TRANSVERSE, STATIC FATIGUE
OF DENTAL AMALGAM

A Thesis submitted to the University of Sydney in support of my candidature for the Degree of Master of Dental Surgery. (1979)

John P. Giblin, B.D.S.
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REVIEW OF THE LITERATURE

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ORIGINAL INVESTIGATION

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BIBLIOGRAPHY
ACKNOWLEDGEMENTS

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I would also like to thank Mr. Ken Tyler, for his technical expertise in the design and fabrication of the testing units used in this investigation.

To Miss Moira Poynter, I am most grateful for assistance in the typing of this thesis.
INTRODUCTION

Research on dental amalgam and a wide range of alloys has occupied a considerable proportion of the dental literature in the fields of Operative Dentistry and Dental Materials, particularly since the late nineteenth century.

Essential work on the physical properties of amalgam was carried out in the early decades of this century and led to the promulgation of the first Specification for this material in 1934, an important guideline to both manufacturers and clinicians.

The development of improved techniques for polishing and etching amalgam specimens in the 1960’s regenerated interest in basic research on dental amalgam and the advent of the scanning electron microscope and electron probe microanalyser has added further stimulus to the efforts of workers in this field.

Coupled with these research advances, the development of the spherical particle, dispersion-modified and, more recently, the high copper ternary amalgams, has maintained a keen interest in this material both at a research and a clinical level with the aim of coming closer to the ideal clinical restorative.
The Modulus of Rupture or Transverse Strength of dental amalgam is a composite strength. A review of the literature indicates minimal work on this strength test compared with other strength tests. Recent work by Forsten has increased interest in this test and its effect on various amalgam alloys.

In the last decade a great deal of interest has centered on the creep tests for amalgam as a predictor of clinical behaviour. This test uses compressive loads very much below those necessary to fracture amalgam.

The present study was undertaken in an attempt to investigate the behaviour of different types of amalgam alloys subjected to low transverse loads, to evaluate bend characteristics of tested amalgams and to investigate the microstructure of the fractured surfaces of selected specimens following failure, in an effort to find possible causes of failure of amalgam.

In reviewing the literature pertinent to Modulus of Rupture (transverse strength test) testing generally, a detailed review of all the basic studies has not been attempted. Essential information has been reviewed principally from a number of authoritative works where large bibliographies may be found.
Review Of The Literature
CHAPTER 1

AMALGAM FAILURES

Failure of amalgam restorations has been a prime concern of dental practitioners from the earliest days of the use of dental amalgam. In the earliest times "charlatans", such as the Crawcour brothers, caused problems for their patients and ethical practitioners because of their improper use of this material with improper cavity preparation and manipulative techniques (Sweet, 1959).

Following the "Amalgam War" (1841-1850), workers began to study amalgam scientifically with the aim of improving restorations and reducing failures. The most significant investigator following a number of eminent dentists in the United States and United Kingdom, was G. V. Black (1895, 1896) who gave us fundamental knowledge concerning composition, manufacturing, testing of physical properties and manipulation of dental amalgam.

Following World War I, an investigation was begun in 1919 at the National Bureau of Standards (Washington, D.C.) because of concern by the United States Army at the failures of amalgam restorations. This investigation led to the ultimate promulgation of the American Dental Association
Specification No. 1 for Dental Amalgam, published in 1934. This specification, putting a premium on chemical composition, dimensional change, ultimate compressive strength and flow, was the first reliable guide to members of the profession to help them in selecting satisfactory alloys. This specification also led the way to scientific testing of dental amalgams by other workers and was followed by studies of the causes of failure of amalgam restorations.

Brekhus and Armstrong (1936) surveyed 418,152 teeth, 70% of which carried amalgam restorations, and found that a lower percentage of amalgams needed replacement than any other type of filling.

Crawford (1938) listed amalgam failures as being due to: recurrent caries, overhanging gingival margins, protruding margins on axial surfaces, broken fillings, lack of properly carved occlusal and proximal surfaces and discolouration. Crawford also stated that these faults could be caused by flaws in the physical or chemical properties of the amalgams, but were more likely to be due to inadequate cavity preparation, or condensation and lack of operator skill. He wrote, "Careful operators are securing excellent results with amalgam fillings".

Healey and Phillips (1949) examined 1,521 defective amalgam restorations in an attempt to analyse the causes of failure. Failure was generally due to fracture,
dimensional change, recurrent caries and pulp or periodontal involvement. The aim of these workers was to classify each failed restoration according to the causative factor contributing to failure. Their findings are summarised in Table 1. (p.4).

These workers found that 56% of all failures could be attributed to improper cavity preparation, particularly inadequate proximal extension, while 40% were due to faulty manipulation or contamination of the amalgam. Although not stated, one might infer that some of the fractures were due to complex wedging, shear or tensile forces.

Healey and Phillips (1949) agreed with the findings of Crawford (1938) and Easton (1941) that careful cavity preparation, proper manipulation, and restoration of normal anatomical contour can reduce amalgam failures markedly.

Wolcott (1958) noted that while it is essential to follow the well established cardinal principles to produce a sound restoration, it is also necessary to realise the importance of the caries susceptibility of the patient. Attention was drawn to marginal "ditching", which occurs with time under masticatory forces because of certain "intrinsic weaknesses" of the amalgam in conjunction with poor finishing of the cavo-surface margin.
Table 1: **TYPES OF FAILURES OBSERVED AND THE CAUSATIVE FACTORS CONTRIBUTING TO THEM**

<table>
<thead>
<tr>
<th>CAUSE OF FAILURE</th>
<th>NUMBER OF FILLINGS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fracture</td>
<td></td>
</tr>
<tr>
<td>Faulty manipulation</td>
<td>325</td>
</tr>
<tr>
<td>Cavity preparation</td>
<td>66</td>
</tr>
<tr>
<td>Traumatic occlusion</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>398</td>
</tr>
<tr>
<td>Dimensional change</td>
<td></td>
</tr>
<tr>
<td>Expansion</td>
<td>253</td>
</tr>
<tr>
<td>Contraction</td>
<td>38</td>
</tr>
<tr>
<td></td>
<td>291</td>
</tr>
<tr>
<td>Recurrent caries</td>
<td></td>
</tr>
<tr>
<td>Cavity preparation</td>
<td>786</td>
</tr>
<tr>
<td>Failure of material</td>
<td>27</td>
</tr>
<tr>
<td></td>
<td>813</td>
</tr>
<tr>
<td>Pulp or periodontal involvement</td>
<td></td>
</tr>
<tr>
<td>No cement base</td>
<td>3</td>
</tr>
<tr>
<td>No restoration of normal anatomic contour</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>19</td>
</tr>
</tbody>
</table>

Inadequate proximal retention grooves combined with too narrow an isthmus or too great a proximal flare were also noted as possible reasons for failure of Class II amalgam restorations. Wolcott (1958) mentioned the need to finish amalgam restorations in order to remove amalgam flash which may subsequently cause "ditching" of the margins, and the need to provide a more homogeneous surface by polishing so that future corrosion and tarnish may be minimised.

Stibbs (1958) wrote, "It is unusual to see an amalgam restoration which has been in place for even six months that still is worthy of the creative operator's pride. Yet this is not the fault of the material as such". Most of the responsibility for amalgam failures is placed with the dentist - his lack of diligence and attention to detail. Poor diagnosis and treatment planning have also been cited as a reason initiating failure.

Stibbs (1958), in agreement with Wolcott (1958), emphasised the importance of the application of a well-formed matrix to provide proximal contour and contact with adjacent teeth, and to prevent periodontal problems following interproximal amalgam overhangs.
Wilkinson and Haack (1958) gave three factors which affect the quality and life of amalgam restorations:

1. the dentist's skill in manipulation of the restorative material;
2. the shape of the cavity preparation, and
3. the strength of the material.

Because amalgam restorations are subjected to fluctuating stresses in the mouth, these investigators believed that fatigue strength of the material is of greater importance than static strength as a possible cause of amalgam restoration failure, particularly at margins.

Mahler (1958) pointed out the problem that the design of the cavity preparation limits the structural effectiveness of both the restoration to be placed and the remaining tooth structure. Provision of bulk in restorations can be achieved only at the expense of tooth structure, and, conversely, conservation of tooth structure can only be accomplished at the expense of the restoration's strength.

Mahler (1958) used photoelastic studies to investigate the stresses occurring in a Class II amalgam restoration and concluded that the stresses of greatest significance were tensile in nature, and concentrated at the isthmus of the restoration. This investigator advocated the use of a flat pulpal wall in combination with a sloping axial wall and with the isthmus placed as close to the axial wall as possible.
Nadal, Phillips and Swartz (1961) placed 257 amalgam restorations of varying final mercury contents (48%, 58% and 62%) into two cavity designs - large and small - and observed these over two years. Sixty-four other restorations of high-mercury content were also placed and observed over a one year period. In all cases, no evidence of dimensional change or flow was observed. Marginal failures increased at a relatively constant rate up to approximately fifteen months in all instances. The number and degree of severity of marginal failures was related to mercury content, with least deterioration occurring in the low mercury content restorations. Surface roughness was also related to mercury content with the greatest roughness observed at twelve months in the high-mercury content restorations. Gross fractures at the isthmus within the first twenty-four hours occurred in five cases out of seven where restorations were placed in small cavities. One isthmus fracture occurred in the large cavities in the first year. All gross fractures were related to traumatic occlusion or insufficient bulk of material. No correlation could be found between residual mercury content and the degree or rate of tarnish or corrosion.

Wilson and Ryge (1963) observed approximately 1,500 amalgam restorations over a one year period and concluded that non-zinc alloys produced amalgam restorations with a higher incidence of marginal fracture than zinc-containing alloys.
This was not indicated in any way by laboratory tests. Coarse-cut dental amalgam alloys produced restorations with poorer surface characteristics than fine-cut alloys, particularly in students' hands.

These workers, in agreement with investigators listed earlier, found that manipulative variables influence the clinical success of dental amalgam restorations to a large degree.

