a plastic mix in a considerably shorter time than lathe-cut alloys. This situation arises because of the shape of the spheres and because of the way this material is manufactured.

Wing (1961), and Caul *et al.* (1963) investigated the effect of temperature on the compressive strength of amalgam. The strength at 60°C (140°F), a possible temperature for hot coffee, may be 50 per cent of that at room temperature. Even at body temperature, 37°C (98.6°F), a loss in strength of approximately 15 per cent occurs compared with the strength at 25°C.

Eames (1959), Wing (1961), Mahler and Mitchem (1964), and Phillips (1973) studied the effect of mercury content on compressive strength of amalgam. It is agreed that the mercury content in the amalgam restoration is a very important factor in the control of the strength of restoration. The advantages of the minimal mercury technique were first pointed out by Eames (1959). The method is based on the finding that increasing the residual mercury content of an amalgam decreases its strength (Swartz and Phillips, 1956), and that the residual mercury

content increases proportionally with the mercury content in the original mix. Wing (1961) found that a decrease in the initial mercury alloy ratio resulted in a general increase in the one-hour compressive strength. For fully-set amalgam there appears to be a levelling off of the influence of the original alloy:mercury ratio. Where the amount of mercury available is increased above the recommended level there is in all cases a reduction in the one-hour and seven-day compressive strengths. Wing and Hewitt (1965) claimed that the early compressive strength of Eames' technique results from more thorough trituration than the low mercury content used initially. Phillips (1973) and Mahler (1964) found that there seems to be no important effect of the mercury content on the strength of the amalgam within the limits of approximately 45 to 53 per cent. Above approximately 55 per cent Hg content, the strength decreases markedly with increase in mercury content. The results of the investigations by Phillips and Mahler were for fully set lathe-cut amalgams. Wing (1975) showed that for spherical amalgams, mercury contents above approximately 45 per cent produced lowered compressive strength.

Ward and Scott (1932), and Wing (1965, 1971) studied the effect of condensation on the strength of amalgam. It has been assumed that an increase in condensation pressure produces an increase in compressive

strength, and a figure of approximately 56 MPa has been accepted as optimal, and necessary for the condensation of amalgam.

Wing (1965) pointed out that condensation is not related only to pressure, but a number of factors are of significance. Good condensation techniques have the effect of increasing the proportion of original alloy or core at the expense of the amount of the matrix formed. Although there was a tendency for poorly condensed restorations to exhibit slightly lower-strength values in the fully set condition, it was found that, provided initial proportioning had been correctly carried out, followed by proper trituration and mercury removal, these differences in compressive strength were insignificant. The influence of condensation pressure on mercury content, porosity, and strength is less significant when spherical alloys are used. This may be due to the superior flow characteristics of the spherical particle amalgams, which allow more complete compaction at the lower rate of packing. These results are in agreement with the later work of Basker and Wilson (1973).

Bryant (1979) investigated the rate of development of compressive strength in amalgam. The results show statistically significant increases in compressive strength between 30 minutes and one hour, and between one hour and one day. The compressive strength after one day was almost

100 per cent of the fully set strength. However, the maximum strength is not achieved until after one to six months.

Many of the recently developed alloys possess improved properties of dynamic and static creep, compressive and tensile strength. (Mahler and Van Eysden (1969*a*), Bryant and Wing (1976), Malhotra and Asgar (1978), and Eames and MacNamara (1976)). These alloys have a total copper content much greater than in the conventional Ag_3Sn alloys and show a reduction in, or virtual elimination of, the γ_2 (Sn-Hg) phase in the matrix of the set amalgam. Wing (1979) suggests that the structures of different types of amalgams are probably reflected to some degree in the mechanical properties. The fact that the dispersion modified amalgams do not have a higher early strength than "Ag_3Sn" amalgams is probably a reflection of the early presence of the γ_2 phase; the failure of this type of material to develop higher strengths even when fully set may be a reflection of a poor interface between the γ_1 matrix and the Cu_6Sn_5 halo following setting. As opposed to this, the rapid development of early strength of ternary high copper amalgams is probably a measure of the good alloying between original alloy and γ_1 matrix and the rapid development of strength of the Sn-Cu-Hg phase (Wing, 1979).

Mahler (1969*b*) demonstrated that as the loading rate was reduced the correlation coefficient between

compressive strength and dynamic creep was increased. It is suggested that conducting the test of 7-day compressive strength at a loading rate .001 in/min. will produce significant results for evaluating amalgam alloys for their creep characteristics. Mahler *et al.* (1970) found that compressive strength, tensile strength, transverse deflection, and flow as determined by ADA Specification No. 1 were ineffective, whereas the rheological properties of dynamic creep, static creep, and slow compressive strength appeared effective in relation to marginal fracture.

Even though compressive strength may not always be the major physical property associated with the clinical fracture of amalgam, it seems to be a reliable test for predicting resistance to stress. Also, the compressive strength value is reasonably indicative of the level of other strength properties. Furthermore, the manipulation variables which influence compressive strength generally have a comparable effect on the other mechanical properties.

Tensile Strength

Amalgam has only a limited tensile strength; tensile tests which have been made by Ward (1924) and Taylor (1930) indicate that amalgam has between one-seventh and one-tenth as much tensile strength as crushing

strength. Rodriguez and Dickson (1962), and Nagai *et al.* (1968) gave it as one-quarter to one-fifth respectively. The low tensile strength of dental amalgam limits its usefulness as a restorative material. The tensile strength of amalgam is approximately 56 MPa, or even less, while the tensile strength of dentine is approximately 276 MPa. Therefore, the cross-sectional area of the isthmus in the prepared cavity should be sufficient to compensate for this weakness, at least in part. The cavities receiving it must be so constructed so that all walls will meet the cavo-surface at as nearly right angles as possible, to provide greater bulk of material than is necessary for gold restorations (Coy, 1957).

Mahler (1958) used photoelastic studies to investigate the stress acting in a Class II restoration, concluded that failures at the isthmus are due to tensile stresses.

Nagai and Ohashi (1968) studied the tensile properties of spherical amalgam. They found that the tensile strength of spherical amalgam, in its initial phase in particular, is much better than conventional amalgam, even if it is condensed under a low pressure. The tensile strength of the former material, after 1 hour, was approximately twice as much as that of the latter. They also noted that the amalgam which has a large tensile strength is found to have a correspondingly

large modulus of elasticity. These results are in agreement with those of many investigators (Koran and Asgar (1967), Nagai *et al.* (1970), and Eden and Waterstrat (1967)). Although excellent properties of spherical amalgam compared with conventional lathe-cut amalgam have been variously confirmed, it is pointed out by Nagai and his associates (1970) that these properties vary from spherical amalgam product to product with some being inferior to conventional amalgam products.

In those studies previously undertaken, test specimens were made in the form of dumb-bells and for this reason, they tended to be broken by the chuck at testing times resulting in a wide scattering of measurement data. Burns and Sweeney (1965) applied the diametral compression test for tension to dental amalgam and confirmed its reliability in enabling them to obtain the initial tensile strength of amalgam, eliminating the possibility of breaking by the chuck and also the need for a grip. This method has now become very popular and is frequently used.

Nagai (1970) compared two methods for measuring tensile strength of amalgam; the diametral compression and direct tensile tests. It was found that the tensile strength obtained from dumb-bell shaped specimens and diametral tests indicates frequently a higher value for

this property from the latter test. Nagai (1970) showed that the percentile distribution of the average tensile strength with 7-day strength as 100 per cent is as follows: 15 minutes (8 per cent), 30 minutes (15 per cent), one hour (29 per cent), 6 hours (82 per cent), and 24 hours (96 per cent). The further increase in strength after 90 days was only 2 per cent.

The compressive to tensile strength ratio is 6.9 for spherical and 7.5 for lathe-cut amalgam. These results are in agreement with those of Bryant (1979) who also noted that compressive to tensile strength ratios tend to be higher for those alloy types containing higher levels of copper and less γ_2 phase in the set amalgam.

Lautenschlager and Harcourt (1970), and Turchyn and Youdelis (1970) pointed out that even though amalgams have generally been characterised as brittle materials, they have demonstrated that substantial plastic deformation occurred prior to fracture. Therefore, the diametral compression test does not give a true value for tensile strength of amalgam, particularly for alloys with high creep values. Recent high copper ternary and dispersion modified amalgams have very low creep values and brittle characteristics, so that diametral tensile test may be a good gauge as to the true tensile strength of these materials.

Young and Johnson (1967) have shown that the way to improve the tensile strength of amalgam may lie in reducing the amount of the tin-mercury present.

Young and Wilsdorf (1968, 1972) showed that surface contaminations of the Ag_3Sn particles could cause flaws in the amalgam and possibly would act as crack nuclei or, in any event, be detrimental to the bonding between phases. They prepared surface clean dental alloy by washing Ag_3Sn powder in 5% HCl before amalgamation. The resulting amalgam showed a considerable decrease in the scatter of strength data and exhibited 20 per cent higher tensile strength. The reason for this substantial improvement in the strength of dental amalgam was revealed by electron fractography. It was found that the increase in strength may be due simply to a reduction in the size and number of voids present; this reduction is a consequence of the superior bonding of washed particles to the amalgam matrix, and the fracture propagated through the alloy particles as well as the matrix crystals. Wing (1971, 1975) has placed great importance on the alloy-matrix interface.

Bryant (1979) compared the compressive and tensile strengths of fifteen conventional and high copper amalgams. He found that the amalgams tend to achieve maximum tensile strength at an earlier time than maximum compressive strength. Alloys of the single melt high

copper type generally possess the greatest strength at all times of testing.

Vrijhoef, Vermeersch and Spanauf (1979) investigated diametral tensile strength of available commercial amalgams. The study suggests that a diametral tensile strength in the range 35-55 MPa is sufficient to prevent bulk fractures as long as cavity preparation and materials handling are carried out carefully. Therefore, there is no clinical relevance to the prevention of bulk fractures of amalgam restorations under oral conditions. In that respect, the introduction of the high copper alloys did not change the dental picture as to the property of the tensile strength. The tensile strength of a particular dental amalgam is considered to be a relatively poor guide to the selection of a dental amalgam alloy, because the occurrence of bulk fracture depends so strongly upon the actual cavity design.

Transverse Strength

It is accepted that amalgam restorations in the mouth are subject to combinations of compressive, tensile and shear stress. In clinical usage, the term "edge strength" is frequently used to describe the ability of a restorative material to withstand fracture of a thin edge. In engineering testing terms, this property might

be more appropriately named bending strength or transverse strength, or modulus of rupture, or flexure strength. The transverse strength is obtained by loading a bar or beam, which is supported at each end. The load is usually a single centre one, but may be applied symmetrically at two points between the supports. Such a loading will obviously cause a deformation of the test sample and, if continued until failure occurs, will produce comparative strength values for different materials. The formula for transverse strength is:

$$T.S. = \frac{3PL}{2bd^2}$$ for rectangular cross-sections

where:

P = fracture load;

L = length of span;

b = width of beam;

d = thickness of beam.

Notice that the strength is more critically dependent on the thickness than on the width: Transverse Strength is inversely proportional to the first power of b, but to the second power of d. The resulting deformation in such a beam can be calculated from another equation:

$$\text{Deformation } (g) = \frac{PL^3}{4Ebd^3}$$

where:

P = fracture load;

L = length of span;

E = modulus of elasticity;

b = width of beam;

d = thickness of beam.

It is interesting that both the length of span and the thickness are quite critical. This knowledge may be of importance in the occlusal step portion of a Class II restoration.

Gabel (1954) pointed out that when the modulus of elasticity of the material of a Class II restoration is higher than that of tooth structure, the restoration function is a curved beam. In the case of these restorations, the proximal portions tend to rotate out of the cavity. The amount of shift is dependent on the ratio of the radius of curvature of the inner surface to the depth of the beam. As the radius of curvature decreases, the bending stresses at the axiopulpal line angle increase (see Fig. 1.1). Therefore, gingival retention and rounding of the axiopulpal line angles are required, as in the proximo-occlusal cavity. The gingival retention required depends on the extent to which the modulus of elasticity of the material exceeds that of tooth structure. The stress in each material is the modulus of elasticity

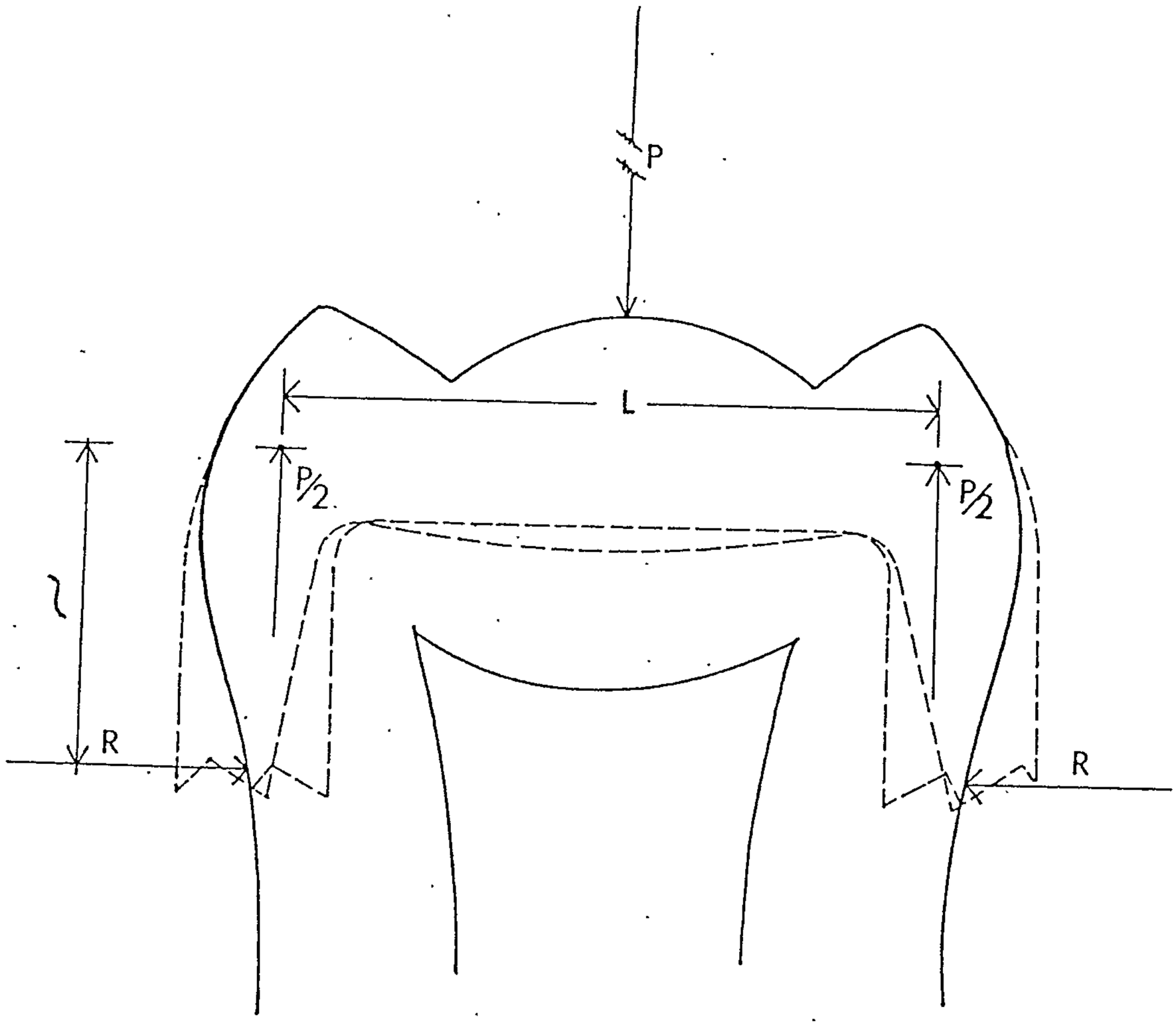

Fig. 1.1. Diagrammatic representation of a possible three-point loading situation with respect to a Class II restoration (after Gabel, 1954).

times the unit of deformation or strain. The modulus of amalgam is higher than that of deciduous dentine; this may account for the failure of Class II amalgam restorations in deciduous teeth.