Asgar and Sutfin (1965) used the concept of a two-phase material to observe microscopic failure of amalgam induced by bending stresses. Using etching techniques developed by Schmitt (1960) and Wing (1961), these workers found that crack initiation, leading to failure, occurred most readily at voids, then Gamma-2 phase, through the grain boundaries of the Gamma-1 phase, then through the Gamma phase. In no case did failure initiate at the Gamma phase.

Jørgensen (1965) described two types of marginal fracture of dental amalgam. The first type of fracture forms an acute angle with the occlusal filling surface, leaving a wedge-shaped gap between the restoration and the cut-tooth surface. The second type of fracture forms an obtuse angle between the filling and fracture surface, leaving a more or less butt-joint where the amalgam meets the cut-tooth surface.
Jørgensen claims that the acute angle fracture is associated with pressure while the obtuse angle fracture is related to pulling forces. The acute angle fracture is due to tensile forces greater than can be withstood by the amalgam wedge in question. An explanation suggested by Jørgensen (1965) was that the wedge-shaped margin will deform elastically under load and return to its original shape following load removal, until such time as fracture occurs due to tensile fatigue.

Jørgensen (1965) states categorically that a slit between restoration and cavity surface is a prerequisite for acute angle type fracture of amalgam margins. Seven factors are listed which may lead to slit formation between amalgam restorations and cavity walls:

1. Corrosion
2. Dimensional change
3. Faulty condensation
4. Plasticity (flow)
5. Marginal excess
6. Enamel fracture
7. Caries.

Nagai, Ohashi and Hasegawa (1967) devised a method for testing marginal strength of dental amalgam specimens at twenty-four hours. Specimens 2 mm x 4 mm x 15 mm were prepared with different marginal angles (30°, 45°, 60° and 90°).
and tested in a modified Micro Vickers hardness tester. A steel ball of 1.0 mm diameter was released from 60 mm and a pressure of 500 gm was exerted on the specimen which was moving at 50 micrometres/second in such a manner that the load was gradually applied toward the margin. The movement of test specimens was stopped at the moment of fracture, the specimen removed, and examined at a magnification of 100X and the marginal strength evaluated according to the extent of fracture of the specimen. These workers found that spherical amalgam, as tested by them, had a greater marginal strength than conventional amalgam. This strength reached a maximum in ten hours for spherical amalgam alloy and was not sensitive to condensation pressure. Conventional lathe cut amalgam specimens took up to twenty-four hours to develop maximum marginal strength and were sensitive to condensation forces. Marginal angles (30°, 45°, 60° and 90°) were in direct proportion to resistance to fracture with marginal strengths increasing correspondingly with an increase in marginal angle.

Attalla and Gibb (1968) examined 560 Class II amalgam restorations and re-emphasised the classical requirements for successful Class II restorations. "Extension for prevention", a well contoured, wedged matrix, adequate condensation, lining of deep cavities and polishing of restorations are mandatory requirements for serviceable amalgam restorations.
Mahler et al (1970) evaluated the clinical marginal fracture characteristics of three commercial dental amalgams in the light of their mechanical properties in order to see which properties would relate best to clinical performance. Three alloys were chosen which exhibited varying mechanical properties (Dispersalloy, New True Dentalloy, 20th Century Micro). It was concluded at the end of one year that the mechanical properties of compressive strength, tensile strength, transverse strength, transverse deflection and A.D.A. flow do not appear to predict clinical marginal fracture, whereas the rheological properties of dynamic creep, static creep and slow-compressive strength do appear to predict clinical marginal fracture.

Wing (1971) suggests that the percentage of failures with amalgam is probably lower than for any other dental restorative material. This is in agreement with an earlier observation by Brekhus and Armstrong (1936).

Failures were regarded by Wing as falling into the same categories as Healey and Phillips (1949) as well as tarnish and corrosion and post operative pain.

1. Western Metallurgical Ltd., Edmonton, Saskatchewan, Canada.
3. L. D. Caulk Co., Milford, Del, U.S.A.
The six categories of failure are:

1. recurrent caries
2. fracture
3. dimensional change
4. post-operative pain
5. periodontal disease
6. excessive tarnish or corrosion.

Recurrent caries, fracture and post-operative pain may usually be considered to result from faulty cavity preparation, while fractures and the remaining factors may often be related to faulty manipulation of the amalgam.

Wing (1971) reinforces the importance of adequate cavity preparation and emphasises the importance of operator manipulative variables which affect the quality of amalgam restorations, because of changes in structure of the amalgam within the restoration.

Duperon, Nevile and Kasloff (1971) evaluated the corrosion resistance of three basic types of dental amalgam - conventional, spherical, dispersion-modified - to determine whether any one type was more resistant to corrosion IN VIVO. One hundred and sixty-four restorations were placed and evaluated according to lustre, surface texture, marginal breakdown and tooth discolouration at the margin of the restoration. On the basis of their results, these workers concluded that dispersion-modified dental amalgam alloys are more resistant to corrosion than spherical particle or conventional type amalgam alloys.
Jørgensen (1972) investigated the strength of corroded cylindrical amalgam specimens (5 mm x 5 mm) following immersion in a 10% solution of sodium citrate for thirty-two weeks. Using the diametral compression test, Jorgensen (1972) noted an average reduction in tensile strength of 16.4% and, as a result, supposed that corrosion may reduce the durability of clinical amalgams considerably.

Stevenson (1973) investigated the comparative compressive and tensile strengths of spherical and lathe cut amalgams stored in 1% sodium citrate for six months and found no statistically significant reduction in either strength compared with specimens stored in air. The discrepancy between the results of Jørgensen and Stevenson may be due to differences in specimen preparation, Gamma 2 content, and concentration of the storage solutions used.

Mahler, Terkla and Van Eysden (1973), in a follow-up to an earlier paper (Mahler et al, 1970), evaluated the marginal fracture of the same three amalgams (Dispersalloy, New True Dentalloy, 20th Century Micro), after a four year period, by the use of photographic records. These workers reached similar conclusions to Nadal, Phillips and Swartz

1. Western Metallurgical Ltd., Edmonton, Saskatchewan, Canada.
3. L. D. Caulk Co., Milford, Del, U.S.A.
(1961) that marginal fracture increased at a relatively constant rate with time. Mahler et al (1973) suggested a minimum period of two years be used to evaluate marginal fracture characteristics of dental amalgam restorations. The rate of increase of marginal fracture was different for different alloys while the relative order of degree of marginal fracture among all alloys remained the same during the four year period with the best performance shown by Dispersalloy, then New True Dentalloy, then 20th Century Micro.

Galan, Phillips and Swartz (1973) investigated the deformation of IN VITRO Class II amalgam restorations in relation to types of alloys, cervical retention grooves and bevels. The specimens were tested at three hours and seven days by subjecting them to a dynamic load of 11.35 gm delivered through a 1.5 mm diameter pin placed at either the marginal ridge or isthmus of the specimen and cycled six times/minute for twenty-one hours. It was found that the alloy system plays a far greater role in the mesiodistal deformation of restorations than does the presence of a cervical retention groove or a cavosurface margin bevel. These workers found the best performance was given by Dispersalloy,\(^1\) and the worst by Micro Alloy,\(^2\) with a

\(^{1}\) American Silver and Mercury Producers, El Cajon, Calif., U.S.A.

\(^{2}\) L. D. Caulk Co., Milford, Del, U.S.A.
range of other alloys in between. They suggested that the creep characteristics of alloys may be related to mesiodistal deformation or marginal breakdown.

Osborne et al (1976) assessed the clinical marginal breakdown of the conventional alloys Aristaloy\(^1\) and Microcut\(^2\) adjacent to a dispersion alloy, Dispersalloy\(^3\) and concluded that the proximity of restorations is not a critical factor in the design of clinical studies involving dissimilar alloy systems.

Forsten and Kallio (1976) devised a method to accelerate the testing of dental amalgams with respect to marginal fracture, in a semi-clinical way. Occlusal cavities were cut in acrylic teeth of patients who were to receive dentures. The cavo-surface margin of the cavities were bevelled to \(45^\circ\). Amalgam alloy restorations (Amalcap F.G.\(^4\), Dispersalloy\(^5\), Amalcap non-gamma\(^6\)) were placed and the dentures issued. This semi-clinical technique gave gross marginal fracture in many cases within six months. Cavity size and shape were easy to standardise and the greater resilience of the

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1. Baker Dental Division of Englehard, Inc., Carteret, N.J., U.S.A.
2. L. D. Caulk & Co., Milford, Del., U.S.A.
acrylic compared with enamel gave less support to the amalgam restoration and margins particularly. These workers found no statistically significant difference between marginal fracture of conventional and dispersion-modified amalgams even though a trend showed less marginal fracture for the dispersion-modified alloys.

It was postulated that perhaps the large difference found in earlier clinical studies (Mahler et al, 1970, 1973) may have been due to the effects of corrosion rather than to differences in creep. It may therefore not be justifiable to predict clinical marginal behaviour by creep values alone.

Charbeneau et al (1977), in a comparative study over eighteen months of Tytin,¹ Dispersalloy² and Spheraloy,³ three alloys with creep values of approximately 0.1%, 0.6% and 1.9% respectively, showed that for margin adaptation, Spheraloy is the best, Dispersalloy is rated next and Tytin produced the poorest results, when rated using the Dental Health Center Criteria (Ryge, 1973).

It appears that from the earliest times there has been an attempt at the development of strength as a prime requirement for better clinical performance in dental amalgams. Mahler (1969), in his now "classic" paper, threw doubts on this type of thinking and drew attention to the possible relationship between creep and clinical marginal integrity. The recent development of alloys aimed at lower creep values and their subsequent testing and clinical evaluation leaves doubts as to the simple relationship between creep and clinical behaviour proposed by Mahler and accepted by many others.

A clinical survey of the need for replacement of silver amalgam restorations was carried out by sixty South Australian General Practitioners over 271 working days, and a comparison made with a similar study carried out by fifty Canadian Practitioners, over 246 working days. The South Australian figures were corrected to fifty dentists where necessary to obtain a straight comparison. See page 18.
REPLACEMENT OF SILVER AMALGAM RESTORATIONS

A CLINICAL SURVEY AND COMPARISON

by General Practice Study Group S.A.