Mahler and Mitchem (1964) investigated the transverse strength of amalgam. The results showed decreasing values of compressive, tensile and transverse strength of amalgam as the residual mercury content increases, with a sharp decrease evident at approximately 54 per cent mercury content, for fully set lathe-cut amalgams. There are good correlations between these three strength types and residual mercury content. This indicates that the strength of amalgams may be represented by any of these three strengths. Mahler and Mitchem (1964) try to relate a transverse strength of amalgam to clinical failure of amalgam restorations, especially in marginal failures. However, Mahler *et al.* (1970, 1973) found no apparent relationship between the compressive strength, tensile strength, transverse strength and marginal fracture under clinical conditions.

Forsten investigated the influence of manipulation techniques on transverse strength of amalgam. Forsten (1969) found that delayed condensation had a marked weakening effect on the fully set transverse strength of amalgam specimens, particularly when a minimal initial

mercury, or "dry" technique was used. It has been suggested that the decrease in strength due to delayed condensation is caused partially by disrupting the crystalline structure (Skinner and Phillips, 1967). Forsten (1971) evaluated the influence of different mixers, pestles, trituration time and precondensation mercury contents on the transverse strength of amalgam. The strength values obtained in this study indicate that the use of an ultra-high-speed mixer for mixing amalgam will produce maximum strength more easily than the high-speed amalgamators. A medium sized cylindrical metal pestle was found to be the most efficient. An increase of the trituration time increases the strength of the amalgam only to a certain limit. He also noted that although Dispersalloy* showed the lowest transverse strength of the amalgams tested, it was the least susceptible to manipulative variables. Forsten (1972) investigated the influence of changing the precondensation mercury content on the early and final transverse strength of different amalgams. The results show that the early strength was lower for the preamalgamated amalgams than for the other amalgams. A moderate excess of precondensation mercury does not seem to decrease the early strength of amalgams, but does tend to increase the final strength. Thus, the claim of gaining higher final strength by using the minimal mercury techniques (Eames, W.B., 1959) cannot be justified. In fact, there is a greater risk that a restoration made using the minimal mercury technique

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* Unitek Co., U.S.A.

will have lower strength, especially since this technique produced a higher variance of the test results. Forsten suggests that, clinically, excess precondensation mercury should be used.

The testing transverse strength will be considered in more detail in Chapter 2.

Creep

When a metal is stressed, it undergoes immediate plastic deformation and in the first short period of time makes additional plastic adjustment along flaws within its internal structure. After these initial changes, a slow, almost steady rate of strain occurs, which is called creep. Depending upon load, time and temperature, the creep of solid system is often as represented by Fig. 1.2. Four regions may be identified:

Region I - Instantaneous deformation during initial application of load.

Region II - Transient creep, in which the creep rate decreases continuously with time.

Region III - Steady state creep, in which a constant creep rate is observed.

Region IV - Tertiary creep, in which creep accelerates and finally leads to fracture.

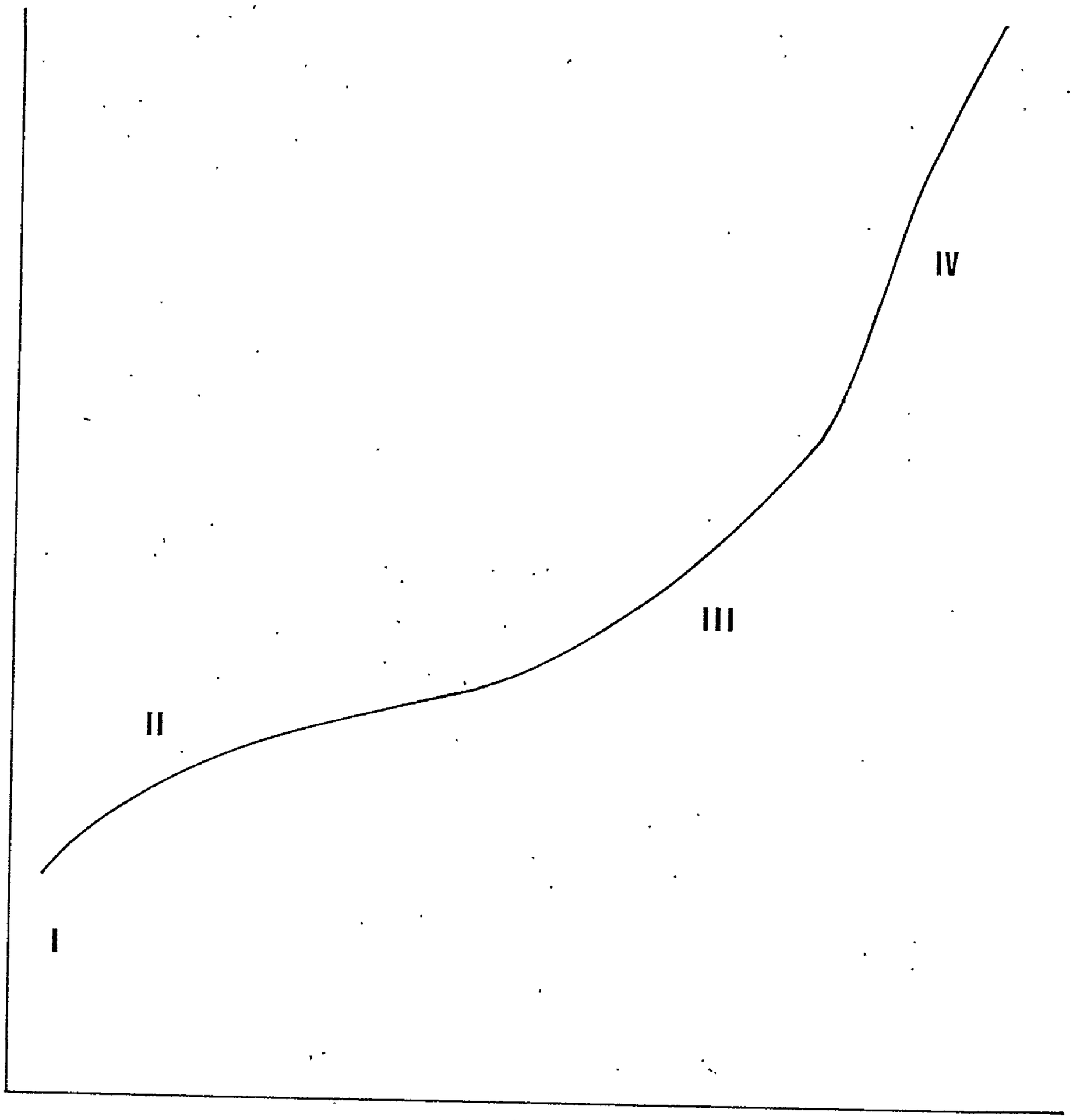


Fig. 1.2. Four regions of creep often observed in solid systems (after Greener, Szurgot, and Lautenschlager, 1980).

Amalgam creep is measured in a region of steady state creep. Experimental values for the creep of amalgam are expressed quantitatively by determining the progressive permanent deformation of the set material which occurs under a constant stress of 36.2 MPa (static creep) or intermittent stress between 3.5 MPa - 68.8 MPa (dynamic creep). It measures the viscoelastic response of the amalgam matrix phases to static loading and can be correlated directly with the amount of matrix present.

Dickson, Oglesby and Davenport (1968) studied the constant creep rate region III for an unspecified amalgam in the tensile stress range of 3 to 28 MPa and in the temperature range 23 to 52°C. They found that the extent of creep in amalgam is a function of stress and temperature.

Several clinical studies have shown that the margins of restorations made of alloys with high creep break down more rapidly than margins of restorations made of alloys with low creep (Duperon *et al.* (1971), Binon *et al.* (1973), Mahler *et al.* (1970, 1975)).

Mahler *et al.* (1970) found a correlation between the creep test result and the incidence of clinical marginal fracture of amalgam restorations by comparing a dispersion strengthened amalgam with conventional alloy amalgam. They concluded that the decreased marginal

fractures of the dispersion strengthened amalgam were the result of an improvement in the mechanical properties caused by the absence of the γ_2 phase. It was thus suggested that, although there may not be a direct causal relationship between creep and marginal fracture, measurement of creep may be predictive of clinical marginal behaviour.

From Jørgensen's theory (1965, 1974) of the so-called *P*-type marginal breakdown, it might be concluded that the amalgams which corrode but do not release mercury will not show marginal breakdown of the *P*-type. For those amalgams, creep is not expected to correlate with marginal breakdown. Furthermore, it may be concluded that the materials which corrode with the release of mercury will show marginal breakdown of the so-called *P*-type. For these amalgams, the study of creep is expected to be of value in predicting marginal fracture in oral cavities.

Osborne *et al.* (1974*a*) reported that a spherical alloy amalgam containing a γ_2 phase also showed a creep value comparable to that of the dispersion strengthened non γ_2 amalgam, and they found no significant decrease in clinical marginal fracture with the spherical alloy amalgam, despite a low creep value. This result is, of course, quite different to those obtained by dispersion strengthened non γ_2 alloys. This report was supported

later by Takatsu *et al.* (1977), and Jordan *et al.* (1978) who also noted that low creep may be important to enhanced marginal integrity only when a mechanism exists for the elimination of the γ_2 phase. It seems probable that the low incidence of marginal fracture of the dispersion strengthened amalgam is caused by the improved chemical resistance of the γ_2 free amalgam.

Osborne *et al.* (1974*b*) indicate that manipulation factors such as trituration time and condensation pressure can affect the creep within the same alloy. The graph of creep vs. trituration time was found to be parabolic, with an optimal trituration time. Condensation pressure markedly affected creep. In general, the higher the condensing force, the lower the creep.

Spanauf, Vrijhoef and De Graaf (1977), and Espevik (1975*a*) have shown that ageing of dental amalgam at mouth temperature, and above, leads to a reduction in creep. These results are in agreement with those obtained later with Patel, Cruickshanks and Boyd (1979) who found that ageing leads to a reduction in creep but does not affect the compressive strength.

Espevik and Sorensen (1975*b*) studied the compressive creep of 22 dental amalgams at 20^{o}C and 20 MPa. They identified the steady state creep rate by evaluation

of the slope of the straight line portion which began some 10 hours after application of the load. Correlating their data with microstructure, these authors found that a high creep was associated with small concentrations of γ and large volume fractions of γ_1 phase. They point out that the Cu-Sn phases dispersed in γ_1 may have a role in decreasing creep through hindrance of dislocation motion. Espevik (1977*a*) has subsequently confirmed these results by investigating the creep of compounds with a composition equivalent to the γ, γ_1 and γ_2 phases in dental amalgam. He showed that heat treatments of both conventional and copper-rich amalgams at 60°C and 70°C dramatically reduced the dental creep through conversion of γ_1 matrix to β_1. These results are in agreement with Mahler, Adey and Marantz (1977*a*) who found also that the creep decreases as the grain size of γ_1 increases in γ_2 containing amalgams.

Osborne *et al.* (1974*a*), Eames and MacNamara (1976), Bryant and Wing (1976), Malhotra and Asgar (1978), and Bryant (1980) have observed a relationship between static creep and the composition of the original alloy particles. The results showed that the silver-copper dispersed alloys and the single melt high copper alloys were generally found to possess markedly lower static creep than conventional silver-tin amalgams. The silver-copper dispersed alloys show a reduction in creep of 40-65 per cent in a

period of three hours to one day. This compares with a reduction of only 6-25 per cent in this period for the conventional alloys (Bryant, 1980).

Mahler (1979) investigated the behaviour of three high-copper amalgams. The curves of creep vs. final mercury content all showed a sudden creep jump at approximately 46 per cent final mercury content. This creep jump has been determined to be a forerunner of the formation of the Sn-Hg (γ_2) phase (Mahler and Adey, 1977*b*). However, under clinical conditions, amalgams made from these alloys normally contain between 40-42 per cent mercury and γ_2 would not be expected. Therefore, their behaviour at and beyond 46 per cent mercury is only of theoretical interest.

The importance of the creep of dental amalgam has resulted in the inclusion of a creep test in the revised American Dental Association Specification 1977 and the Australian Dental Standard 2100 for Dental Amalgam Alloy. These specifications allow a maximum measured static creep of 5 per cent and 4 per cent respectively. Although the exact significance of the creep characteristics of the alloy and marginal breakdown has yet to be established, this property should be useful to the dentist in the selection of amalgam alloys. It must be emphasized that factors other than creep

influence the clinical behaviour of amalgam; these factors include the patient's oral hygiene, operator variables, cavity design, moisture contamination, and corrosion potential of the alloy.

CHAPTER 2

THE TRANSVERSE TESTING

Transverse testing has been determined for a wide range of materials in many fields where the prediction of performance of structures is considered important. This strength has not been widely determined for amalgam and might possess some advantages over compressive and tensile strength.

The actual test involves placing an appropriate specimen on two fulcrum-type supports set a known distance apart, applying a gradually increasing load, and measuring deflections using a suitable deflectometer. Various different systems of loading are employed, the two most common systems being central-point loading and loading at the centre-thirds. Different methods of load application induce different stress distributions within the test beam. Figure 2.1 shows the bending moment distributions for the two previously mentioned load systems.

A neutral plane exists in a beam subjected to pure bending, the bending stresses along this plane being zero. The neutral plane is usually located along the centroid of the beam. Compressive stresses exist in those parts below it. Most materials fail in tension

rather than in compression under bending loads. The results of flexure tests are usually expressed in terms of the transverse strength at the proportional limit, when:

$$T.S. = \frac{\text{Bending Moment at Proportional Limit}}{\text{Section Modulus}}$$

Since the section modulus = $\frac{bd^2}{6}$ for rectangular beams, where b = width and d = depth of the beam, this becomes:

(a) $$T.S. = \frac{\frac{PL}{4}}{\frac{bd^2}{6}} = \frac{3PL}{2bd^2}$$

if centre-point loading is used, or:

(b) $$T.S. = \frac{\frac{PL}{6}}{\frac{bd^2}{6}} = \frac{PL}{bd^2}$$

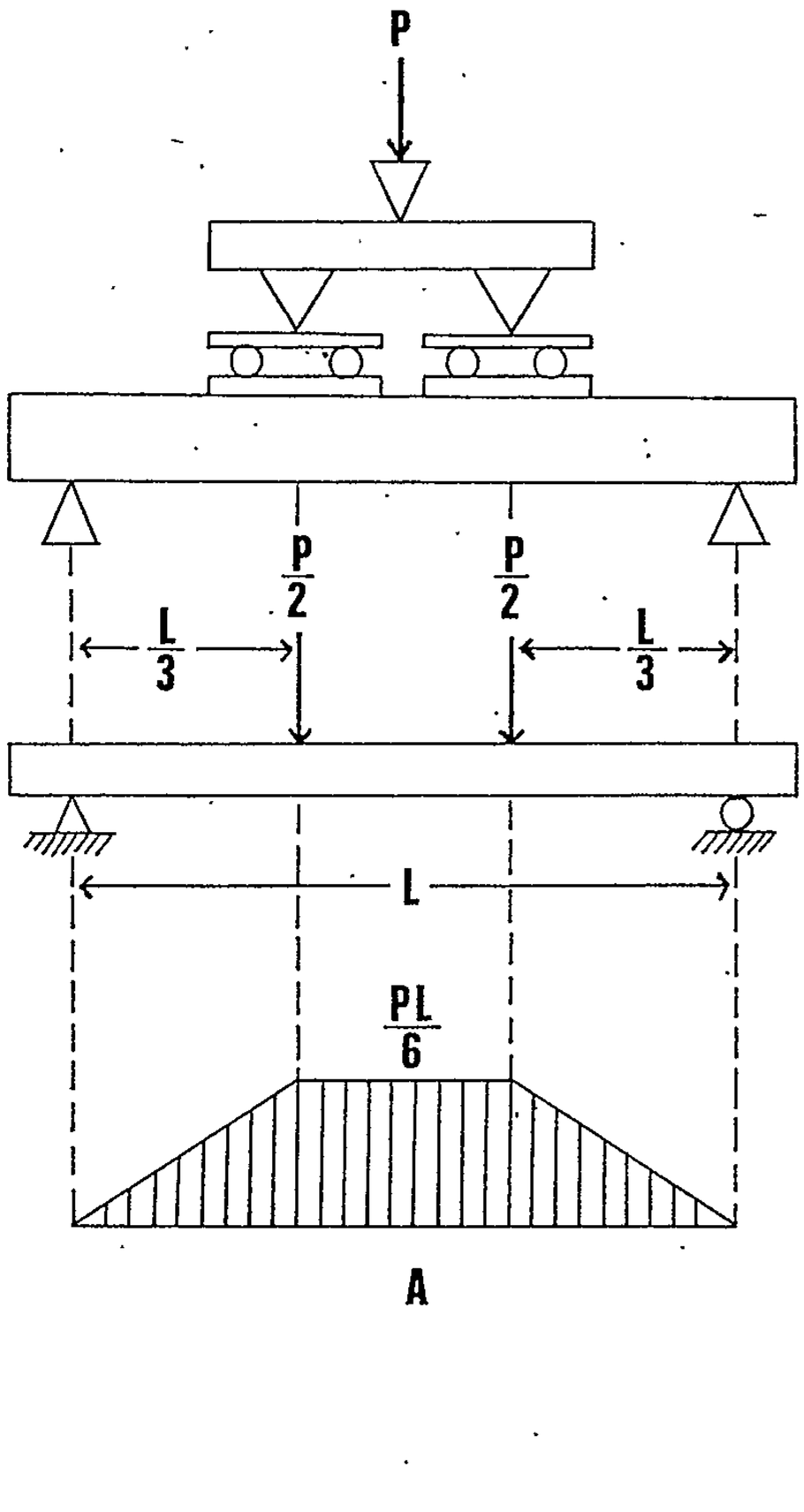
if centre-third loading is applied,

where:

P = applied load, and

L = gauge length.

Amalgam can be considered to be a brittle material when subjected to reasonably rapid loading rates, and the design of the transverse strength test was patterned after



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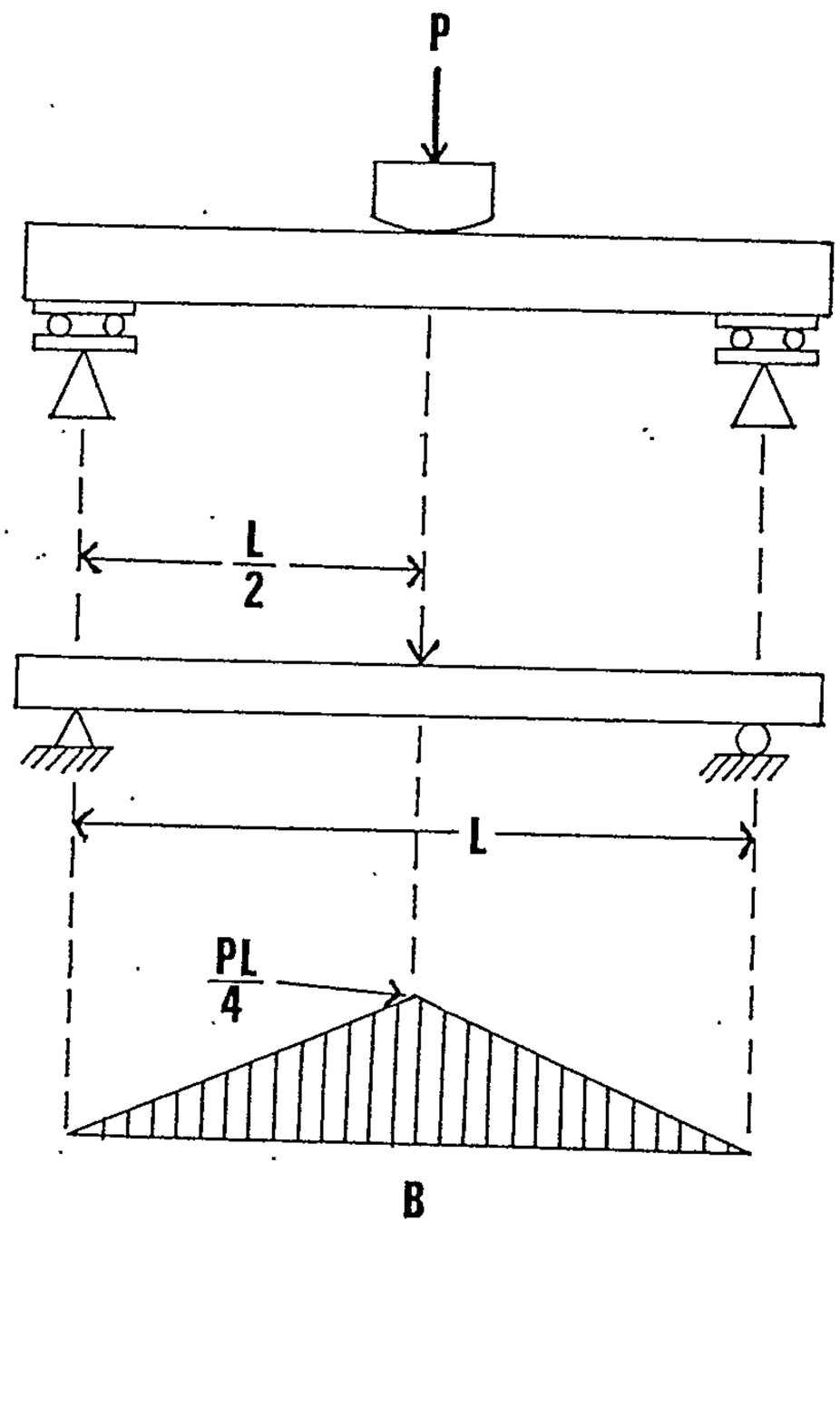

B

Fig. 2.1. Bending moment distributions in beams subjected to:

a) centre-thirds loading; and

b) centre-point loading

(after Schlenker, 1971, p. 89).

that prescribed by Gray (1948) for testing brittle materials in engineering practice. Mahler and Mitchem (1964) studied the transverse strength of amalgam. They designed their transverse test after the method of Gray (1948) for testing brittle materials. The test sample measuring 1 x 4 x 12 mm is placed between two cylindrical supports and the load applied through a ball on the top of the sample at the centre. The rate was selected to be 0.127 cm/min., and the transverse or bending strength was determined. The evaluation of the transverse strength of amalgam at specific amounts of residual mercury was made by comparing it to compressive and tensile strength. They found that the transverse strength appeared well suited to laboratory testing of the strength of amalgam because of its low variation in strength values among test samples, its direct correlation to both compressive and tensile strength, and the relative simplicity of its testing procedure. They also noted that the rate of loading does not significantly affect transverse or tensile strength, whereas compressive strength is sensitive to this variable.

Asgar and Sutfin (1965) observed the effect of bending strain on dental amalgam specimens by using a Flinn strain-viewer. Because of the brittle nature of amalgam, the concept of a two-phase material was applied to restrict crack propagation. In two-phase materials, a strong and rigid amalgam was embedded in a weaker, but

more ductile self curing resin material. The ductile material absorbs the strain energy release when an individual fibre of the brittle material fails and transfers the stresses to the remaining fibres of the brittle phase. The specimen, in form of a beam, is restrained from any movement other than pure bending. The convex side of the beam undergoes tension and the concave side undergoes compression. By using this technique, the neutral axis was not in the middle of the amalgam specimen but instead was approximately one-fourth of the thickness from the tension edge. Therefore, only one-fourth of the amalgam specimen was under tension and the remaining three-fourths under compression. The crack originated on the tensile side. As the deformation was increased, the crack lengthened but usually became completely arrested about halfway through the amalgam. Another crack originated from the compression side to complete the failure. After initial preparation, the specimens were cyanide etched and remounted to reveal grain structure (Wing, 1961). Analysis of these photomicrographs indicates the crack passes most frequently through voids, γ_2 phase and γ_1 phase. Usually the crack goes around the γ phase, in none of the specimens does the crack originate in the γ phase. However, when the crack is almost one-half the depth of a beam and meets a γ phase, the direction does not change to go around the phase. Here the crack goes through the γ phase, which appears to be fractured at grain boundaries or along slip

planes. When no voids or γ_2 phases are available for passage of the crack, it goes along the grain boundaries of the γ_1 phase. This type of failure is described as intergranular and is characteristic of brittle materials. This result is in agreement with later work of Young and Wilsdorf (1968, 1972).

Mahler and Mitchem (1964), and Mahler (1958) have thought the bending strength or modulus of rupture best represents the edge strength. Iwaku *et al.* (1966) compared the edge and bulk strength of powdered gold with amalgam by using the three-point loading system. The bending strength determined in this study was, however, similar for both gold foil and amalgam fillings, which are generally believed quite different in edge strength. Therefore, there must be some other technological property that better represents the edge strength. They suggested that the edge strength of metallic restorative material is considered to be represented by the maximum bending, and the bulk strength by the bending strength.

Fenner (1965) stated that the transverse test was a simple mechanical test. The basic requirements of the bend test are met by the simple arrangement. The test piece is laid on two parallel supports and bent by pressure applied through the mandrel at the centre. The mandrel is usually of hardened steel and has a radius

equal to that specified as the internal radius round which the test piece is to be bent. Rounding the edges of the supports is necessary. As the test piece bends it is drawn over these edges; if the edges were too sharp they would damage the test piece and impede the movement. On the other hand, frictional forces here help to keep the test piece in contact with the roller and tend to prevent *peaking*. A test piece is said to 'peak' when it suffers a very severe deformation over a short length at the middle, where it loses contact with the mandrel. The distance between the supports needs to be between $D + 2.5a$ and $D + 3.5a$, where D is twice the radius of the mandrel and a is the thickness of the test piece. Bend up to 120^{o} can be accomplished. Fenner introduced a modified bend test which used roller supports. This technique is a simple modification to the form of the supports, to cater for tests of materials of different thickness, while using a single fixed pair of supports and insert rollers of various diameters (see Figs. 2.2a and b).

Sutfin and Ogilvie (1970) studied the transverse fracture of dental amalgam and the nature of the grain boundaries of Ag-Hg phase using the SEM. There was observed evidence of microplastic deformation in the matrix, and no discrete grain boundary segregations. Etched specimens did reveal evidence of a compositional inhomogeneity across the diameter of larger Ag-Hg grains.

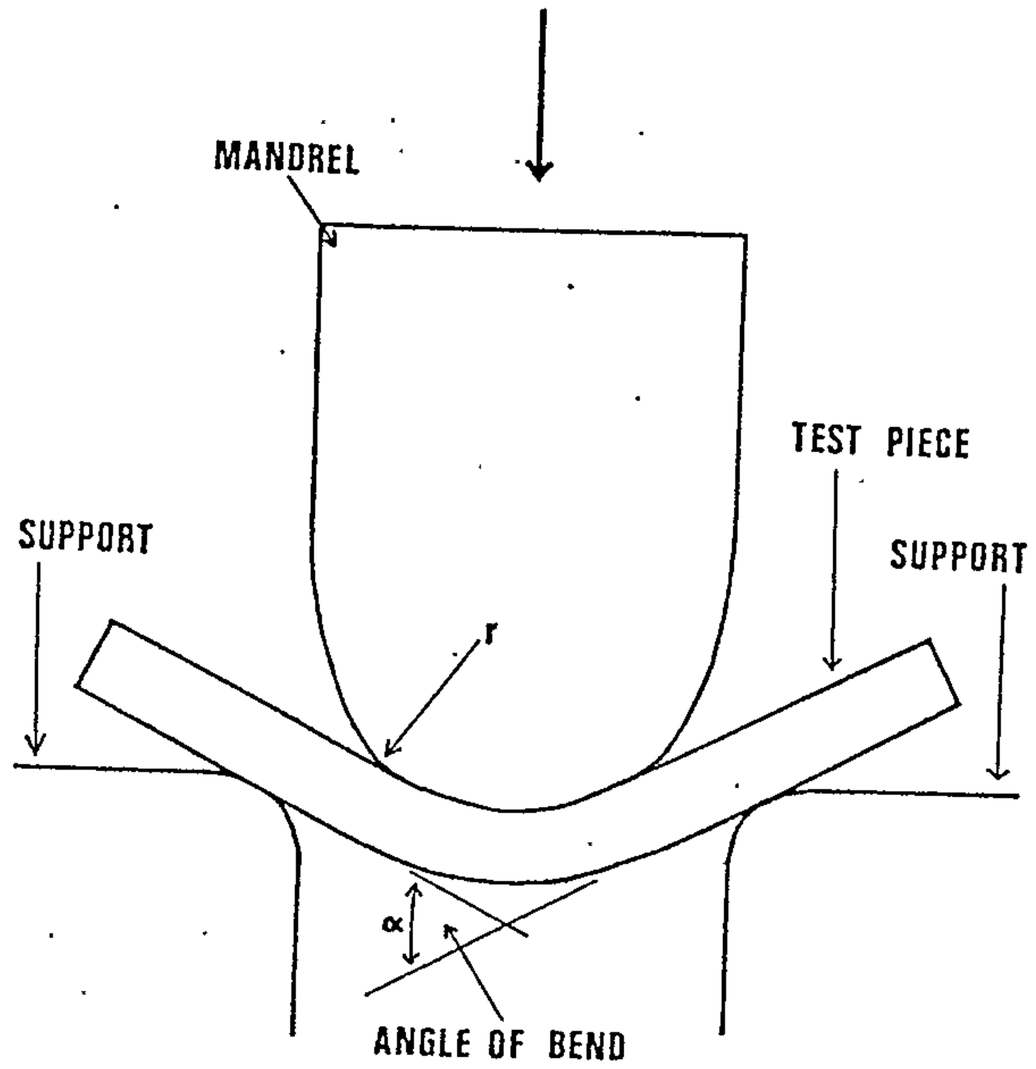

Fig. 2.2*a*. Simple bend test (after Fenner, 1965, p. 76).