A survey of the replacement of silver amalgam restorations by 60 S.A. General Practitioners during 271 working days compared to the results of a similar Canadian Survey by 50 dentists during 246 working days. Both surveys produced similar results.

The reasons for removal are shown in the table below. A, B and C which involve caries constituted 64 per cent in S.A. and 68 per cent in the Canadian survey of the total recorded reasons.

The number of teeth in which restorations were removed per dentist per day was: Canada, 3.8, S.A., 3.87. Surfaces removed per day per dentist were: Canada, 6.7, S.A., 7.3.

In order to obtain a straight comparison, the S.A. figures have been corrected to 50 dentists where necessary.

<table>
<thead>
<tr>
<th>Mean Age of Patient in Years</th>
<th>Canada</th>
<th>S.A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of teeth from which amalgams were removed</td>
<td>937</td>
<td>874</td>
</tr>
<tr>
<td>Surfaces removed</td>
<td>1643</td>
<td>1638</td>
</tr>
<tr>
<td>Surfaces restored</td>
<td>1991</td>
<td>2036</td>
</tr>
<tr>
<td>Crowns replaced</td>
<td>53</td>
<td>17</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Reasons for Removal of Amalgam Fillings</th>
<th>Canada %</th>
<th>S.A. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Recurrent caries at margin</td>
<td>23</td>
<td>23</td>
</tr>
<tr>
<td>B. New caries elsewhere than at margin of existing restoration</td>
<td>31</td>
<td>27</td>
</tr>
<tr>
<td>C. Caries under an existing restoration</td>
<td>14</td>
<td>14</td>
</tr>
<tr>
<td>D. Chipped margin</td>
<td>5</td>
<td>7</td>
</tr>
<tr>
<td>E. Poorly contoured restoration</td>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td>F. Overhanging margin</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>G. Fractured tooth</td>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>H. Pulpal Pathology</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>I. Fractured amalgam</td>
<td>9</td>
<td>9</td>
</tr>
<tr>
<td>J. Gold replacement</td>
<td>1</td>
<td>0.2</td>
</tr>
<tr>
<td>K. Original amalgam fell out</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>L. Other reasons</td>
<td>1</td>
<td>0.8</td>
</tr>
</tbody>
</table>

CHAPTER 2

TRADITIONAL MECHANICAL PROPERTIES TESTING OF AMALGAM

Successful amalgam restorations depend in part on the in-service behaviour of the amalgam placed in the cavity following manipulation of the amalgam alloy, and mercury. The behaviour in the mouth is likely to be largely related to the mechanical and physical properties of the material.

It has long been assumed that the properties of importance in dental amalgam are strength, dimensional change, flow and creep, marginal adaptation and resistance to tarnish and corrosion. Of these properties, strength, dimensional change, flow and creep are relatively easily measured (in the laboratory) and have formed the basis for Specifications and Standards for Dental Amalgam Alloys since the first American Dental Association Specification for this material was formulated in 1934, and subsequently for the drawing up of the Australian Specifications (1949, 1977) and International Specifications (1960).

Until recently, STRENGTH has been regarded by the majority of workers in this field as probably the most important property of amalgam, giving an indication of likely clinical behaviour because amalgam restorations are constantly subjected to forces of occlusal loading. It has been assumed that stronger amalgams will last longer and resist fracture better than weaker amalgams. Early
draft American Dental Association Specifications contained a requirement of fully set compressive strength, but later revisions eliminated this property from the Specification, assuming that no additional information was given concerning likely behaviour of amalgam over that provided by the flow test. Amalgams complying with the flow test would always meet fully set, compressive strength requirements.

The Australian Specification, from its promulgation in 1949, has included a requirement of one hour compressive strength and, since 1977, a test for compressive strength at thirty minutes has been added, with the retention of the sixty minute test. This test is regarded as a method of determining setting rate.

**Dimensional Change**: has always been regarded as an important property of amalgam because of an apparent relationship between this property and marginal sealing of amalgam restorations. Dimensional change requirements were such that until recently, amalgams, as tested in the laboratory, were expected to exhibit a minimal initial shrinkage and a final small expansion in order to comply with the American, Australian, and International Specifications. Current Specifications (Australian Standard AS2110, 1977; American Dental Association Specification No. 1, 1970) reflect changed thinking with regard to this property - for example, both the Australian
Standard and the American Dental Association Specification set limits of expansion of 0 ± 0.2% at twenty-four hours.

**Flow:** is a measure of plastic deformation occurring when a sub-fracture load is applied to the specimen. Flow was assumed by many to relate to the development of overhanging margins and flattened contacts. Its main relationship to amalgam behaviour is now regarded as being an indication of rate of setting. This property is currently included in Specifications and Standards for Dental Amalgam Alloys.

**Creep:** is a property which appears to resemble flow, but is tested on fully set amalgams and is a true rheological property. Mahler and Van Eysden (1969) described static and dynamic creep tests for amalgam and have subsequently attempted to relate these tests and other mechanical property tests to clinical behaviour of amalgam as assessed by the resistance to marginal fracture of restorations (Mahler et al, 1970, 1973). Mahler et al (1970) found in their study that there was an "apparent" relationship between low creep and resistance to marginal fracture over a four year period. This has been confirmed by Osborne et al (1974). No direct relationship exists between creep and compressive, tensile or transverse strengths, as usually tested. Static Creep is now incorporated in the Australian Standard 2110 - 1977, for Dental Amalgam.
Strength: the most usual method of testing strength of dental amalgams has been compressive strength.

It is accepted that the major forces loading clinical amalgams, although related to compressive loads due to opposing cusps, are likely to produce tensile stresses which may lead to the subsequent fracture of a restoration. This is particularly so in the isthmus region of a Class II restoration and at the occlusal cavo surface of bevelled restorations (Mahler, 1958).

It would seem logical that in view of the tensile stress in amalgam restorations, tensile strength should be the test of choice. For a variety of reasons – not the least of which is difficulty of specimen preparation for this test – there has been relatively little work done with tensile strength testing of amalgam.

2.1 Tensile Strength Testing

Early work (Ward, 1924) on tensile strength testing of amalgam was modelled on a test for Portland cement, with a briquette model of dimensions 0.25" (6.35 mm) thick by 0.2" (5.08 mm) wide at the narrowest part (Fig. 2-1, p.26). Ward reported tensile strength of approximately 5,600 p.s.i. (38.6 MPa) but neglected to report either specimen age or rate of loading. Little effect of rate of loading on tensile strength was found.
Taylor (1930) used Ward's model to test several amalgams for tensile strength and commented on the difficulty of triturating and adequately condensing such large specimens in a reasonable time. Taylor reported the tensile strength values as approximately one-tenth of the compressive strength for the alloys tested, and predicted higher tensile strength values for smaller specimens.

Coy and Liebig (1938) reported that at one day, the tensile strengths of amalgams prepared from fine-grained alloys were higher than those prepared from more coarsely cut amalgam alloys. At five days tensile strength values of amalgam prepared from the two types of alloys did not exhibit significant differences from one another.

Sweeney (1940) used an Hollenback pneumatic mallet for preparing specimens to test the physical properties of an amalgam alloy and reported a tensile strength of 11,000 p.s.i. (75.9 MPa); 44% higher than that given by the manufacturer. However, Sweeney failed to supply data relating to age of specimen, rate of loading or number of specimens tested.

Mahler (1958) used photoelastic studies to investigate the stresses acting in a Class II restoration and concluded that most fractures were due to tensile forces. Mahler
wrote, "Amalgam acts like a brittle material under the rapid rates of loading present during the application of occlusal forces. While the shear strength of amalgam is about half of its compressive strength, the tensile strength is about one-tenth of its compressive strength. It would be logical to give tensile stresses major consideration for this restorative material." Mahler, in his photoelastic studies, found the isthmus to be the most tensile-vulnerable region of Class II restorations under loading. It was the work of Mahler which revived interest in the tensile testing of amalgam.

Rodriguez and Dickson (1962) used a modified version of Ward's briquette model for tensile testing of four alloys. To facilitate specimen preparation, the cross-sectional area of the straight section of the specimen was reduced from 0.05 in.$^2$ (32.26 mm$^2$) to 0.01 in.$^2$ (6.45 mm$^2$), while the length was increased to 0.3" (7.62 mm) to accommodate strain gauges (Fig. 2-1, p.26).

From present knowledge (Forsten, 1972(b); Jørgensen, 1965; Nagai et al, 1971) it seems likely that, because of specimen size, the method of specimen preparation used by Rodriguez and Dickson, involving considerable delay (3.75 minutes) between condensation of increments, would have reduced the optimal strength of the specimens and involved some "layering" or inhomogeneity of the
amalgam, even though the total preparation time was six minutes. These workers found the tensile strength of "specification-type" amalgams to be between 1/4 and 1/5 of their compressive strength at seven days and that, in agreement with Ward (1924), tensile strength of amalgam is independent of the rate of loading. Tensile strength at one hour was reported as 10% - 15% of the fully set strength: making Class II restorations most susceptible to fracture from early application of masticatory forces.