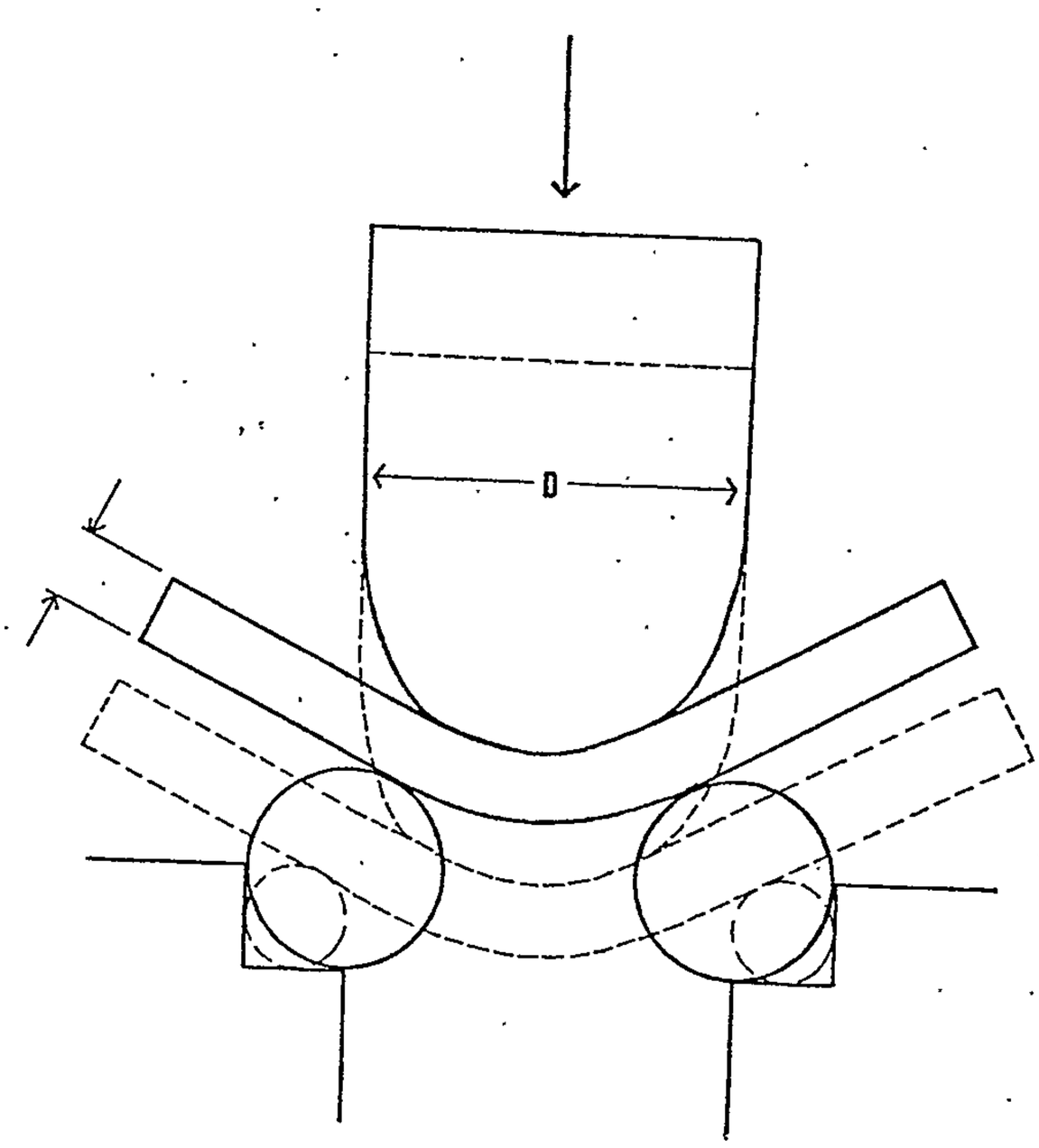

Fig. 2.2*b*. Modified bend test using roller supports (after Fenner, 1965, p. 76).

Peyton and Craig (1971) determined the transverse strength of a beam by the photoelastic method of analysis. This test indicates both the strength of the material and the amount of distortion to be expected. Phillips (1973) considers that the transverse test is "in a sense, a collective measurement of all types of stresses simultaneously".

Turchyn and Youdelis (1970) used a three-point load transverse test to determine the flexure strength of cylindrical 4 x 12 mm specimens. Their results showed that the plastic deformation of the specimen decreased the actual tensile stresses, and accordingly a higher load must be applied to achieve fracture resulting in higher apparent tensile and flexure strength.

Forsten (1969, 1971, 1972) conducted numerous experiments concerning transverse strength. The transverse strength testing was used by Forsten for the following reasons:

1. The 'clinical' fracture of amalgam, and breakage of the transverse strength specimens are both due to a complicated mechanism involving different types of stress.

2. The test is easy to perform, time-saving and almost independent of the crosshead speed.

3. Specimens of 'clinical' size and shape may be used. Furthermore, it may be expected that improper

trituration or large internal voids in the restoration, will show up better in this type of test than when a compressive test is used.

Younis, Asgar and Powers (1975) studied the force required to initiate cracks in dental amalgam beams and related this to the microstructure by using the Flinn Strain Viewer. The force was dependent on age and on the distribution of phase on the tension side of the beam. It was observed that in every instance where the three phases (γ, γ_1 and γ_2) were presented, the crack initiated in the γ_2 phase, followed by the γ_1 phase did not propagate further when it encountered a γ particle. This suggests that the γ particle is a strong phase. In dispersion strengthened alloys, there are two unreacted particles, Ag-Sn particles and the centre of the Ag-Cu particles. A Sn-Cu phase forms around the unreacted Ag-Cu particle. The crack initiated in the Ag-Hg phase in all of the samples except one in which the crack initiated through the Ag-Sn phase. This suggests that the Ag-Cu phase is more resistant than the Ag-Sn phase or that the interface represents a weakness along the crack to pass around the spherical Ag-Cu particle.

Giblin (1979), and Giblin and Wing (1979*a*, 1979*b*, 1980, 1981) reported on studies involving transverse testing of dental amalgam. In these studies, a bar type of loading mandrel was used and a jig to ensure reproducible

centering of the applied load was developed. The results presented confirmed a lack of direct relationship between transverse strength and compressive and tensile strengths. Information on the microstructure of the propagated crack during testing was presented. This information largely parallels the findings of Younis *et al.*, 1975.

It would appear then that transverse strength testing, in view of its lack of sensitivity to rate of loading, its respectably low variation between test samples and its relative ease of testing would have considerable advantages when testing the strength of dental amalgam. It is suggested, therefore, that this test be considered if a future revision of the present ADA Specification No. 1 were to involve a strength test.

CHAPTER 3

FATIGUE OF DENTAL AMALGAM

Low Load Fatigue and Relationship to Creep

In the mouth, silver amalgam restorations are subjected to fluctuating stresses and, for this reason, it is believed that the fatigue strength of material is actually of greater importance than the static strength. Davis, Troxel and Wiskocil (1955) claimed: "Most structural assemblages are subjected to variation in applied loads, causing fluctuations in stresses in the parts. If the fluctuating stresses are of sufficient magnitude, even though the maximum applied stress may be considerably less than the static strength of the material, failure may occur when the stress is repeated a sufficient number of times. A failure induced in this manner is called a 'fatigue failure'". Such determinations are of considerable importance for certain types of dental restorations subjected to small alternating forces during mastication. Restorations may fail entirely as a result of fatigue without any other cause, although such failures probably are rare. It has been found that for most materials there is a limiting stress below which a load may be repeatedly applied an indefinite number of times without causing failure. This limiting stress is called the endurance limit. The magnitude of the endurance

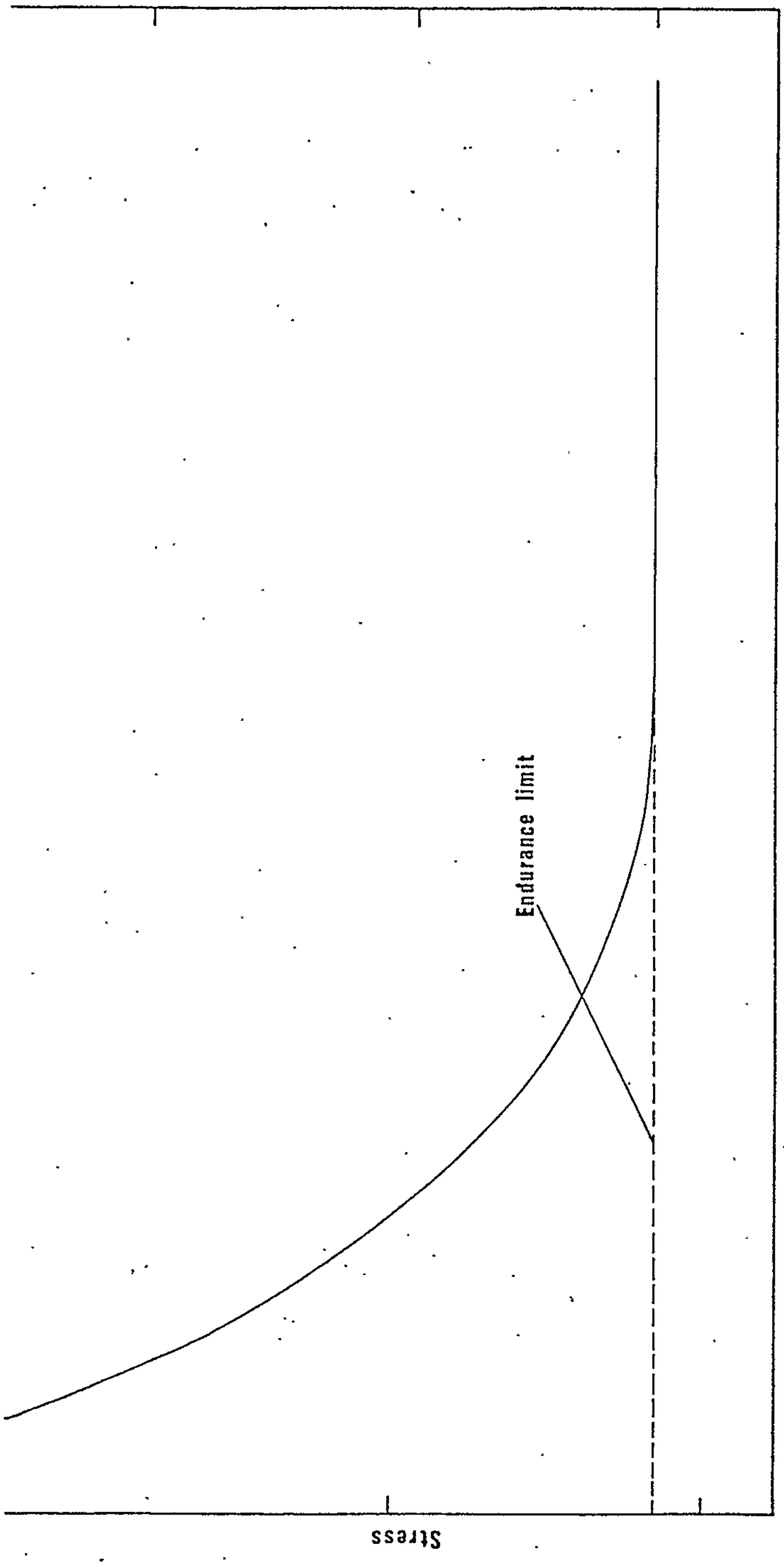

Fig. 3.1. *S-N* curve, showing the endurance limit. For a given stress (*S*) the material fractures after *N* cycles. Log *N* is usually plotted for convenience (after Smallman, 1970).

limit depends on the kind of stress variations to which the material is subjected. It is important in the design of restorations to know what stress it can withstand for essentially an unlimited number of cycles. The fatigue curve is illustrated in Fig. 3.1 where stress (S) is plotted against log N, where N is the number of times the stress has been applied.

Wilkinson and Haack (1958) determined the endurance limit of amalgam under fluctuating stress conditions (fatigue). The results show that, while a restoration might withstand static stress up to 435 MPa, or infrequent short duration stress applications of the same intensity, the highest fluctuating stress which this silver amalgam might be expected to withstand is only 97 MPa, or 22 per cent of the maximum crushing strength. However, Wilkinson and Haack did not relate the fatigue strength of silver amalgam to its other properties; nor was cavity form related to the failure of silver amalgam when subjected to repeated blows.

Forrest (1962) found that those alloys which possess good creep resistance are also resistant to fatigue, although the condition of an alloy giving the maximum creep strength is not necessarily the condition for maximum fatigue strength. The fatigue strength and the creep rupture strength are markedly dependent on

temperature. There is a close correlation between the two for some materials.

Bunshah (1971) has investigated the mechanism of fatigue and has found that the overall process can be divided into several stages, namely, crack initiation, crack propagation, and final rupture; the last occurring when the crack length attained is sufficient to trigger unstable fracture of the remaining section. The important link between each of these aspects of fatigue is dependent upon plastic deformation. The basic processes of "failure" in fatigue are illustrated in Fig. 3.2.

Fatigue test results can exhibit a large amount of scatter. It is seen that the scatter increases with decreasing stress level. The factors which can influence fatigue response were the following:

1) Variables associated with the *material* itself.

2) Variables associated with the *specimen*.

3) Variables associated with the *test conditions*.

It is generally accepted that both fatigue strength and creep depend strongly on temperature and time. Creep rate increases rapidly with increasing temperature, while the fatigue strength decreases gradually with rising temperature. The creep rupture

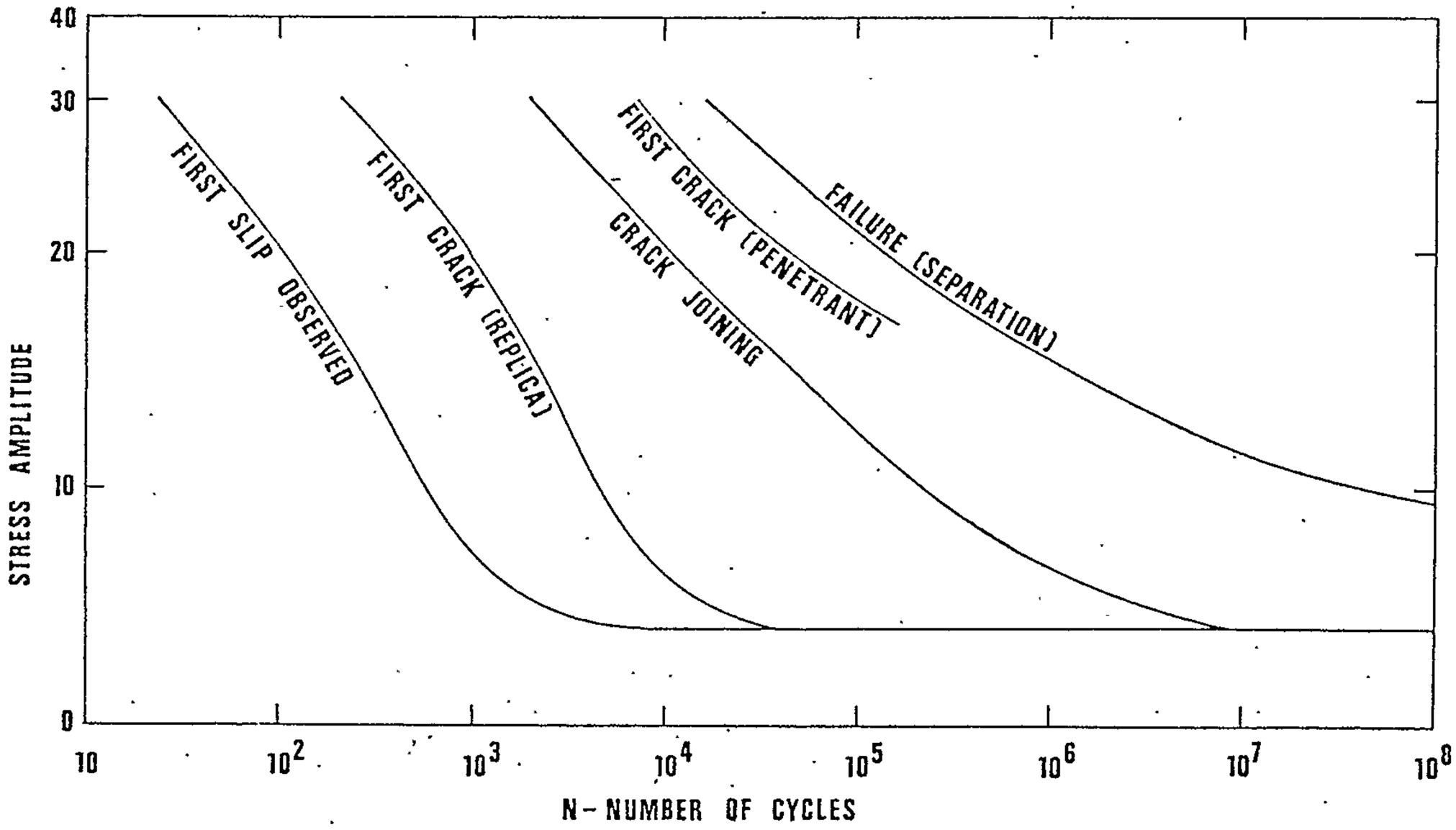

Fig. 3.2. Illustration of various interpretations of 'failure' in fatigue (after Bunshah, 1971, p. 278).

strength, which is the stress that a metal can withstand for a given time without breaking, decreases rapidly with increase in temperature to values which may be considerably lower than the fatigue strength. Consequently, the first requirement of metals or alloys at high temperatures is that they should withstand static loads, and heat resistant alloys have been developed primarily to give high creep strengths.