Mahler and Mitchem (1964) used a specimen 1.0 mm thick, 4.0 mm wide and 12.0 mm long for their direct tensile strength tests. The specimen had a central constriction, giving a width of 2.0 mm. Following removal from the mould, a further 0.2 mm was removed from each side of the central constriction, leaving a final sample with a width at the fracture region of 1.6 mm (Figure 2-1, p.26). A cross-head speed of 0.5 mm/minute was used to test these specimens. These workers found a decrease in tensile strength as residual mercury increased, with a sharp decrease evident at and above approximately 54% mercury content. In agreement with the findings of Rodriguez and Dickson (1962), the rate of loading did not appear to affect the tensile strength of the amalgam specimens tested.
<table>
<thead>
<tr>
<th>Reporter</th>
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<tr>
<td>N. O. Taylor</td>
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<td><img src="image" alt="Diagram" /></td>
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<tr>
<td>M.S. Rodriguez &amp;</td>
<td>0.16 cm³</td>
<td>Thickness: 0.10&quot;</td>
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<td>G. Dickson</td>
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<tr>
<td>D B Mahler &amp;</td>
<td>0.04 cm³</td>
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<tr>
<td>J C Mitchem</td>
<td></td>
<td></td>
</tr>
<tr>
<td>K Nagai et al</td>
<td>0.16 cm³</td>
<td>Thickness: 2.5mm</td>
</tr>
</tbody>
</table>

Fig. 2-1. Specimen shapes used for the direct tensile testing of amalgams.
Nagai et al (1968a) used a similar specimen to that used by Rodriguez and Dickson (1962) (Fig. 2-1, p.26) to compare differences between spherical and conventional amalgams relative to tensile strength, elongation and modulus of elasticity. These workers found that for the alloys tested, spherical amalgams had approximately twice the tensile strength of conventional lathe cut amalgams at one hour. The one hour tensile strength of the conventional amalgams compared with the one week strength was given as 13% - 17% (corresponding to 10% - 15% given by Rodriguez and Dickson) while the spherical amalgams achieved 24% - 26% of the one week tensile strength. The one week tensile strengths of the amalgams tested were not significantly different and on the whole, the amalgam which has a large tensile strength is found to have a correspondingly large modulus of elasticity.

Certain disadvantages exist with the direct tensile test which have caused its almost universal replacement by an indirect tensile test: the Brazilian test.

With the direct test, there is the need to produce a specimen which can be gripped in conventional testing machines without damaging the specimen. This requires a specimen so large as to bear little relationship to the size of clinical amalgams. Rodriguez and Dickson
(1962) stated: "The low tensile strength of amalgam and the inability to make large specimens by conventional dental techniques have required that the testing machine be sensitive to small loads and provide low head speeds." Taylor (1930), had previously commented on the inability to triturate and adequately condense the large amounts of amalgam required for these specimens in a reasonable time.

Nagai et al (1970) commented on the need for a grip mechanism whereby the specimen was clasped without jeopardising the specimen strength or affecting the axial loading potential of the test piece.

Nagai et al (1970, 1971) stated that the dumb-bell shaped tensile specimens tended to be fractured by the chuck on load application. This, they reasoned, was responsible for a "relative dearth" of research efforts for tensile strength testing of amalgam, owing to the difficulty in fabricating specimens of "homogeneous quality" without an attendant wide spread of data (Nagai et al, 1971).

Basker and Wilson (1973) commented that large specimens require multiple mixes of amalgam, therefore increasing the risk of amalgam layering within the specimen.
Forsten (1972(b)), has shown that delayed condensation, as occurred in the fabrication of earlier tensile specimens, resulted in weaker amalgams. This is probably due to the disruption of initial crystallisations in the first few minutes following trituration. This situation is exaggerated when a "dry" mix is used.

The Brazilian, indirect tension, or diametral compression test, was introduced for testing tensile strength of amalgam by Burns and Sweeney (1965), following its successful introduction into the dental field by Rudnick, Hunter and Holden (1963) for testing silicate cement, and zinc phosphate cement. This test was originally devised by Akazawa (1943) for testing fragile materials such as concrete. This test method is the one currently used by most workers for the determination of tensile strength of amalgam. The advantages claimed for this method of tensile testing are:

1. The convenient size and ease of specimen preparation
   - 8 x 4 mm cylindrical specimens are the same as those used for compressive strength testing.
2. The same equipment can be used for both tensile and compressive strength testing.
3. The need for intricate specimen grips and aligning mechanisms is eliminated.
4. Specimens are of a size comparable with clinical amalgams, and do not require an elaborate mould or condensation technique as do the briquette or dumb-bell specimens.

5. Condensation can be effected in a reasonable time without the risk of "layering" or specimen weakening.

Nagai et al (1970), compared the relative merits of the new diametral compression and the old direct tensile tests for amalgam, for lathe cut and spherical amalgams prepared by varying manipulative techniques. Except for those cases where consecutive tensile strengths were required, all other values were read at twenty-four hours. Since the diametral compression test is noted for its accuracy in determining initial strength of brittle materials (Burns and Sweeney, 1965) these workers measured the initial strength inside one hour, which they state was an impossibility with the previous direct method. Nagai et al concluded that:

"1. Diametral compression test is found to be quite easy in the preparation of test specimens with a minimum of data scatter. It can be recommended as a reliable test method of the tensile strength suitable for fragile materials."
2. The tensile strength varies from amalgam product to product. Some spherical products failed to come up to standards expected of spherical amalgam.

3. Percentile distribution of the average tensile strength with 7-day strength as 100 is as follows: 15 minutes (8%), 30 minutes (15%), one hour (29%), 6 hours (82%) and 24 hours (96%). An increase in the strength after 90 days was merely 2%.

4. Spherical amalgam gives a higher tensile strength than conventional amalgam. Its 24-hour strength is over 700Kg/cm² (70 MPa).

5. Although the tensile strength of spherical amalgam is not affected by a condensation pressure to be applied, that of conventional amalgam will be reduced in proportion to a decrease in the pressure applied.

6. There exists a definite correlation between the compressive and tensile strengths. The ratio is 6.9 for spherical and 7.5 for conventional amalgam."

A large number of investigators have now used the diametral compression test as an evaluative method for different aspects of dental amalgam.
In a paper investigating the effects of manipulative variables on tensile strength, by the diametral method, Nagai et al (1971) found that the tensile strength of amalgam decreases proportionally to the time between trituration and condensation. Similarly, prolonged condensation of increments results in lower tensile strength, while more increments condensed quickly result in a higher tensile strength. Tensile strength of conventional alloys is more susceptible to condensation pressure than spherical alloys (Nagai et al, 1970). These results parallel similar findings of Eden and Waterstrat (1967) and Koran and Asgar (1967), who advocated the use of a mechanical condenser to reduce total condensation time.

Mahler (1970) investigated the relationship of marginal fracture of amalgam restorations to physical properties and concluded that tensile strength (diametral) was one of the properties bearing no correlation with marginal fracture.

Jørgensen (1972) used a diametral tensile test to evaluate the strength of corroded amalgam. Using cylindrical 5 mm x 5 mm specimens, Jørgensen found an average 16.4% strength reduction for the alloys tested, compared with uncorroded amalgams.
Acharya et al (1973) used the diametral compression test to evaluate an admixed high copper amalgam. This amalgam was prepared by triturating a copper amalgam pellet, then an equal amount of conventional alloy: then triturating the two together for fifteen seconds. These workers claimed an increase in tensile strength of 42% after fifteen minutes, compared with a conventional amalgam. Surprisingly, at twenty-four hours, the tensile strength of the conventional alloy was four times greater than the admixed amalgam.

Basker and Wilson (1973) criticised existing specifications for specimen preparation saying that "some aspects of specimen preparations bear little resemblance to normal clinical practice and do not take into account the difference in handling characteristics between conventional and spherical particle amalgams". Further, they considered the size of specimen (8 mm x 4 mm) unrealistic, requiring multiple mixes and therefore increasing the probability of layering within the cylindrical specimens. Basker and Wilson proposed the use of a specimen 3 mm x 3 mm with a hand-packing force of 15N for conventional and 8N for spherical alloy specimens. These workers endorsed the use of the diametral compression test for tensile strength determinations at one hour and suggested a minimum value for tensile strength should be 8 MPa (1,160 p.s.i.) at that time.
Ohashi et al (1975) investigated the effects of size of mix and trituration time on physical properties of amalgam. Unfortunately, no details were given as to length, path, or number of oscillations of the amalgamator used, so that no idea of actual energy input into the amalgam could be deduced. These investigators used the diametral compression test at twenty-four hours, for comparison of tensile strengths and concluded that the size of mix has little effect on tensile or compressive strength, or dimensional change, while trituration time influences these properties to a large extent. Insufficient trituration time reduces strength and too much trituration increases shrinkage in dimensional change. These findings are in agreement with other workers (Phillips, 1957; Ward and Scott, 1932; Caul, Longton, Sweeney and Paffenberg, 1963; Holst, 1965). There appears to be no necessity to change the optimal trituration time regardless of the size of a mix.

Investigating the effect of anodic polarisation on the tensile strength of dental amalgam, Wang Chen and Greener (1976) compared results for the direct tensile and the diametral compression test methods. After setting in air for twenty-four hours, specimens were anodically polarised
in Ringer's solution for ninety-six hours before tensile testing. For the direct tensile test a dumb-bell shaped specimen similar to the one used by Rodriguez and Dickson (1962) was used. Instead of using hand condensation, as did Rodriguez and Dickson, an hydraulic press was used for compacting the specimens. A pressure of 2,030 p.s.i. (14 MPa) as indicated by the American Dental Association Specification No. 1 for preparing standard dental amalgam specimens, was applied. It is significant that so large a specimen preparation took less than three minutes, following the completion of trituration.

The diametral tensile specimens were prepared following the procedures outlined in the American Dental Association Specification No. 1 (1972-73).

Using controls, these workers evaluated the conventional amalgam, Aristaloy,¹ and the high copper amalgam, Dispersalloy,² and found a 27% reduction in the mean direct tensile strength of the conventional amalgam following anodic polarisation, whereas the Dispersalloy was unaffected by anodic polarisation, when the direct tensile test method was used.

When the diametral tensile strengths of polarised and unpolarised conventional amalgam and Dispersalloy specimens were compared, no significant difference in strength was observed between the polarised and the unpolarised specimens.

These workers were in agreement with Johnson and Wilsdorf (1972) who noted that one of the characteristics of the diametral tensile method is its insensitivity to surface conditions of the specimen because the fracture is nucleated in the bulk of the material rather than at the surface.

Wang Chen and Greener (1976) reasoned on the basis that because the corrosion affected the outer specimen surface, it seemed logical that the direct tensile test - with corroded areas acting as stress raisers - was a more sensitive measure than the indirect test. Wang Chen and Greener (1976) concluded that "the diametral tensile test is not sufficiently discriminating to allow for an IN VITRO simulation of clinical properties".

It is interesting to note that Lautenschlager and Harcourt (1970), using photoelastic methods, have shown that the diametral test is only applicable to homogeneous materials without porosity or second phases. Amalgam is
a multiphasic alloy containing pores and, as such, the diametral test may not reflect true tensile strength as does the direct axial tensile test.

Turchyn and Youdelis (1970) interpreted flexure test results for amalgam and argued that since flow occurs in amalgam, the diametral compression test does not give a true value for tensile strength of amalgam, particularly for alloys with high creep values. Visibly observable flattening of specimens occurs during indirect tensile testing, due to plastic deformation. These workers claimed to show that the alloys having the lowest compressive strength and hardness and the highest flow, also showed the highest (apparent) flexure or transverse strength. "In both the diametral test and the flexure test, plastic deformation of the specimen decreases the actual tensile stresses, and accordingly a higher load must be applied for fracture, resulting in a higher 'apparent' tensile or flexure strength." These workers advocate the use of a three point loading system, with a cylindrical specimen 12 mm x 4 mm as a more suitable test compared with the diametral tensile test, based on the precision of the results obtained. Unfortunately, no measurements were given as to the distance between the centres of the load-bearing rods or the equipment suggested for testing.
Although there are shortcomings with the diametral compression test for tensile testing of amalgam, this method has the advantage of ease of specimen preparation, ease of testing without the need for complicated specimen grips or apparatus as occurs with the dumb-bell type specimen. Ideally the diametral compression test should be used for the tensile testing of homogeneous materials. Dental amalgam is an inhomogeneous material which is subject to plastic deformation and as such should be tested for tensile strength by the direct axial method. The indirect tension test is convenient and it is to be remembered that, although this test does not give a true tensile strength, as does the direct axial test, a relationship exists between values obtained for this strength and other strength tests.

The high copper ternary and the dispersion modified amalgams have such low creep values that plastic flow as noted by Turchyn and Youdelis (1970) in the diametral tensile test is almost eliminated. Low creep values are associated with brittle materials and therefore the diametral tensile test may be a good gauge as to the true tensile strength of these newer, low creep, high copper amalgams.
2.2 Compressive Strength Testing:

One of the important requirements of dental restorations is strength. The ability of amalgam restorations to withstand the repeated, complex forces of mastication may be vital to clinical success.

Compressive strength determination has been accepted for many years as a reliable measure for evaluating the strength of dental amalgam and most early research was directed at investigation of this property.

Various sizes of cylindrical specimens have been used for compressive strength testing of amalgam. Taylor et al (1949) used cylindrical specimens 8 mm x 4 mm and 12 mm x 6 mm for their work, and found it important to maintain a ratio between height and width of 2:1. The 8 mm x 4 mm specimen, which is the standard size accepted for American Dental Association flow tests, yielded compressive strength values 2 – 7% higher than the larger specimen. Notably, Taylor et al (1949) showed the importance of rate of loading on compressive strength; finding that compressive strength increased with the rate of loading. A suggestion was made that a cross-head speed of 0.003 in./minute be used to determine both elastic and plastic properties of amalgam as related to compressive strength.
The effect of the rate of load application on compressive strength was in agreement with earlier work by Ward (1924) and was later to be reinforced by Mahler et al (1972) who investigated slow compressive strength at various head speeds and found that at a cross-head speed of 0.001 in./minute, a good correlation ($r = 0.931$) with dynamic creep values existed. Peyton and Craig (1971, p.69) illustrate the forces acting in a compression test specimen. (Fig. 2-2, p.41). It is obvious that in a structure, subjected primarily to a compressive force, stresses representing tensile and shear components of this force are also present.

Mahler et al (1972) also noted the greater role of plastic deformation of the amalgam prior to fracture when low head speeds were used. Both Mahler et al (1972) and Taylor (1949) were in agreement that the slower loading rates with their resultant lower compressive strengths correlated better with the clinical behaviour of amalgam.

In an earlier paper, Mahler et al (1970) found an apparent relationship between marginal fracture and dynamic creep of amalgam. Because dynamic creep correlates well with slow compressive strength, it would seem reasonable to predict the marginal fracture characteristics of an amalgam from its slow compressive strength, relative to other amalgam alloys tested.
Fig. 2-2. Drawing of complex stress pattern that developed in cylinder subjected to compression stress.

After Peyton and Craig (1971).
The early compressive strength of amalgam is often of vital importance to the success of a restoration. The early development of high strength minimises the risk of fracture by the patient's premature application of masticatory forces and many workers consider that early strength is more relevant than post-twenty-four hour performance. (Taylor et al, 1949; Phillips, 1949; Ryge et al, 1952; Ware and Docking, 1955; Wing, 1965.)

Taylor et al (1949), Phillips (1949), Wing (1961) and Bryant (1979) investigated the rate of development of compressive strength in amalgam. Results showed that at six hours following condensation, conventional alloys attained approximately 50% of maximum strength, 80% at eight hours, and usually 100% at twenty-four hours. However, some slower setting alloys may require seven days to achieve maximum strength and the rate of development of strength may be dependent on manipulative variables.

Swartz and Phillips (1956) found no appreciable difference in fully set "crushing" strength of specimens with residual mercury contents between 50% - 55%. However, a serious loss of strength resulted above 55% residual mercury.
Wing (1961) found that, when using the recommended mercury:alloy ratio for seven alloys tested, the compressive strength at one hour tended to be higher for the three fine grained alloys than for the coarser alloys and this slight increase in strength was maintained for fully set amalgams. One fine grained amalgam was an exception and showed a comparatively lowered strength at seven days. When the initial mercury:alloy ratio was varied, it was noted that a decrease in the initial mercury:alloy ratio resulted in a general increase in the one-hour compressive strength. For fully-set specimens there appears to be a levelling off of the influence of the original alloy:mercury ratio. While the one-hour compressive strength may be increased by 20 – 25% by reducing the ratio from 7:5 to 5:5, the twenty-four hour compressive strength is increased by only 5 – 8% for the same change in mercury:alloy 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 (1961) and Caul et al (1963) studied the effect of manipulative variables and temperature on compressive strength and, in agreement with earlier workers, found that compressive strength increased with rate of loading. Under-trituration produced weaker amalgams and the compressive strength at 60°C was approximately 50% of its strength at 23°C.

Mahler (1964) found that as the residual-mercury content of fully-set amalgam specimens increases, the strength decreases. In addition, the rate of strength decrease is much greater at residual-mercury contents in excess of approximately 54% than at contents less than this value.

2.3 Transverse Strength Testing

As mentioned previously, most strength testing of amalgam has been performed using the compressive and, more recently, the direct tensile and diametral compression test methods. A review of the literature shows abundant studies of these methods.

A relative paucity of investigations exists regarding transverse testing. This strength may be described as that resulting from a combination of compressive and tensile stresses; separated by a neutral zone devoid of stress.
Transverse strength, also known as bend or flexure strength, is obtained by loading a bar or beam, which is supported at each end with the load applied in the middle (Peyton and Craig, 1971, p.74).

The modulus of rupture (transverse test) is not a true stress but is used to predict and compare the strength of similar beams of the same material (Roark, 1965, p.35). This test provides an excellent basis for comparison of the various alloys under identical conditions of testing, but as with all mechanical tests, can give only a limited appreciation of performance under the state of stress that may be encountered in the oral environment (Peet, 1971, p.87). Chapter 3 of this review will be devoted to this strength test.

Some workers reason that the transverse test is the best "all round" or average test in relation to clinical fracture strength, although compressive strength testing is the most widely used laboratory test for amalgams and is good for comparing the relative strengths of various test methods and for assessing the effect of metallurgical variables (Dental Materials Research, NBS Pub. 354:50, July, 1972).
Since clinical fracture is mainly a tensile event (Mahler, 1958; Jorgensen, 1965), it would seem to negate compression testing as a useful method for appraising clinical strength. However, Wing (1971) has suggested that since parallel strength properties exist for various test methods for amalgam, it is more convenient to test compressive strength and realise that certain ratios exist between this and other strength tests. This has been shown by Mahler (1964) to be so. (Fig. 2-3, p.47)

2.4 Flow

When subjected to a static, sub-fracture compressive load, amalgam exhibits a plastic deformation, or flow. Skinner and Phillips (1967) commented that it would seem that the main merit of the flow test lies in its ability to evaluate general strength properties of amalgam restorations. Wing (1971) stated "that flow is primarily a measure of the rate of setting of the amalgam and probably bears little relationship to clinical behaviour of the material".

A cylindrical specimen measuring 8 mm x 4 mm is loaded at 10.3 MPa (1,500 p.s.i.) three hours after trituration and the percentage decrease in length at twenty-four hours is given as the flow. The American Dental Association
Fig. 2-3. Relationship of strength and residual-mercury content of amalgam. Tensile and transverse strength curves have been adjusted to conform to the position of the compressive-strength curve.

(After Mahler, 1964)
Specification No. 1 allows for a maximum flow of 3%. Factors affecting the flow of an amalgam are temperature, condensation pressure and mercury content. Worner (1937) showed that at higher temperatures, flow increased, or less load was required to produce a comparable degree of flow.

The early American Dental Association Specification for flow testing required a load of 3,550 p.s.i. (24.5 MPa) to be used at a temperature of 20 - 25°C to produce a comparable flow to the Australian Specification which required a load of 10.3 MPa (1,500 p.s.i.) at 37°C for the same flow value. Now, the Specification of the American Dental Association, Australian Dental Association, and Federation Dentaire Internationale all require testing at 37°C and a comparable load.

Skinner (1938) considered that, clinically, flow is important and with time manifests itself by the disappearance of contact points, appearance of gingival overhangs and fracturing of occlusal margins.