Chalmers (1959) described the characteristic appearance of fatigue failure. It contains two distinct zones; one is a smooth region in which the fatigue crack propagates outwards from its point of origin, often a stress concentration, while the remainder, usually of "granular" or "rough" appearance, is the part of failure which occurs when the fatigue crack has so reduced the section that the remaining material cannot sustain the load. The "fatigue" region often has concentric marks showing a series of positions at which the crack was stationary for some period.

Vaidyanathan and Schulman (1979) studied the in vitro deformation failure analysis of conventional and high copper dental amalgams at different strain rates, and the SEM was utilized to study the micro-structural features of amalgam fracture surfaces. The results show that there is a strong dependence of the dental amalgam failure mode on alloy composition,

particle size and shape at low strain rates. Significant plastic deformation of conventional amalgams at slow compressive loading indicates that dislocations may play an important role in the deformation of these amalgams. Continuous plastic deformation occurred in some specimens with no catastrophic failure even after a 30 per cent reduction in length. This would indicate that plastic deformation of γ_2 alone is inadequate to account for the extent of plastic flow observed. Mahler *et al.* (1977*a*) has correlated the extent of creep in dental amalgams to the grain size in γ_1. This would suggest that a similar correlation is possible between plastic flow, slow compressive loading and creep, because all are time dependent rheological properties. Two distinct types of failure resulted:

1) Catastrophic Failure through Crack Propagation

This type of failure occurred in the amalgams containing high levels of copper content at all the strain rates but only at the higher strain rates in the conventional silver-tin amalgams. The crack propagates primarily through the γ-γ_1 interface. In the case of Dispersalloy, fractures also propagated along the γ_1-AgCu eutectic interface.

2) Significant Plastic Deformation prior to Fracture or Continuous Plastic Deformation with No Sign of Catastrophic Failure

This mode was typical of conventional silver-tin

amalgams at low strain rates. The presence of voids in the matrix phases of γ_1 and γ_2 can result in stress concentration effects around these voids. Fracture propagation will then be energetically favoured to pass through these voids, as observed by Asgar and Sutfin (1965).

The present work should be considered an extension of the experiment performed by Giblin (1979), and Giblin and Wing (1979*a*, 1979*b*, 1980, 1981) who have studied the mode of failure of different types of amalgams. They observed the effects of static, sub-fracture loads on specimens prepared from amalgam alloys with a range of static creep characteristics. Specimens were prepared by hand condensation and by a single load application technique. One group of fully set specimens was tested on an Hounsfield Tensometer using a three-point loading system and the transverse strength determined. A further group of specimens was subjected to sustained three-point loading at loads below that necessary to produce transverse fracture in the Hounsfield Tensometer. The time necessary to produce fracture was determined under these conditions. From the investigation carried out, it appears that in the cases in which fracture occurs at "sub-fracture" loads, there is a correlation between the times of load and creep. Fracture time was inversely related to creep values. A low load applied for a long time on an amalgam with high creep will lead to *bending*

which in turn will lead to *crack initiation* and subsequent *fracture*. High creep amalgams tended to bend more than low creep amalgams before fracture. Low creep amalgams (creep less than 0.2 per cent), when subjected to low load fatigue will not undergo any significant plastic deformation and will not fracture. High creep amalgams (creep greater than 1 per cent), when subjected to low load fatigue will bend significantly, with crack initiation and fracture.

After testing, specimens were mounted, polished and etched, and the relationship of fracture to structure was observed using optical microscopy. It was found that the nature of interfacial fracture between original alloys and matrix depends on the rate of loading. Each fracture occurring in amalgams which has been subjected to low loads may be divided into a slow zone which has formed over almost the whole time required for fracture to occur, and a fast-zone, which has formed almost instantaneously to produce final failure of the amalgam specimen. In high creep amalgams, the slow zone is relatively long and the fast zone relatively short, whereas in the intermediate creep amalgams there is a relatively short slow zone and a longer fast zone. The nature of failure in the slow zone is primarily associated with a failure of the original alloy-matrix interface. In the fast zone, there is a greater tendency for failure to be through

original alloy particles rather than around particles. There is no relationship between the conventional mechanical properties of compressive, tensile and transverse strengths and fracture or bending of amalgams at "sub-fracture" transverse loads. There does, however, appear to be a relationship, for dental amalgam, between the rheological properties of *creep*, *bend* and *resistance to fracture*, at sub-fracture transverse loads, when tested for extended times. This may be a factor in the failure of clinical amalgams, which may be subjected to low loads, as well as bending relating to creep failure, particularly in marginal areas. In the great many clinical investigations reviewed, it was found there is almost universal agreement that low creep amalgams show less marginal breakdown than those with higher creep.

CHAPTER 4

CORROSION OF DENTAL AMALGAM

Effect of Corrosion on Mechanical Properties and Microstructure

Dental amalgam has a heterogeneous structure which consists of the amalgamation reaction products and unconsumed alloy particles. In view of the presence of numerous phases in the set amalgam, it seems possible that the destructive deterioration of the amalgam in the mouth may be the result of galvanic corrosion between these phases. It has generally been accepted that one of the major causes of failure of amalgam restorations is corrosion.

Wagner (1962) suggested that the corrosion process, although detrimental in some respects, may also have beneficial effects. He has credited the corrosion process with sealing of leaky margins and mechanical anchoring of the restoration to cavity walls by deposition of corrosion products. On the other hand, Schoonover and Souder (1941), and Jørgensen (1972) have blamed the corrosion process for loss of mechanical strength in the amalgam restorations.

Jørgensen (1965) presented the theoretical consideration of a corrosion mechanism in the marginal area of restorations. He claimed that the corrosion is responsible for the release of liquid mercury which

diffuses into the amalgam and gives rise to the mercuroscopic expansion and marginal upheaval. In a number of amalgam restorations which were examined by metallographic methods, it was found that the Sn-Hg (γ_2) phase is the most susceptible to corrosion, and that this phase forms a continuous network throughout the material.

Jørgensen and Saito (1970) have carried out very well controlled in vitro studies of the corrosion occurring during immersion of amalgam specimens in sodium chloride and sodium citrate solutions. It has been claimed that preferential dissolution of the tin-mercury phase occurs as a function of time in 10 per cent sodium citrate solution, and complete removal of the tin-mercury phase can be effected to depths of approximately 2.4 mm after 32 weeks. However, the reports of these studies present photomicrographs of unetched specimens, which appear to have an abnormally high proportion of the tin-mercury phase. This may be due to the type of specimen preparation used by these workers. Jørgensen and Saito claimed that their observations indicate, very strongly, a galvanic corrosion resulting from differences in concentration of oxygen on the surface of the specimens. In the corrosion process, the base surface of the specimens (with a relatively low oxygen concentration) forms the anode, while the side and top surfaces form the cathode.

The anode process is: $Sn \longrightarrow Sn^{++} + 2e$

while the cathode process must be: $2H_2O + O_2 + 4e^- \longrightarrow 4OH^-$

The combined corrosion process is thus: $2Sn + 2H_2O + O_2 \longrightarrow 2Sn^{++} + 4OH^-$

The mercury released by the anodic corrosion has been the subject of an earlier study (Jørgensen, 1965). This study has verified that porosity in an amalgam considerably accelerates corrosion of the amalgam, since porosity permits the penetration of electrolyte solutions into the amalgam, thus increasing the contact area between these solutions and the γ_2 phase. These authors suggested that a "wet" technique should be used in amalgam condensation and added that a reduction in porosity has a much greater effect on the strength of the amalgam than a high percentage reduction of the mercury content. In this study they claimed that different types of electrochemical process occur in sodium chloride and sodium citrate solutions.

Jørgensen (1972) showed the effect of selective total dissolution of the γ_2 phase in dental amalgams on their tensile strength. Specimens were stored in 10 per cent sodium citrate solution for 33 weeks before testing, using a diametral test method. The results of this study indicated that complete removal of the γ_2 phase resulted in a mean reduction of tensile strength of 16.4 per cent. The percentage reduction in strength is proportional to the mercury content of the set amalgam. This correlation tended to parallel the work of Otani

and Jørgensen (1967), who showed that the mercury content of hardened amalgam was directly proportional to the volume of the γ_2 phase.

Guthrow, Johnson and Lawless (1967) investigated the corrosion of the individual phases of dental amalgam in Ringers solution and artificial saliva by optical and electron microscopy and correlated the results with measured corrosion potentials and currents. They showed that the γ_2 phase (Sn_8Hg) had the most active potential and was observed to be the most severely attacked phase, the type of attack being general pitting. The γ phase (Ag_3Sn) had an almost neutral potential and observations showed little attack or deposition. There is little breakdown of the γ_1 phase (Ag_2Hg_3) and only deposition (or tarnishing) occurred in the Ag_2Hg_3 phase.

Mateer and Reitz (1970) examined the nature of the corrosion product formed on amalgam restorations. They carried out x-ray diffraction studies on scrapings obtained from the surfaces of teeth and also used optical microscopy on ground-polished sections through tooth and amalgam. This revealed two types of corrosive attack:

1) A bulk chemical conversion of amalgam to a two-phase structure that deposited, for the most part, as layers in the space between the restorations and the cavity floors and walls. The corrosion products

consist of Sn_2S_3 and SnO_3. The identification of the layers as oxide and sulphide is consistent with Jørgensen (1965), and Lyell, Barber and Massler (1964). The bulk chemical attack consumes both the γ_2 phase, and, to a lesser extent, the γ_1 phase. Original Ag_3Sn particles are resistant to direct attack but appear to react with released mercury. This is consistent with Jørgensen's hypothesis (1965) that mercury released by the corrosion process diffuses into the body of the amalgam and reacts with residual Ag_3Sn (γ) to yield Ag-Hg (γ_1) and Sn-Hg (γ_2).

2) A selective, penetrating crack propagation localized at margins and at notches in the surface of the restoration. Penetration is along the continuous γ_1 phase. This type of attack should probably be classified as corrosion fatigue. These cracks are clearly associated with margin breakdown and guttering in the specimen examined.

Unfortunately no control specimens were used and it is not certain that the voids present in amalgams which had been in service were due to condensation techniques or to dissolution of phases. These workers did not etch their specimens to reveal differences in the structural element present.

Stevenson (1973) studied the corrosion of dental amalgam. She has used similar methods to those

of Jørgensen and his co-workers, but has used metallographic etching techniques which allow the presence of the tin-mercury phase to be determined, and a distinction to be made between this phase and voids. Stevenson showed that corrosion of the tin-mercury phase in vitro was restricted to a depth of approximately 200 μm from the surface for properly condensed amalgams using hand condensation techniques. Greater depths of removal of the tin-mercury phase were possible only in specimens which were packed using very poor condensation techniques, leaving gross excesses of mercury. These techniques lead to the production of increased amounts of the tin-mercury phase and increased voids, and the possible formation of a continuous network of tin-mercury plus voids throughout the amalgam, as shown by Jørgensen and Saito (1970).

Stevenson's work appears to indicate that the storage of amalgam specimens prepared from conventional alloys or Dispersalloy in 1 per cent and 10 per cent sodium citrate solution for periods up to six months causes no significant reduction in the compressive, tensile or transverse strengths of these specimens, compared with the same properties of specimens stored in air. Reduction of the strength of amalgams prepared from an experimental alloy containing 70 per cent tin was highly significant following storage in sodium citrate. Stevenson's work has been supported by

information concerning weight changes of specimens during storage in chemical solutions, and microstructural observations following the passing of an electrical current through amalgam specimens. She also showed that the complete removal of the γ_2 phase does not occur clinically and, in fact, very little loss of this phase from the surface of amalgams appears to have taken place. It is possible, as postulated by Phillips (1973), that passivation occurs in the amalgam restoration because of tarnish.

Mateer and Reitz (1972) measured galvanic potentials of the amalgams in a clinical situation in order to determine corrosion mechanisms and their effects on structural changes and loss of strength in amalgams. The effect of corrosion on strength was assessed by micro-hardness tests on partially corroded restorations. These authors suggested that freshly placed amalgam restorations have adequate strength for prolonged service in the oral environment, but inadequate corrosion resistance. The corrosion interface progresses toward the centre of the restoration over a period of years and reduces hardness and embrittles the matrix structure. The tin ions migrate through the dentine, are deposited in elemental form, and appear as a black discolouration. The cavity liners apparently do not block this current flow. The liners offered no detectable hindrance to the ion transport.

Unfortunately, the microprobe technique used here does not distinguish between tin in the elemental, ionized, or chemically combined forms.

From the above discussion, it is clear that the corrosion of conventional amalgam is related to the tin-mercury (Sn_8Hg) phase and leads to marginal deterioration. The recent introduction of the "Dispersion Modified" amalgams by Innes and Youdelis (1963) and of ternary single melt high copper alloys (e.g. Sybraloy, Tytin) may significantly improve the longevity of amalgam restorations. Laboratory testing of corrosion (Greener, 1976) and mechanical properties (Malhotra and Asgar, 1978; Osborne and Gale, 1979) and a variety of clinical trials (Mahler *et al.*, 1973; Larson *et al.*, 1979) have shown that many amalgams rich in copper have superior characteristics when compared with conventional amalgams. The copper-rich amalgams contain little or no tin-mercury (γ_2) phase (Greener, 1976; Malhotra and Asgar, 1978). This suggests that deterioration mechanisms and phases formed during clinical use may be quite different for the copper-rich and conventional amalgams. Basically, the final structure of dispersion modified amalgams following a two-stage setting reaction is a mixture of the Ag_2Hg_3 (γ_1) matrix, Ag-Sn (γ) original alloy, Cu_3Sn from the original Ag_3Sn alloy, and a halo of the newly formed Cu-Sn (Cu_6Sn_5) phase surrounding the original Ag-Cu (eutectic) material. The final structure of the single

phase high copper ternary amalgams is a mixture of the original ternary alloy, a Ag-Hg (γ_1) matrix, Cu_6Sn_5 reaction zone and a reaction phase containing tin, copper, mercury and a very small amount of silver (Wing, 1979).

Marek and Hochman (1973), and Sarkar and Greener (1975) reported that the Cu-Sn reaction product (Cu_6Sn_5) is substantially susceptible to corrosion, the conclusions being reached on the basis of electrochemical tests on metallurgically prepared samples and observations of corrosion effects on amalgam surfaces.

Marek and Okabe (1978) studied the effect of crevice corrosion on the major structural phases of a high copper amalgam in vitro. Specimens were examined in a SEM before and after the exposures to a corrosion environment. The two phases of greatest interest, the Ag-Hg (γ_1) phase and the Cu_6Sn_5 phase, were prepared by plating mercury on polished tablets of the high copper, single composition alloy (Tytin, S.S. White, Dental Products International, Philadelphia).

This technique, developed for studies of the amalgamation reaction (Okabe *et al.*, 1975), allowed preparation of samples with well developed separate crystals in the reaction phases. The results showed that the Cu_6Sn_5 phase, which forms in the amalgamation reaction

of high copper dental amalgam alloys, exhibits good corrosion resistance, better than Ag_2Hg_3 (γ_1) phase containing tin. The observation is in conflict with previous reports (Marek and Hochman, 1973, and Sarkar and Greener, 1975). However, it is in general agreement with the report of Fairhurst *et al.* (1978) concerning polarization tests on high copper amalgam. The anodic polarization curves did not indicate the presence of phases highly susceptible to corrosion. It is possible that the reported data obtained on ingot specimens were affected by small amounts of a highly susceptible second phase, which is often difficult to eliminate completely.