Healey and Phillips (1949) observed flow in some amalgam restorations but concluded that this was not of sufficient magnitude to cause restoration failure. However, these investigators conceded that in some restorations, failures due to fracture, particularly those with narrow isthmuses, may have been hastened by flow causing a reduction in the bulk of material at the isthmus.
Many workers have claimed that flow is not a clinical problem and rarely, if ever, manifests itself. Mosteller (1953) claimed that since intermittent dynamic loads act in the mouth and not static loads, then the clinical implications of flow are doubtful. Phillips et al (1945) were also critical of the flow test and noted that the static test was not comparable to conditions in the mouth. These workers conducted an experiment using an amalgam with a flow of 6.5%. A two year follow up of eighty-one cases led to the conclusion that "flow is not evidenced in the mouth when cavity preparation is properly carried out".

Early reports (Black, 1896; Ward and Scott, 1935) showed some degree of strain hardening when amalgam specimens were subjected to flow testing. Worner (1937) concluded that work hardening, an increasing stiffness with time owing to the setting process, and a decrease in stress value as cross-sectional area increased with time, were responsible for relatively high initial flow over the first few hours of the test procedure.

Kimmel (1961) used a modified flow test to evaluate the early resistance to flow of several alloys. Kimmel used a load of 1,500 p.s.i. (10.3 MPa) to test flow at ten and fifteen minutes following commencement of trituration. One group of alloys showed little flow during the entire
two-hour test, while another group exhibited high initial flow in the first ten minutes with little flow after this time. A third group of alloys showed considerable flow throughout the test period. This investigator suggested that an early flow test be considered in later specifications due to its wide difference in performance of alloys. It is likely that these "differences in performance" of the tested amalgams may relate more to setting rate of the material as mentioned by Wing (1961).

Sweeney and Burns (1961) conducted flow tests in accordance with the Specification No. 1 for Dental Amalgam Alloy and concluded that initial mercury:alloy ratio has little effect on flow of dental amalgam as long as the amalgam is firmly condensed and excess mercury removed. They suggested that flow may be interpreted as an index to the early strength for various mercury:alloy ratios.

Caul et al (1964) mechanically prepared cylindrical 8 mm x 4 mm test specimens to evaluate early strength, flow and dimensional change. These workers began flow measurements as early as three minutes from the end of triturating. Flow specimens were subjected to a load of 4.9 MPa (711 p.s.i.) for ten minutes. Flow data showed that the rate of hardening is similar to that shown by the compressive strength data. "As strength
increases linearly with time, the flow decreases at a nearly linear rate. The weakest alloy has the largest flow." Further, these workers found that altering the mercury:alloy ratio from a value of one to three did not affect flow appreciably. They concluded that the early flow test is a convenient method for determining setting rate or hardening of dental amalgam.

Fuse (1969) investigated the flow of several dental amalgam alloys and found flow to be temperature dependent. Flow for a spherical alloy at 37°C was twice the value as that at 16°C. The value for a fine-cut alloy at 37°C was 2.5 times greater than at 16°C, and three times greater for a regular-cut alloy, at the same temperature. Fuse (1969) also compared the flow of regular-cut, fine-cut and spherical particle alloys and found a ratio of 4:3:1 for the flow values of these alloys.

Basker and Wilson (1971), quoting Anderson (1956, a & b), and Anderson and Picton (1958), following a suggestion by Braden (1967), used a specimen of 3 mm diameter and 2 mm height to investigate flow by a method of repetitive indentation. A 2 mm diameter indentor was used to strike the specimen 120 times/minute at a maximum force of 150N/thrust (33.7 lbs). Specimens were tested at varying times following trituration. These workers related indentation depth, the number of thrusts made by the indentor, and the
time of testing following trituration. They found that as most of the indentation was produced by the first two hundred thrusts, this relationship was not linear. The age of the specimen had a marked effect on the indentation produced.

Basker and Wilson gave several probable reasons for their findings:
1. "The increasing hardness of the alloy as amalgamation proceeds";
2. "Work hardening of the material by the application of an intermittent force";
3. The consolidation of the surface of the amalgam with the consequent elimination of porosity.

Whereas flow has traditionally been regarded as an early static test, the study by Basker and Wilson would best be described as of a cyclic or dynamic nature.

2.5 Creep

Mahler and Van Eysden (1969) stated that the property describing the plastic deformation of fully-hardened material under cyclic or fatigue loading is known as "dynamic creep". This has been confirmed by Phillips (1973). Mahler et al designed a dynamic test where seven-day, cylindrical specimens measuring 8 mm x 4 mm
were subjected to a load cycling between 500 p.s.i. and 10,000 p.s.i. (3.5 MPa - 68.8 MPa). The mean stress of 5,250 p.s.i. (36.2 MPa) was applied 1,800 times per minute. The creep parameter was selected to be the percentage decrease in original length between one and four hours of testing. Similarly, a static creep test was performed using a constant stress of 5,250 p.s.i. (36.2 MPa). The static creep was evaluated as for the dynamic test - i.e.: the strain developed between one and four hours using seven day specimens. These workers found that the higher the residual-mercury content, the higher the creep values - both static and dynamic. The American Dental Association flow test was also considered valuable in approximating creep characteristics. In a later paper, Mahler et al (1970) showed evidence to relate slow compressive strength (0.001 in./minute or 0.025 mm/minute) and dynamic creep characteristics (correlation coefficient $r = 0.931$). Mahler et al (1972) commented that while creep tests were relatively new, most laboratories were equipped to perform slow compressive strength tests which would relate well to clinical performance, particularly with respect to marginal fracture (Mahler et al, 1970, 1973).
Binon et al (1973) used amalgam alloys of widely varying creep values in Class II restorations IN VIVO. At nine months recall, 31.8% of restorations placed had marginal defects. Marginal fracture, in the middle and high creep amalgams, was reported as 2.8 and 6.6 times respectively greater than that observed in the low creep amalgam. Compressive, slow compressive and tensile strengths, hardness and flow values did not correlate to clinically observed marginal fracture. These workers corroborated the findings of Mahler et al (1969) that static creep predicts the clinical marginal behaviour of amalgam better than other mechanical properties.

Galan et al (1973) investigated the hypothesis that Class II cavity preparation design influences the plastic deformation characteristics of amalgam restorations. Alloys of known creep values were subjected to IN VITRO dynamic loading. A force of 11,935 p.s.i. (82.3 MPa) was applied six times per minute over twenty-one hours, beginning at either three hours or seven days after trituration. These workers found that the alloy itself is of greater importance in mesiodistal deformation of amalgam restorations than either retention grooves or cavo-surface bevels. In agreement with earlier workers (Mahler et al, 1969, 1970), Galan et al (1973) concluded
that the creep properties of the alloy may be related to this deformation or marginal breakdown. It is interesting to note that seven day old restorations of 20th Century Micro Alloy\(^1\) (a high creep amalgam) produced a plastic deformation twenty times greater than that of Dispersalloy\(^2\) restorations (a low creep amalgam).

Osborne et al (1974) investigated the static creep of fourteen commercial alloys and corroborated the earlier findings of Mahler and Van Eysden (1969) and Binon et al (1973) who found no correlation between creep and compressive strength values of amalgams of similar residual mercury content. Mahler et al (1969) did, however, find higher creep values if the residual mercury content of the specimens was in the order of 53% or more.

Vrijhoef and Driessens (1974)(a), confirmed the hypothesis that the creep of dental amalgam is determined by the low melting phases in the matrix of reaction products of mercury and alloy.

Vrijhoef and Driessens (1975) investigated the influence of conventional alloy on the creep resistance of dispersion-modified amalgam. Using conventional alloys of known creep values - high, medium, low - in combination

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1. L. D. Caulk, Milford, Delaware, U.S.A.

2. American Silver and Mercury Producers, El Cajon, Calif., U.S.A.
with silver-copper eutectic alloy, these workers found that creep characteristics of the dispersion-modified amalgams paralleled the creep values of the original conventional amalgam alloy.

Schulman and Vaidyanthan (1976), in a laboratory and clinical study, were in agreement with Mahler et al (1969, 1970) and Osborne et al (1974) that amalgam alloys containing a silver-copper eutectic dispersant had significantly lower static and dynamic creep values and improved marginal integrity.

Espevik (1977) investigated the effect of mild heat treatment (37°C and 60°C) for varying times on the creep properties of several alloys. Espevik found that increased heat treatment of the specimen (both time and temperature) prior to testing resulted in decreased creep values, probably due to a transformation of gamma-1 phase to beta-1 phase, which was detected by metallographic and Microprobe analysis.

2.6 Dimensional Change

It has generally been accepted that dental amalgams should exhibit a slight overall expansion, during setting (p.20), as measured in the laboratory during testing for specifications. For many years it had been assumed that this laboratory test related to marginal sealing of amalgam restorations and degree of adaptation to cavity walls.
McDonald and Phillips (1950) used an amalgam alloy, which shrank 31 micrometres/cm. at twenty-four hours, to fill six deciduous teeth. After three years, no clinical evidence of shrinkage was found. This was in agreement with earlier findings by Phillips et al (1945).

Dimensional changes as measured in the laboratory reflect not only contractions and expansions of the amalgam during setting but also dimensional changes due to low loads applied by the measuring instrument used (Wing, 1964).

Many workers have used different instrumentation, different techniques and times of initial reading for the determination of this property.

The early American Dental Association Specification No. 1 (1937) required that the measurement for dimensional change begin fifteen minutes following trituration. Many workers have begun measurements sooner than fifteen minutes, since clinical amalgam restorations are placed within several minutes of trituration. The current American Dental Association Specification No. 1 (1970) requires the initial measurement to be made five minutes after the end of trituration, at 37± 1°C, with the final measurement made at the end of twenty-four hours. The Specification
required the use of a measuring instrument with an accuracy of 0.5 micrometres/cm. The instrument of choice for such measurement is the light interferometer, first used by Souder and Paffenbarger (1942). Optical micrometers have been used by other investigators (Ward and Scott, 1932; Worner, 1940; Gayler, 1934), while accurate dial gauges have also been used (Worner, 1937; Van Gunst and Hertog, 1957; Rantanen, 1960; Langeland, 1961 and Wing, 1961).

Fusayama et al (1964) used an air micrometer for their work, claiming that this instrument, with an accuracy of 0.5 micrometres/cm., made it possible to obtain early measurement of specimens which were still soft, since contact with the specimen was not necessary. These workers took measurements five minutes after the start of trituration.

Fisher and Lehwald (1954) commenced readings for dimensional change calculations seven to eight minutes after the start of trituration, while Mitchell et al (1955) began readings at ten minutes.

While most researchers had measured vertical dimensional change along the axis of specimens, Fusayama et al (1964) reasoned that clinical "marginal closure" correlates to horizontal dimensional change of amalgam in the occlusal or open-surface layers. These investigators found a
considerable initial contraction during the first ten minutes which was not observed in measurements begun at fifteen minutes according to the conventional test of the time. The greatest transverse expansion was in the top layers while smaller expansion occurred in the bottom layers of cylindrical specimens. These findings were compared to measurements of vertical dimensional change and it was concluded that "Transversal expansion of the top layers really affecting marginal closure of fillings was greater than vertical expansion of the whole length previously used".

Vrijhoef et al (1974) gave a brief but thorough resumé of the literature pertinent to the adaptation of restorative materials to cavity walls and the measurement of dimensional change. No studies were cited which were able to detect clinically either a high contraction or expansion of amalgam restorations IN VIVO which would parallel IN VITRO American Dental Association Specification No. 1 dimensional change measurements. The non-correlation between dimensional change and the adaptation (or interspace dimension) of amalgam restorations has, in fact, been reported (Wolcott et al, 1963; Granath and Nyquist, 1964; Wing and Lyell, 1965; Granath, 1971; Letzel, 1972).
Vrijhoef et al (1974) proposed a new method for the determination of dimensional change during setting in a clinically relevant way. Cylindrical specimens of amalgam were measured after setting in a mould and compared with dimensional change measurements according to American Dental Association Specification No. 1.

Vrijhoef et al (1975) used cylindrical specimens of 4 mm diameter which were removed from the mould at periods varying from six to ninety minutes according to mercury content of the specimens. After annealing for some time at 37°C the diameters of the specimens were determined. It was found that significant long-term dimensional changes occurred above 50 wt % mercury. At 60 wt % mercury, dimensional changes of the same order as the so-called "delayed expansion" were recorded over a period of 150 days.
CHAPTER 3

TRANSVERSE STRENGTH

Transverse testing has been widely used in the testing of materials. As a result of this type of testing, a value for transverse strength, modulus of rupture, or flexure strength, has been determined for a wide range of materials in many fields where the prediction of performance of structures is considered important. In actual fact, this test is somewhat controversial in so far as different interpretations have been placed on the test and the determined strength.

Some authors, Doan (1953), Fenner (1965), Wyatt and Dew-Hughes (1974), call this test a simple bend test, but are actually describing a transverse load test. Fenner (1965) also describes several other tests which are truly bend tests. Other authors, Gabel (1954), Bailey (1964), Peyton and Craig (1971), Bunshah (1971) and Phillips (1973) name and describe the test as a transverse test.

The transverse test is a simple mechanical test involving the loading of a beam supported at either end. The load is usually a single central one, but may involve a two-point load applied symmetrically between the supports. A static load is most commonly used.
In this test, not only is the strength of a material established; but the amount of distortion that may be expected from a material in service can be predicted (Peyton and Craig, 1971).

Doan (1953) described the bend test as one used chiefly for brittle materials, and which may serve as a simpler substitute for the tension test. He considers it a good shop test for "acceptance purposes", but not for research purposes.

Gabel (1954) stated that if a beam is not to be permanently deformed, then the maximum bending stresses, i.e. those in the material at the top and bottom of the beam at the point of the greatest bending moment, must not exceed the proportional limit of the material. These stresses depend on the bending moment at the specific beam section under consideration and on the size and shape of the cross-section. An increase in vertical depth of the cross-section has a far greater effect on the transverse strength than a corresponding increase in width.

Bailey (1961) remarks that the transverse test is one fairly commonly applied to cast iron, where a specimen of standard size is tested until a fracture load and maximum deflection are recorded.
Phillips (1973) considers that the transverse test is "in a sense, a collective measurement of all types of stresses simultaneously". Wyatt and Dew-Hughes (1974) state that brittle materials are often tested in bending, combining both the tensile and compressive behaviour of a material. "Even in brittle materials there is some slight plastic flow before fracture and, since this is not allowed for in the analysis, the modulus of rupture is higher than the tensile strength. For example, in concrete and other ceramics it is about (sic) double."

Fenner (1965) gives a simple step-by-step description of bending phenomena and the derivation of several of the formula used for determination of the transverse strength of a material. When materials, initially straight, are stressed in bending, there exist relationships between the applied bending moment and the deformation, on the one hand, and between the deformation and the stress at any point in the material on the other hand. These relationships are given by the elastic formulae:
\[
\frac{M}{I} = \frac{E}{R}
\]

and \[
\frac{M}{I} = \frac{f}{\gamma}
\]

where \( M \) = bending moment
\( I \) = second moment of area of the section about the neutral plane
\( E \) = Young's modulus of elasticity for the material
\( f \) = the tensile or compressive stress in the material at any distance 'y' from the neutral plane
\( R \) = radius of curvature.

For a circular section, \( I = \frac{\pi d^4}{64} \) \((d = \text{section diameter})\)

For a rectangular section, \( I = \frac{ab^3}{12} \) \((a = \text{section width}, b = \text{depth of cross-section})\)

In bending, stress and strain both vary linearly across the section, increasing from zero at the neutral plane to a maximum at the maximum value of 'y', so long as the material is everywhere behaving elastically.

The loaded surface suffers a maximum compression while the surface distant from the load point is subject to a maximum tensile force. A neutral zone - an area of zero stress - is found between the areas of compressive and tensile forces. This is represented diagrammatically in Fig. 3-1, p.65.

It is to be noted that if fracture occurs in a simply loaded beam, then the location of each zone - tensile, neutral, compressive - moves towards the loaded side (P) as the fracture progresses.
Fig. 3-1. A diagrammatic representation of the stresses in a transverse tested specimen.

(Reproduced from Gabel, 1954)
An excellent photograph of a photoelastic analysis of transverse bending is presented in Peyton and Craig (1971, p.75). This is reproduced in Fig. 3-2, p.67.

For materials which behave elastically under a simple three point bend test, the deflection of the mid-point of a uniform beam, seated on a span 'l' and subjected to a central load 'W', remains a linear function of the load and is given by:

\[ \frac{WI^3}{48EI} \]

where E is the normal expression relating stress and strain, i.e. \[ \frac{\sigma}{\varepsilon} \]

Once plastic flow commences, there is departure from a linear relationship, such that a load/deflection or bending/moment diagram for such a beam should show when the material passes its proportional limit. (Fig. 3-3, p.68)

For the simply loaded beam, the plastic flow is confined to the mid region where 'y' is a maximum distance from the central load point. (Fig. 3-4, p.69)

For this reason, if it is desired to estimate the limit of proportionality in bending, the symmetrical four-point system, as shown in Fig. 3-5, p.70, is usually adopted.
Fig. 3-2. Analysis of transverse bending using a photoelastic model showing isochromatic fringes.

(After Peyton and Craig, 1971, p. 75)
Fig. 3-3. Stress/strain curve from a bend test of structural steel

(After Fenner, 1965)
Fig. 3-4. Elastic stress distribution in bending

(After Fenner, 1965)
Fig. 3-5. Bending under symmetrical four-point loading

(After Fenner, 1965)
This system produces a uniform bending moment over the span length 'l' of the beam between the loads, and has a value \( \frac{Wx}{2} \). When plastic flow occurs in this case, it will occur over an appreciable length of the beam, and bring about a more noticeable departure from linearity of the load/deformation relationship.

Fenner pointed out that from the stress/strain diagram it can be seen that the curve shows no sudden drop such as would be seen at the yield point on a tensile stress/strain curve; the diagram shows a closer resemblance to a torsional stress/strain curve. After the limit of proportionality is passed, the load supported by the beam continues to rise until it reaches a maximum value beyond which further bending will continue under a lower load. The maximum value, the failing load of the beam, is sometimes used to calculate a figure known as the modulus of rupture in bending, which is analogous to the modulus of rupture in torsion. This is the nominal value of the surface stress in the material at the maximum load, calculated by the use of the elastic formula for stress as if the linear relationship between stress and strain were maintained to this point, and is used particularly for tests of fairly brittle materials.
In testing transverse strength, the load at failure is noted and the cross-breaking strength, i.e. the modulus of rupture, is computed as:

\[ MR = 1.5 \frac{Wl}{bd^2} \]

where
- \( MR \) = Modulus of Rupture
- \( W \) = load at failure
- \( l \) = span of supports
- \( b \) = width of test piece
- \( d \) = thickness of test piece

Bunshah (1971) states that the total energy, \( E_T \), to fracture a specimen, is divided into two components (Fig. 3-6, p.74). The first component is the energy to initiate the crack, \( E_I \), while the second is the energy to propagate the crack, \( E_P \).

In Figure 3-6, p.74, the first plastic instability 'PGy', is due to the spread of plastic deformation across the 'ligament' of the specimen; that is, from the notch to the opposing free surface, to cause 'general yielding'. The notch may be any irregularity on the tensile surface of a loaded specimen and is responsible for what is termed the "notch effect" - a focal spot for stress raising and concentration. The second instability occurs at "maximum load" due to necking. When the fracture occurs by fast
cleavage, there is a sudden drop in the load, and the fracture load, 'Pf', is easily identified from the load-time curve. Cleavage fracture may occur either before or after general yield or even beyond the maximum load.

At about one fifth of the load to cause general yielding, the longitudinal stress, at the "notch root", is raised by elastic stress concentration to the tensile yield stress of the material, and "local" yielding occurs. Further loading causes the plastic zone to spread into the ligament and under "plane strain" conditions the longitudinal stress is distributed "within the plastic zone" according to the slip line field solution of Hill (1956) (Fig. 3-6, p.74).
Fig. 3-6. Elastic-plastic stress distributions ahead of a notch.