Marek (1980) pointed out that there are large differences in corrosion resistance between amalgams of similar composition. Indeed, certain conventional alloys seem to display better corrosion resistant properties than some high copper alloys.

Marshall *et al.* (1980) investigated the deterioration of conventional and high copper amalgams after clinical use, by means of SEM/energy dispersive x-ray spectroscopy (which determines the distribution of elements and the composition of various phases). They demonstrated that there are at least two distinct types of compound which form in vivo in amalgam restoration: tin rich layers on the exposed surface of many amalgams and tin-chloride and smaller amounts of stannous oxide

products in the interior.

Conventional Alloys (Ag_3Sn)

Compositional changes were consistent with the presence of a γ_2 phase and resulted in the formation of a tin-oxygen-chlorine or tin-oxygen product. The volume of the tin-oxygen-chlorine product appears to exceed the normal amount of γ_2 phase, while the fraction of unreacted alloy particles (Ag_3Sn) was much lower than in fresh conventional amalgam. Corrosion induced changes were present throughout the entire restoration.

High-Copper Alloys

Near the surface of restoration, tin-rich and stannous-hydroxy-chloride deposits were found around the reaction zones of the residual Ag-Cu eutectic particle or in the Cu_6Sn_5 areas. This suggests that the Cu_6Sn_5 is electrochemically active. The copper is preferentially removed by the corrosion process. This result is supported by laboratory corrosion studies by Espevik (1977*b*), and Marek and Mahler (1979). In the system rich in copper, corrosion was confined to the surface and to a few isolated areas. The relatively small changes found in the system rich in copper could account for the superior marginal integrity reported for copper-rich amalgams in clinical trials.

Compositional Changes at the Tooth-Restoration Interface

In all conventional and high copper alloys, the interface layer extends around the tooth-amalgam interface and is rich in tin. Phosphorus and calcium have been identified in the corrosion products, both inside and outside amalgam restorations. In areas 20 to 30 μm from the margin exposed to oral cavity, these layers also contained sulphur. These results are in agreement with Wing (1974), and Holland and Asgar (1974). It is assumed that the corrosion of amalgam involves a two-way reaction between the restoration and the surrounding tooth in which interdiffusion of tin, calcium, and phosphorus takes place.

There is clear evidence that high copper alloys have better physical properties than conventional alloys and do not possess a γ_2 phase in the final structure; however they are not free from problems related to manipulative variables (Mahler and Adey, 1978). Careful manipulation of these materials is necessary to produce restorations with the optimum properties claimed by the manufacturer. The extent to which an amalgam is capable of resisting corrosion is of course one of its most important properties. In that our understanding of these processes is as yet incomplete, further research in this direction may prove to be of considerable significance.

ORIGINAL INVESTIGATION

CHAPTER I

THE SCOPE OF THE INVESTIGATION

Relationships of conventional properties of dental amalgam, particularly creep, and the behaviour of beams loaded at sub-fracture loads have been reported by Giblin (1979). Bend characteristics, crack propagation and fracture characteristics have been reported for amalgam beams 3 mm thick.

This present investigation aimed to test specimens of different thicknesses, and particularly 1 mm thick since this thickness has been used previously by some investigators in the transverse testing of dental amalgam.

It was a specific aim of the investigation to determine if the formula for modulus of rupture:

$$MR = \frac{3WL}{2bd^2}$$

would take account of beam thickness, and it was originally hoped that thinner specimens may allow testing to be carried out in a shorter time.

After preliminary investigations it became apparent that if possible true creep values for amalgam

beams should be ascertained and investigations allowing comparisons between cylindrical and beam specimens were carried out.

Microstructural determinations to quantitatively determine the phases present were carried out. This led to a study of differences within and between different amalgams to attempt to determine factors contributing to creep differences.

The research has involved the preparation of amalgam specimens from alloys of different composition and physical form. The major portion of the research has involved the application of sub-fracture loads to beam specimens 1 mm, 2 mm and 3 mm thick. Following testing, specimens have been subjected to detailed metallographic examination, principally using reflecting optical microscopy.

A limited study has been carried out on specimens which have been "corroded" in the laboratory.

CHAPTER II

EXPERIMENTAL PROCEDURES

1. Selection of Alloys

Three amalgam alloys have been used in this investigation, as seen in Table II.1. The amalgam alloys used in this study can be divided into three main categories. These were lathe-cut and spherical alloys, of the "Ag_3Sn" type, and an alloy containing a dispersed Ag-Cu spherical phase combined with a lathe-cut Ag_3Sn alloy.

2. The Preparation of Specimens

Specimens for Compressive Strength, Diametral Tensile Strength Tests and Creep

Specimens for these tests were prepared in the same mould cavity. The mould cavity produced cylindrical specimens with a diameter of 4 mm and a length of 8 mm.

The Mould

This consists essentially of an accurately ground hole in a cylindrical piece of steel, and a base with a post which fits closely inside the hole. Two spacers were used to establish the length of the specimen. In preparing specimens, an attempt is made to increase their homogeneity by using an 8 mm spacer and a 1 mm

Table II.1.

Amalgam alloys used in the investigation

Alloy	Manufacturer	Type of alloy	Composition
G & G Superfine	Englehard Industries Australia	Lathe-cut fine	$Ag_3Sn(Cu_3Sn)$
Hi Atomic	G.C. Chemical Co., Japan	Spherical	$Ag_3Sn(Cu)$
Dispersalloy	Johnson & Johnson, U.S.A.	Dispersion Modified Lathe (*L*) + Spherical (*S*)	*L*: $Ag_3Sn(Cu_3Sn)$ *S*: Ag-Cu eutectic

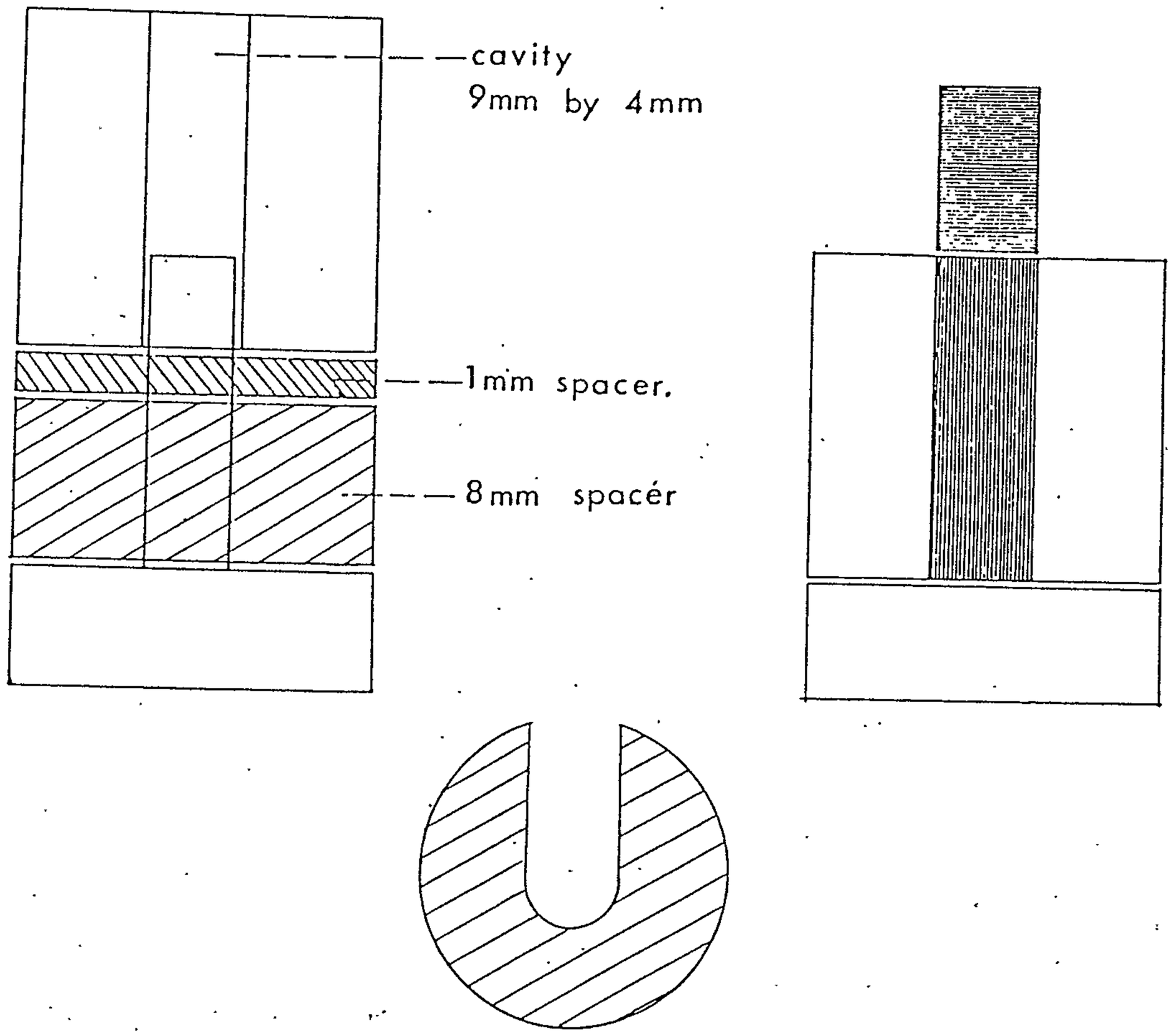

Fig. II.1. Diagrammatic representation of mould used for preparation of cylindrical amalgam specimens for compressive strength, diametral compression strength and creep tests (after Giblin, 1979).

spacer to provide a cavity which is initially 9 mm long. Immediately following condensation, the 1 mm spacer was removed and the specimen extruded 1 mm to allow carving with a sharp razor blade. This top layer represents the mercury rich excess amalgam. The amalgam specimen may then be ejected from the mould by removing the remaining spacer and pushing the base to eject the specimen. The mould is as shown in Fig. II.1.

Manipulation of Amalgam Alloys

a) Proportion of Mercury to Alloy

The ratios of alloy to mercury and the initial mercury content for each alloy are listed in Table II.2.

b) Trituration

A Silamat ultra-speed amalgamator was used to achieve optimal trituration in the shortest possible time. The machine had a throw of 60 mm and a rate of oscillation of 4,500 rpm when loaded with amalgam alloy and mercury. The time of trituration was as recommended by the manufacturer. The advantage of this amalgamator was that multiple mixes could be achieved in a short time, which minimises the risk of layering of the amalgam.

c) Condensation

Condensation was carried out using hand pressure, using either a 1 mm, 2 mm, or a 3.5 mm diameter smooth

Table II.2.

Specifications for amalgam alloy manipulation

Alloy	Presentation Form	Alloy : Hg ratio by weight*	Method of trituration	Trituration time in Silamat
G & G Superfine	Bulk powder	10:10	Dispersalloy plastic capsule with 1 gm cylindrical metal pestle	7 secs.
Hi Atomic	Bulk powder	10:7	Dispersalloy plastic capsule with 1 gm cylindrical metal pestle	7 secs.
Dispersalloy	Tablet 300 gm	10:10.4	Dispersalloy plastic capsule with 1 gm cylindrical metal pestle	8 secs.

* The alloy : mercury ratios are taken from the manufacturer's recommendations.

faced plugger, depending on the alloy type. For the lathe-cut (G & G Superfine) and dispersion modified alloys (Dispersalloy), the 1 mm and 2 mm diameter condensers were used alternately. The 2 mm and 3.5 mm condensers were used for the spherical alloy (Hi Atomic). Cylindrical 8 x 4 mm specimens were prepared using a standardized hand condensation technique of 8 increments, 25 thrusts of 25 N per increment with no precondensation mercury removal. Specimens were stored for 7 days at 37°C before testing.

Specimen Preparation for Static Creep Tests

Specimens were prepared in the same mould as were the compressive strength and diametral tensile strength test specimens. An 8 x 4 mm cylindrical specimen was used for testing. Specimens for the static creep test were divided into 5 groups according to the method of condensation. The condensation effectiveness was altered by varying the size and number of increments from 8 to 1 with a total of 200 thrusts of the condenser being used for the specimen as a whole. It was shown by Wing (1965) that condensation effectiveness related closely to the size and number of increment used in amalgam condensation. However, the initial ratio of mercury to alloy and the trituration time were constant for each specimen. Condensation load was maintained constant for all creep specimens prepared by hand condensation.

Type I specimen - The triturated amalgam mass was divided into 8 increments, and was hand condensed one increment at a time with excess mercury being removed following the condensation of each increment. Twenty-five thrusts of approximately 25 N on a smooth faced circular plugger appropriate for the amalgam type were used per increment, with a total condensation time of 3 minutes for the specimen.

Type II specimen - The triturated amalgam mass was divided into four increments and condensed one increment at a time, with excess mercury being removed following the condensation of each increment. Fifty thrusts of approximately 25 N were used per increment.

Type III specimen - The triturated amalgam mass was divided into two increments. One hundred thrusts of approximately 25 N were used per increment.

Type IV specimen - The triturated amalgam mass was packed into the mould in one increment. Two hundred thrusts of approximately 25 N were used per increment.

Type V specimen - Cylindrical specimens were prepared using a 'one shot' packing technique with a load of 56.5 MPa applied on a 4 mm condensor for 60 seconds.

All specimens were stored at 37^{o}C, 100 per cent humidity for 7 days before testing.

Specimen Preparation for Modulus of Rupture (Transverse Strength) and Sub-Modulus of Rupture Transverse Tests

A specimen in the form of a rectangular prism was used for modulus of rupture and sub-modulus of rupture transverse tests. Three types of specimen were prepared, with dimensions 12 x 10 x 3 mm, 12 x 10 x 2 mm, and 12 x 10 x 1 mm. The specimens were prepared by mechanical condensation using 56.5 MPa pressure. For the sake of description, these will be called type VI, type VII, and type VIII specimens respectively.

The mould

Amalgam specimens were prepared using a five-piece mould fabricated from steel with internal dimensions of 3 x 10 x 12 mm. The mould was similar to the mould used by Peet (1971), Adair (1971), and Giblin (1979). The sides and ends of these pieces can be located accurately by ledges cut into the base section of the mould. The five pieces were fixed firmly in a metal holder by four clamping bolts. One or two separate pieces of steel with dimensions of 1 x 10 x 12 mm are fixed in the mould as spacers when the specimens of type VII or type VIII are to be made.

Dispersalloy, G & G Superfine, and Hi Atomic alloys were used for testing. These alloys were used according to the manufacturer's recommendations. Three

dispensations of alloy and mercury were used per trituration in a Silamat ultra-speed amalgamator. One, two or three triturations were used per specimen, depending on the thickness of the specimen to be prepared.

Condensation

The first capsule load of triturated amalgam was placed into the mould, then tamped down with a rectangular brass packer measuring 11 x 9 mm, followed by the second and third portions in the case of type VII and type VI specimens. This allowed the total amalgam mass to be placed and tamped into the mould within 45-60 seconds. The mould, filled with amalgam, was then placed between the platens of the Hounsfield Tensometer and the machine hand wound to 1,500 pounds force within 15 to 45 seconds, and held for one minute. The load produced a packing pressure of 56.5 MPa (8,000 psi). The mould was removed and the excess amalgam was carved away with a sharp blade. This method of condensation gave consistent results among specimens of the same type; large specimens can be prepared quickly.

Specimen Preparation for Corrosion Test

A specimen in the form of a rectangular prism, 12 x 10 x 3 mm, was used for the corrosion test. The specimens prepared were type VI specimens. The specimens were stored for 7 days after preparation, then immersed

in 1 per cent or 10 per cent sodium citrate solution for a period of 3 months at 37°C before testing. The solution was changed regularly during the test.

3. Testing Methods

Compressive Strength

This test was performed on fully set specimens seven days after preparation. A cylindrical 8 x 4 mm specimen was used. The specimen was placed lengthwise between the platens of a Hounsfield Tensometer*. This unit was mechanically driven, and a cross-head speed of 0.4 mm per minute was used. A series of three specimens was tested to fracture, and the compressive load for fracture was determined in pounds force. The compressive strength was calculated from the force at which the specimen was fractured in megapascals.

Tensile (Diametral Compression) Strength

This test was carried out on fully set, cylindrical, 8 x 4 mm specimens, which were prepared in a similar manner to the compressive strength test specimens. The specimen was placed sideways between the platens of the Hounsfield Tensometer with a sheet of card paper as a cushion between the specimen and platens. A cross-head

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*Hounsfield Tensometer, Hounsfield Tensometer Ltd., 81 Morland Road, Croydon, Surrey, England.

speed of 0.4 mm/min. was used until crack initiation was perceived visibly or audibly. From the load required to produce fracture, the tensile strength was calculated using the formula:

$$T.S. = \frac{2W}{\pi dt}$$

where:

$T.S.$ = tensile strength;

W = fracture load;

π = 3.14159;

d = specimen diameter;

t = thickness.

A mean of three values for the tensile strength was determined in megapascals.

Static Creep

This test was performed with 8 x 4 mm cylindrical specimens (Type I, II, III, IV, V specimens) seven days after preparation. At the time of testing, the ends of the specimen were planed and the specimen was subjected to a pressure of 36.2 MPa (5,250 psi) for four hours. Measurements were taken at one and four hours after the start of the test. A mean of three values for static creep was determined for each type of specimen. The creep is the percentage change in length occurring between one and

four hours. These specimens were tested at 37 ± 0.2°C.

Modulus of Rupture (Transverse Strength)

This test was carried out by applying a three-point loading system developed by Giblin (1979) to a fully set rectangular prism specimen (Type VI, VII, VIII specimens). The support block and specimen were aligned in the Hounsfield Tensometer and the machine was driven at a cross-head speed of 0.4 mm/min. The load was increased until fracture of the specimen occurred. The modulus of rupture was calculated using the formula:

$$MR = \frac{1.5WL}{BD^2}$$

where:

MR = modulus of rupture;

W = fracture load;

L = span of supports;

B = width of test piece;

D = thickness of test piece.

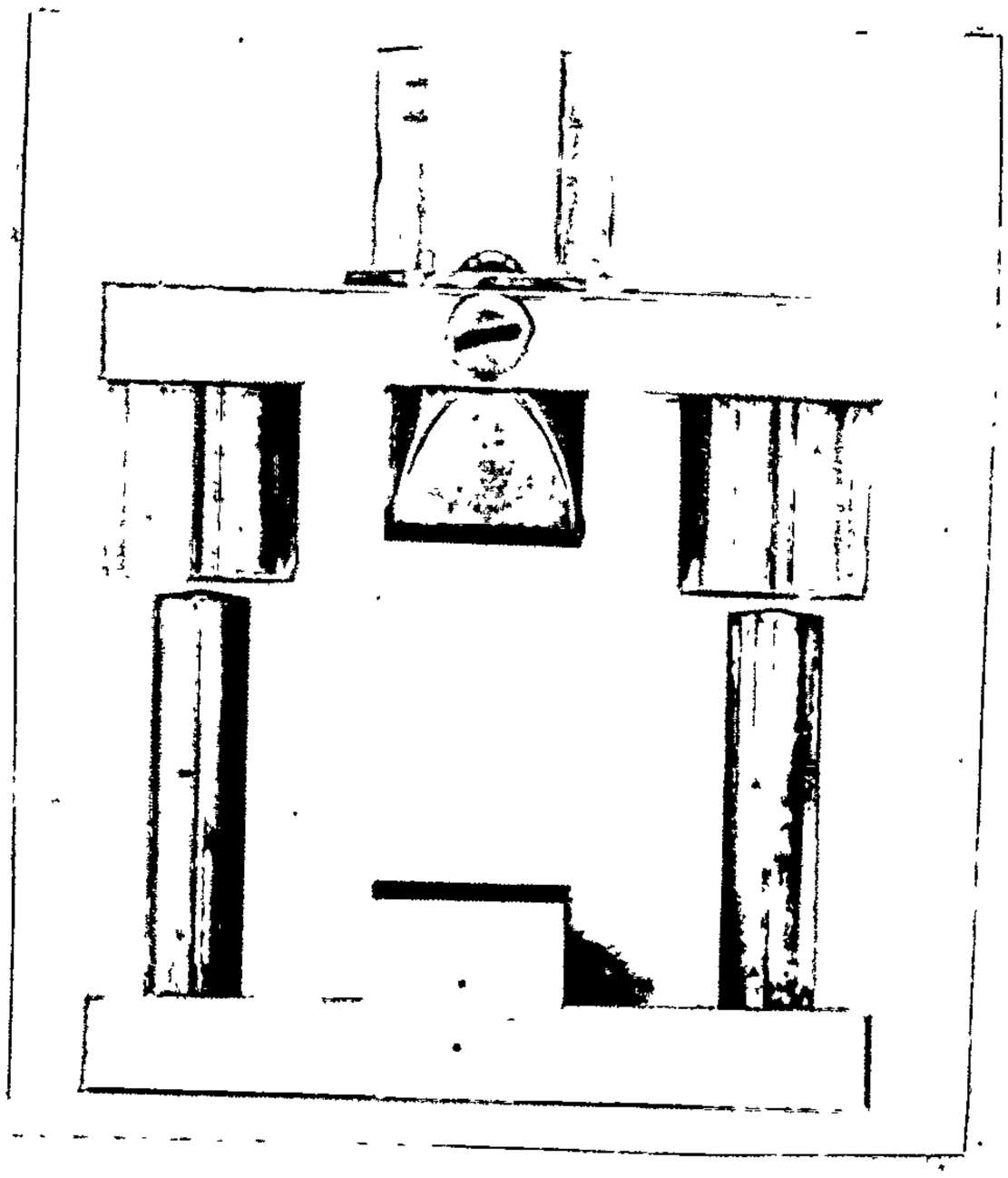
A mean of three values for each type of specimen (Type VI, VII, VIII specimen) was determined. Calculation of Modulus of Rupture was based on the actual dimensions of the specimens tested.

Testing Jig

This consisted of two sections similar to those used by Giblin (1979): one containing the loader, the other containing the load-bearers (see Fig. II.2).

The loader is a cylindrical bar (2.3 mm diameter) set parallel to the load bearers and attachable to either the arm of the static fatigue machine or to one head of the Hounsfield Tensometer. This loader section was set in a brass plate (25.4 x 50.8 x 6.3 mm) similar to the base of the bearer section. A locating screw was placed so as to impinge on the machine loader, thereby locking it into place, parallel to the load bearers in the base section. The loading bar was set on top of the specimen between the bearer rods. Set in the loader plate section were two brass cylinders, milled precisely to receive the capstan pillars of the bearer section. These cylinders were machined to size and press-fitted into the brass loader block (25.4 x 50.8 x 6.3 mm) to provide accurate and reproducible alignment between the loader and load bearers.

The load bearers, two cylindrical sections of hardened steel (2.3 mm diameter) set parallel and with their centres 8 mm apart, were located in a brass block of dimension 12.1 x 7.5 x 25.4 mm. This brass block was then set in another brass block of dimensions 25.4 x 50.8 x 6.3 mm which was machined on the top surface to a depth of 1 x 12.1 x 25.4 mm to receive the bearer block which was then dowelled and screwed into place. At either end of the larger brass



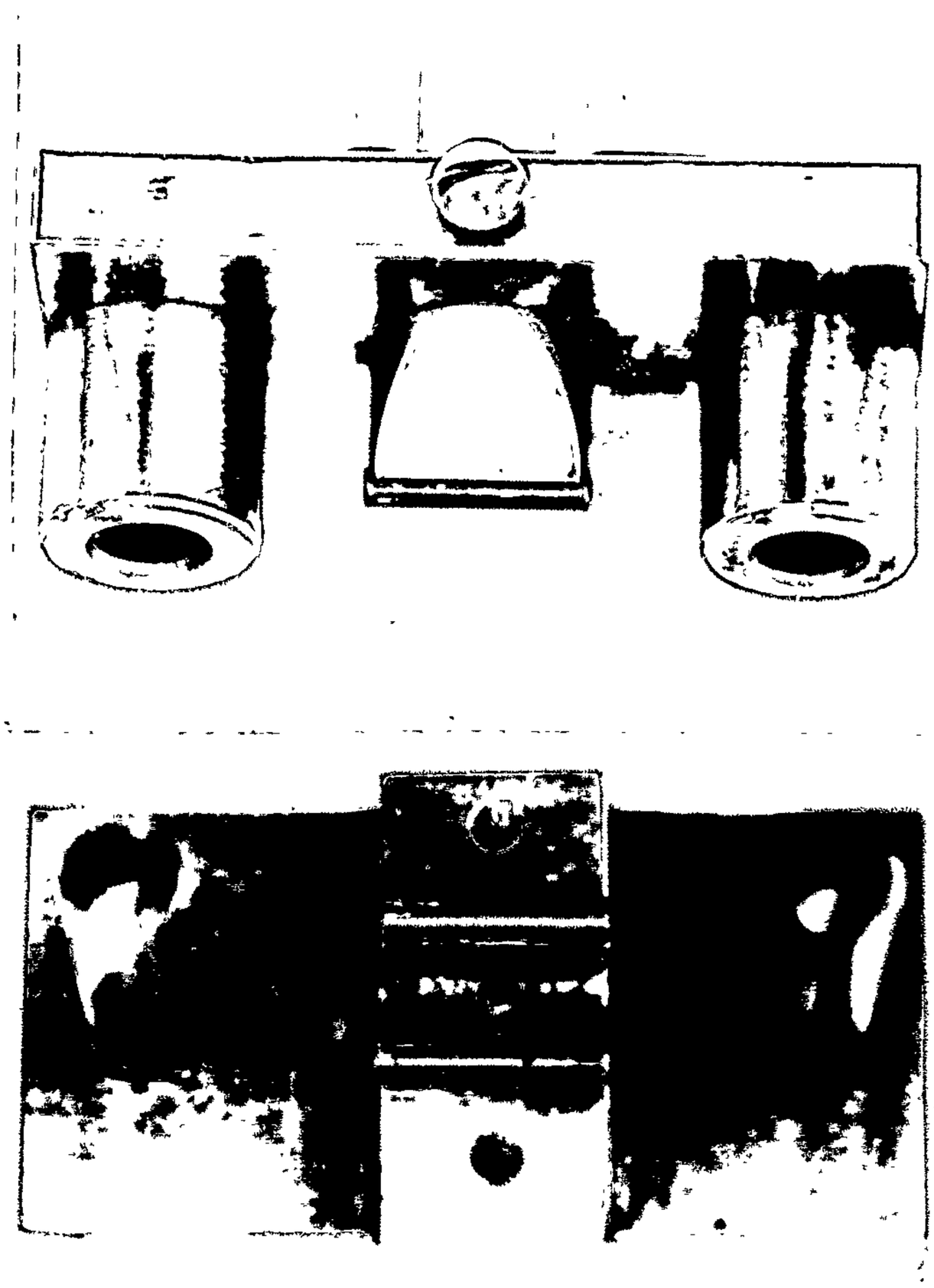


Fig. II.2. Design of apparatus for transverse testing of amalgams (after Giblin, 1979).

base section (25.4 x 50.8 x 6.3 mm) were set two parallel stainless steel capstan pillars (6.3 mm diameter).

With this system (see Fig. II.3), the loader was always centred, the loader and bearer rods were parallel, and the same unit could be used for specimen testing in either the Hounsfield Tensometer or the static fatigue machine. This eliminates variability, which may occur if separate units are used in the two machines. Specimen placement in the unit was simple, being facilitated by the sliding action provided by the capstan pillars of the loader and bearer sections. A simple right-angled brass section was cut to size to align the specimen on the load-bearing bars, before being locked in place by the sliding together of the loader and bearer sections. This transverse testing unit gave accurate results, with a decrease in the scatter of fracture loads.

Sub-Modulus of Rupture

This test used the same types of specimen (Type VI, VII, VIII specimen) and testing jig as for the modulus of rupture determinations. The Sub-Modulus of Rupture test was carried out as follows:

1) The modulus of rupture was evaluated in order to determine the mean fracture load of three examples of each type of specimen (Type VI, VII, VIII). The

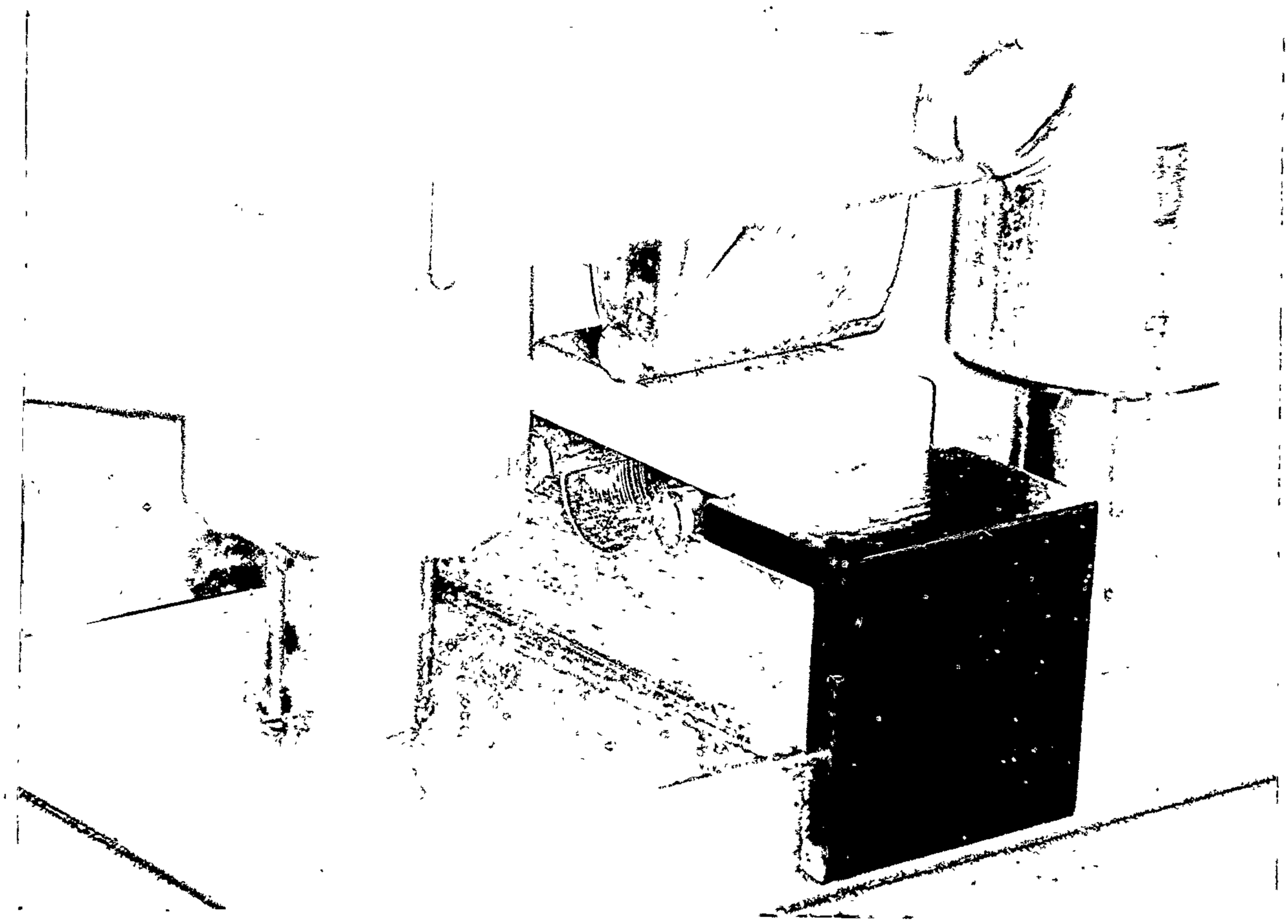


Fig. II.3. Complete assembly for transverse testing of amalgam showing rectangular prism specimen located on the support block with aligning template and cylindrical loader in place (after Giblin, 1979).

modulus of rupture was calculated using the formula as before:

Modulus of Rupture - Determined

Size of specimens - 12 x 10 x 3 mm, Type VI

- 12 x 10 x 2 mm, Type VII

- 12 x 10 x 1 mm, Type VIII

Distance between load bearing bars - 8 mm.

2) The load 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 the following factors:

Force exerted by loading arm at point of load application - 8.72 kg

Mechanical advantage of added load - 12:1.

3) The selected percentages of fracture loads were applied to specimens in the static fatigue machine and the specimens observed. Percentages of 90%, 80%, 60%, 40% and 20% of the fracture loads were used. It was decided to set a five-week limit on the testing of any one specimen. Specimens which had not fractured in five weeks were known to be unlikely to fracture or to fracture only after many months. A mean of three values was determined.

The Static Fatigue Machine

The machine (see Fig. II.4) was designed by Peet (1971) and built by Chapman and Goldsmith Pty. Ltd.* The framework of the machine was fabricated from 25.4 x 3.2 mm angle steel and had a base area of approximately 0.5 m^2. The length of each loading arm from the centre of the pivot to the centre of the hole for the weight carrier was 91.44 cm. Five holes were provided in each arm to allow variation of mechanical advantage, and hence the load applied to the specimen. A ratio of 12:1 was selected for all experiments conducted. Specimens were loaded by adding weights to a weight carrier, manufactured to Peet's specifications (1971) by A. James Wedderburn.** Incremental loads were added by the use of lead shot placed in a bottle; the bottle was then attached to the weight carrier to ensure exact loading at required percentages of modulus of rupture fracture forces.

To record the time at which the specimen fractured, an electric clock was wired to a microswitch positioned on the machine's frame. Both the switch and a stop screw were adjusted so that, on specimen failure, the arm would fall to stop the clock, recording the day and time of fracture, a.m. or p.m. This enables the correct determination of

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* Chapman and Goldsmith Pty. Ltd., Engineers, Goulburn Street, Sydney.

** A. James Wedderburn Scale Manufacturers, 74 Liverpool Street, Sydney.

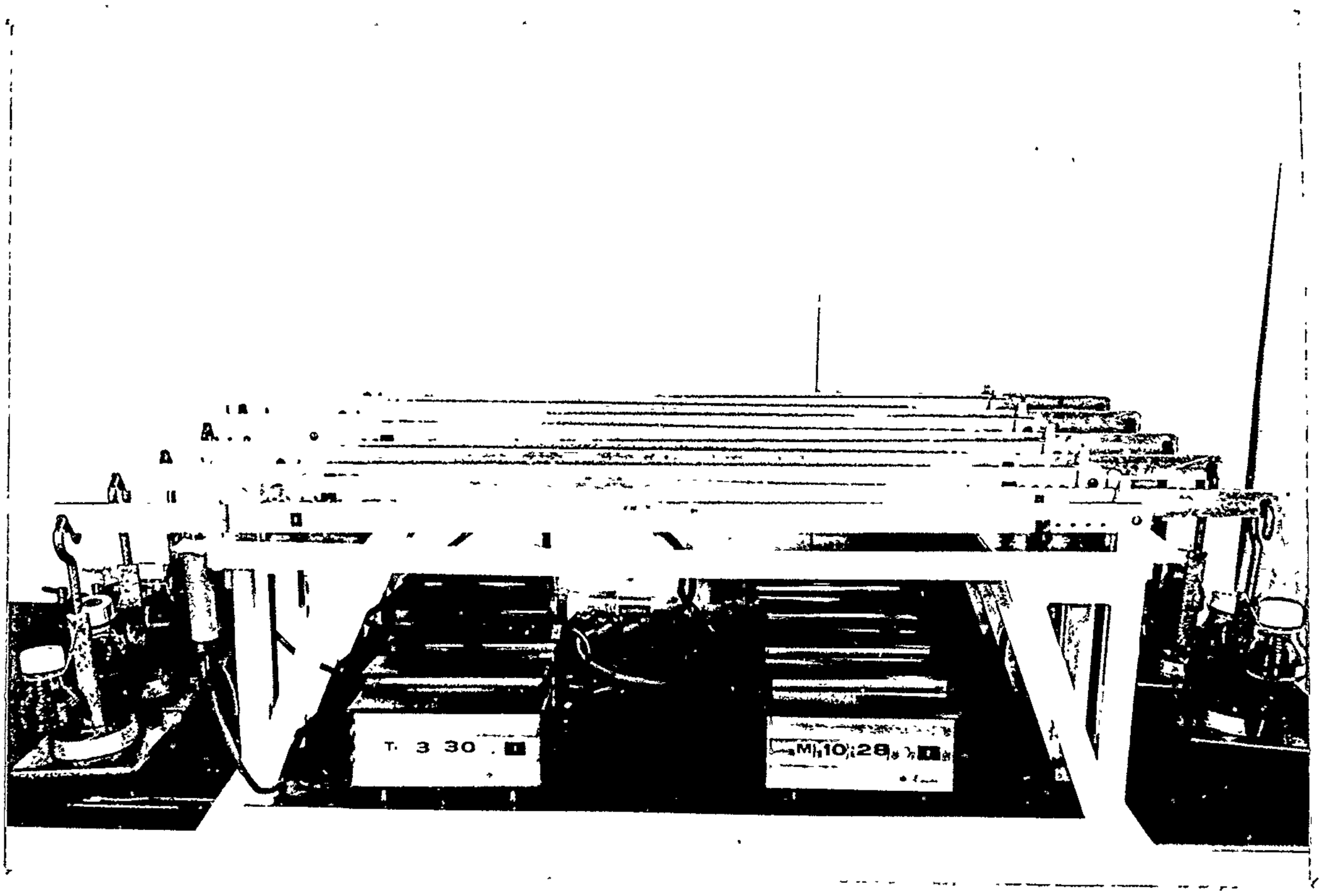


Fig. II.4. Static fatigue machine for testing amalgam specimens at "sub-fracture" loads (after Giblin, 1979).

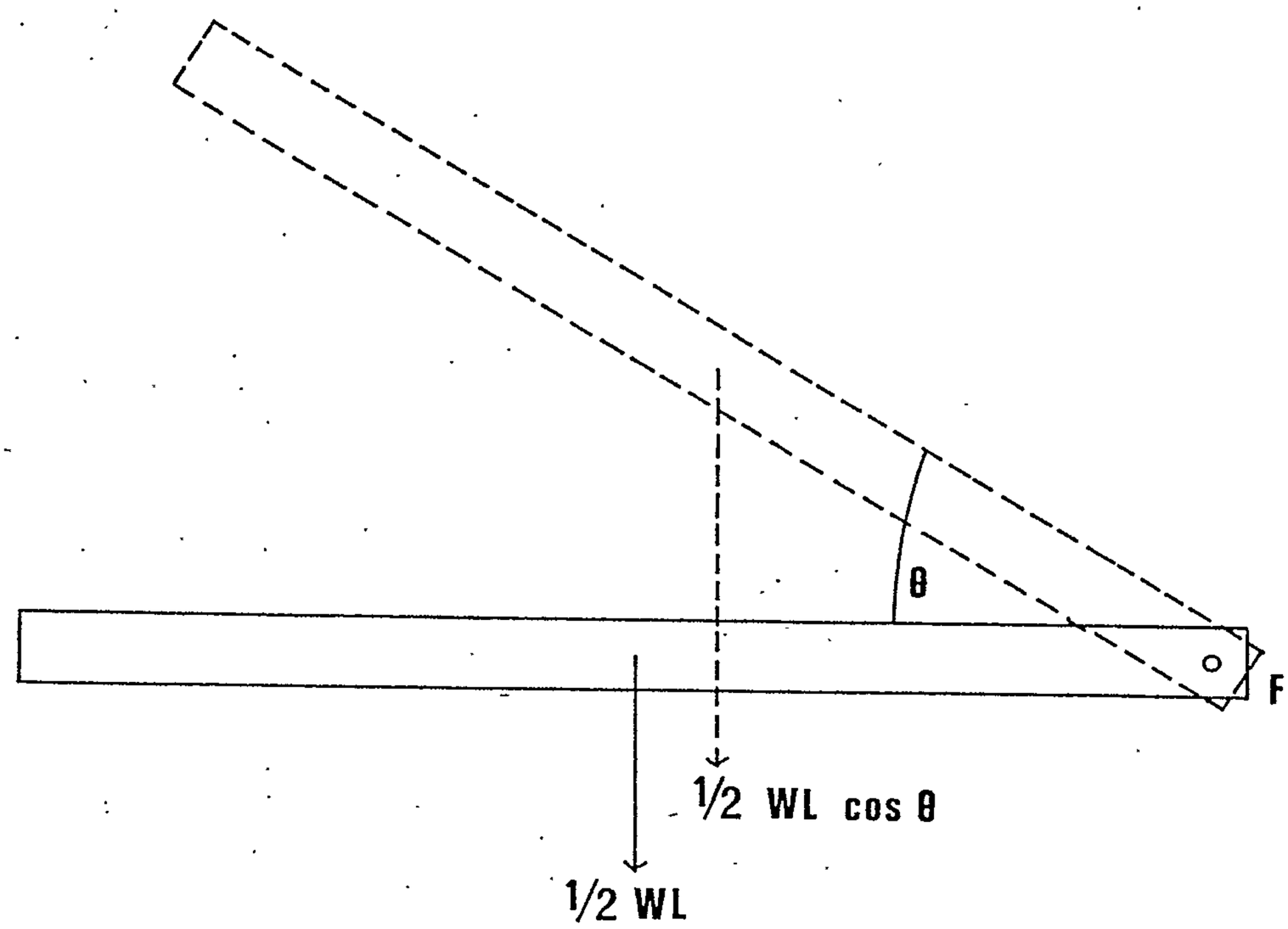
the time of fracture, without the need for constant supervision.

To load the static fatigue machine, the transverse testing unit, complete with specimen, was placed under the loading arm and fixed by a split pin. The load corresponding to the appropriate percentage (90%, 80%, 60%, 40%, 20%, 10%) of the modulus of rupture was set on the end of the loading arm. The timing clock was then set to the time of the day at which the test began. If no fracture occurred on loading, the specimen would only fracture at some later time which was appropriate for that alloy and that percentage load.

For the purposes of analysis, it is essential that all forces acting on the beam should be perpendicular to its length. Otherwise, for example, the moment of the weight of the beam will be $\frac{1}{2}WL\ cos\theta$ (see Fig. II.5) rather than $\frac{1}{2}WL$, and this would necessitate a measurement of the angle θ. However, variations in the thickness of the specimens used tend to alter the position of the beam. Thus it was necessary to counteract this by inserting flat pieces of metal of the appropriate thickness (1 mm and 2 mm thickness in Type VII and Type VIII specimen respectively) under the support.

Bending Characteristic Analysis

Following fracture of the specimens in the transverse fatigue testing machine, specimens were



where: W = weight of beam;

L = length of beam;

θ = angle of elevation of beam.

Fig. II.5. Diagram illustrating moments of horizontal and non-horizontal beam.

relocated and cemented with a cyano-acrylate cement* to allow for an evaluation of the degree of pre-fracture bending, which was ranked between one and ten. A value of one was given to an untested specimen, and ten to the specimen exhibiting the most bending in a manner similar to that of Giblin (1979).

Corrosion Testing

Transverse strength testing was carried out with specimens which had been immersed in 1 per cent or 10 per cent sodium citrate solutions for a period of 3 months. A mean of three values for the transverse strength when the specimens were immersed in 1 per cent or 10 per cent sodium citrate solutions for a period of 3 months was compared with the transverse strength when the specimens were stored in air. The effect of loss of the γ_2 phase when the specimens were immersed in 1 per cent or 10 per cent sodium citrate solutions for 3 months was studied.

Microstructural Preparation

Selected cylindrical specimens from creep tests and reassembled specimens from transverse static fatigue tests and rectangular specimens from corrosion tests were embedded in cavities prepared in Lucite rods, using a

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* Selleys Supra Glue, Japan

minimal amount of autopolymerizing acrylic resin. Full mounts of acrylic resin or other quickly setting resins are contra-indicated for mounting amalgam specimens because of the possibility of the heat generated causing the liberation of mercury from the amalgam. This method was described by Wing (1965*a*). Use was made of the metallographic polishing technique with silicon carbide abrasive papers (200, 440, and 600) on a flat glass bed with a stream of constantly running water. The water acts as a coolant, a lubricant, and prevents clogging of the papers. The silicon carbide abrasive papers were used to abrade the specimen and lucite to a flat surface. Following this, the specimens were polished on a rotating motor driven pad, using a selection of diamond pastes (15, 6 and 1 microns) with a non-volatile lubricant such as equal parts of propylene glycol and water. The advantage offered by the use of diamond abrasives is that they cut rapidly and so tend to cut off the surface layer rather than causing deformation and flow. Etching has been carried out using a double etch technique (as described by Wing, 1961, 1965*a*). The first solution was an aqueous solution of 2 grams of potassium cyanide, 1 gram of iodine crystals in 25 cc of water. The second solution was an aqueous solution of 2 grams of potassium ferricyanide, 20 cc of water, 2 cc of ammonia and a further 18 cc of water. Polished specimens were etched by first wiping in the first solution for four to five seconds with cotton wool. The specimen was then washed

in water and immersed in the second solution for 4 to 5 seconds. Following a further washing in water, the specimen was swabbed well with alcohol, and dried with compressed air. This technique of metallographic polishing produces a surface free from distortion and remarkably scratch free. The "cyanide" etch has the ability to demonstrate the grain structure of the formed amalgam as well as to maintain the surface free from polishing artefacts. Therefore, it is possible to distinguish original alloy particles embedded in a crystalline matrix. The etched and unetched specimens were then viewed on a Neophot II*, metallographic optical light microscope. In unetched specimens, dark areas of porosity related to the technique of amalgam manipulation have been observed. In etched specimens, a microscopic analysis was carried out to identify the phases of amalgam. The quantitative determination of these phases was made by a point counting method (Hilliard and Cahn, 1961, and Hilliard, 1961). In this technique, a regular array of points is superimposed on the microsection and the number of points which fall over each phase is counted. A regular array of 25 points was superimposed on the screen of the microscope; the magnification was 200X for porosity counts and 600X for phase identification counts. In each cylindrical specimen, four different high porosity areas, four different low porosity areas, and four different overall areas were used

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* Carl Zeiss, Jena, East Germany.

for counting. Therefore, three hundred points were counted per specimen. This method offers advantages in that large numbers of fields may be counted easily and accurately. It has been claimed by Otani and Jørgensen (1967), and Young, Wilsdorf and Paffenbarger (1973) to be a reliable method. The volume fraction of each phase is equal to the number of points falling on the phase divided by the total number of points counted. The percentage volumes of these phases, and of the porosity were computed. Etched re-assembled rectangular specimens were viewed on an optical light microscope to determine the nature of crack initiation and passage of the fracture within the specimens.

Data Analysis

Statistical analysis was carried out using a Student *t*-test to test the significance of the difference between means. Differences were regarded as statistically significant when a 98 per cent level of confidence was achieved.