(Reproduced from Bunshah, 1971)
CHAPTER 4

DENTAL ASPECTS OF TRANSVERSE STRENGTH TESTING

In dentistry, Phillips (1973) noted that the stress properties of denture base acrylic resins are generally tested by the use of a transverse test, according to the American Dental Association Specification No. 12 for denture base polymers. A specimen 2.5 mm thick, 10 mm in width and 65 mm in length is subjected to a transverse load at a specific loading rate to predict the performance of the resin. The load bearers consist of 3.2 mm diameter rollers, set with their centres 50 mm apart. The load is applied through a 3.2 mm diameter roller.

The modulus of rupture of dental porcelain is also determined using a transverse test. Skinner and Fitzgerald (1938), Sayre (1938), Sartori (1939), Moldal (1939), Hodson (1959), McLean and Hughes (1956), Southan (1968), Peet (1971) and Adair (1971) used specimens of varying size to determine the modulus of rupture (transverse strength) of dental porcelains by the three point load technique. These workers obtained strength values between 3,700 and 12,000 p.s.i. (25.5 MPa - 82.8 MPa), depending on whether the porcelain was low, medium or high fusing.
Adair (1971) and Peet (1971) chemically treated some of their porcelain specimens by immersion in potassium nitrate at 500°C for varying times and when transverse tested, these specimens achieved strengths of up to approximately 18,000 p.s.i. (124 MPa) - two or three times greater than their untreated transverse strength.

Asgar and Peyton (1962) used a three point load microbend tester devised by Flinn and Trojan (1955) to investigate the behaviour of different phases of cobalt-base and gold alloys used in dentistry for partial denture frameworks when subjected to bending stresses.

These workers used specimens of one inch (25.4 mm) length and a square cross-section of 1/16th inch (1.6 mm), and noted that for cobalt chromium the presence of micro-porosities in castings was the initiation point for fractures when specimens were bent. If no porosity was present fracture initiated at the grain boundary.

Gabel (1954) points out that when the modulus of elasticity of the material of a Class II restoration is higher than that of tooth structure, the restoration functions as a beam. Because of end movements, the gingival portions of these restorations must be locked mechanically to prevent their rotation out of the cavity. In locking the gingival portions of these restorations they become curved beams whose bending stresses at the axio-pulpal line angles increase rapidly with a decrease
in the radius of curvature of the angles.

What takes place in the tooth may be represented by Figure 4-1, p.78.

In any Class II restoration, the bending stresses produced and the retention required depend on how much the modulus of elasticity of the material exceeds that of tooth structure.

Gabel states that casting golds have a modulus of elasticity as much as ten times that of dentine and speculates "that even the modulus of elasticity for amalgam may be higher than that of deciduous dentine which may account for the failure of the Class II amalgam restoration in deciduous teeth".

Mahler and Mitchem (1964) spoke of the "edge strength" of a restorative material and thought this to be best represented in engineering terms by bending or transverse strength. Since amalgam has a low elongation (Rodriguez and Dickson, 1962) when subjected to reasonably rapid rates of loading, Mahler and Mitchem (1964) designed their transverse test after the method of Gray (1948) for testing brittle materials in engineering practice. The 1 mm x 4 mm x 12 mm specimens were supported by two 0.04 inch (1 mm) diameter rods set 10 mm apart. Loading was achieved through a 1/8 inch (3.18 mm) diameter steel
Fig. 4-1. Diagrammatic representation of a possible three-point loading situation with respect to a dental restoration.

(After Gabel, 1954)
ball travelling at a rate of 0.05 inches (1.27 mm) per minute, and the transverse strength was computed by the formula

\[ T = \frac{31P}{2bh^2} \]

where

- \( P \) = fracture load
- \( l \) = sample length between the supports
- \( b \) = sample width
- \( h \) = sample thickness

Mahler and Mitchem tested 140 specimens using New True Dentalloy\(^1\) - a fine grained alloy. The mercury:alloy ratio selected was 4:1 and the mix was amalgamated without a pestle in an S. S. White Amalgamator for five seconds. Three precondensation mercury ratios, 65%, 60% and 50%, were achieved by expressing excess mercury onto a Torsion balance. The amalgam was condensed in the conventional way within four minutes with lighter loads being used for the higher mercury ratios, in order to produce specimens with a residual mercury content ranging between 42% and 63%. All specimens were tested at seven days.

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Iwaku et al (1966), following the reasoning of Mahler and Mitchem (1964), transverse tested six materials, in an attempt to determine their edge strength. The materials tested were:

- Powdered gold A
- Powdered gold B
- Powdered silver
- Foil gold
- Amalgam
- Acrylic resin.

Specimens 1 mm x 3 mm x 5 mm were prepared and tested in a three-point load system with the centres of the supports 3 mm apart. The load was applied at a rate of 125 gm/second. These workers found similar bending strengths for both gold foil and amalgam, materials which are generally believed to be quite different in clinical edge strength performance. They suggested there must be some other technological property to represent edge strength better and considered this to be represented best by the maximum bending of specimen prior to fracture.
Transverse strength testing has been used relatively infrequently in investigations of dental amalgam. Some of the reasons for this are:

(i) the relative complexity of the test compared with compressive strength and diametral compression testing;

(ii) the size of specimen required, and

(iii) difficulty of preparation of specimens which relate to the clinical handling of the material.

Mahler and Mitchem (1961, 1963, 1964) have used this method of testing in an attempt to relate mechanical properties to clinical failure of amalgam restorations.

Mahler and Mitchem (1961) and other workers found that the compressive strength of fully set dental amalgams was not appreciably affected by variation in alloy:mercury ratio. Since a large number of marginal failures of restorations occurs in clinical practice, it was considered that a transverse test would be more predictive of this type of failure.

The effect of variation in alloy:mercury ratio with respect to transverse strength was tested using specimens 1 mm x 4 mm x 12 mm in dimension. Alloy:mercury ratios used were 1:1, 1:1.4, 1:2. and 1:4. These workers found
no differences in transverse strength, provided that the same amount of mercury was present in the precondensation mix. They found the transverse strength of the fine cut alloy tested to be of the order of 17,000 p.s.i. (117.2 MPa), about twice the tensile strength and about one-third the compressive strength of the alloy.

In a later paper, Mitchem and Mahler (1963) found that all strengths decrease with an increasing percentage of residual mercury and that the strength decrease has an increased rate change at approximately 57% residual mercury for the alloy studied and for all strengths tested: compressive, tensile and transverse. A further group of 40 specimens of fixed mercury content was tested to determine the effect of rate of specimen loading. Loading rates of 0.001 (0.025 mm), 0.003 (0.076 mm), 0.010 (0.25 mm), 0.1 (2.54 mm) inches/minute were used.

Mahler and Mitchem (1964) found that as residual mercury content increased, the strength decreased, particularly at mercury contents above 54% (for the alloy tested). This is in keeping with the results of Swartz and Phillips (1956).
Mahler and Mitchem (1964) found good correlation between compressive, tensile and transverse strengths in relation to residual mercury content as shown in Fig. 2-3, p. 47. They point out that the similarity of these curves indicates that the strength of amalgam may be represented by either of the three strength tests. 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) used a Flinn strain-viewer to observe the effect of bending strains on dental amalgam specimens. Because of the brittle nature of amalgam, the concept of a two-phase material was applied to restrict crack propagation. In this system a strong, rigid material (amalgam) is matched with a weaker but more ductile one (self curing resin). The ductile material absorbs the strain energy released when an individual fibre of the brittle material fails and transfers the stresses to the remaining fibres of the brittle phase.

Amalgam specimens 11/16 inch long (17.5 mm) and 1/16 inch (1.58 mm) square were prepared using Caulk's\textsuperscript{1} 20th Century Fine-Cut Alloy, triturated for twelve seconds in an S. S. White Amalgamator, then condensed into a three-piece mould.

\textsuperscript{1} L. L. D. Caulk Co., Milford, Del., U.S.A.
with a 1 mm diameter plugger. At twenty-four hours, the specimens were embedded in a self curing resin, Bioplastic,¹ and shaped after a further two days. A slow load was applied to the beam until visible microscopic cracks occurred, in about thirty minutes. These small cracks occurred in a series, were unconnected, and in areas other than that in which the major crack appeared. After initial failure, the specimens were cyanide etched and remounted to reveal grain structure. Analysis showed that, in order, the cracks pass most frequently through voids, gamma-2 phase, gamma-1 phase; with the gamma phase being the most resistant. Usually the crack passes around the gamma phase and in none of the specimens did the crack originate in the gamma phase. Once the crack has progressed a considerable distance, the gamma phase is not spared as the crack progresses through it.

In summary, Asgar and Sutfin suggested that the properties of dental amalgam may be determined by the impurities segregated at the grain boundaries of the amalgam.

Mahler et al (1970, 1973) attempted to relate the mechanical properties of three amalgams to their observed clinical marginal fracture performance, with time. The

¹. Ward's Natural Science Establishment, Rochester, New York, U.S.A.
amalgams used in these studies were Dispersalloy,¹ New True Dentalloy,² Twentieth Century Micro.³ Black and white photographs at one year and up to four years were used to assess clinical performance. These workers found that no apparent relationship between the compressive strength, tensile strength, transverse strength, transverse deflection, American Dental Association flow, and marginal fracture under clinical conditions appears to exist, whereas the properties of dynamic creep, static creep, and slow compressive strength do appear to relate to clinical marginal fracture to some degree. They concluded that the rheological properties of dynamic creep, static creep and slow compressive strength appear to predict clinical marginal fracture whereas other mechanical properties are not useful in this predicting role.

Basker and Wilson (1970) investigated the effects of packing force, rate of packing, and packing time on the one-hour transverse strength of amalgam. Specimens were made using S. S. White's New True Dentalloy,⁴ manipulated using the Eames' technique (Eames, 1959) to diminish variability.

1. Western Metallurgical Ltd., Edmonton, Saskatchewan, Canada.
3. L. D. Caulk Co., Milford, Delaware, U.S.A.
The transverse testing jig had steel supports of 1.5 mm diameter set with their centres 10 mm apart. The loader was a centrally located 1.5 mm diameter roller, activated with a cross-head speed of 0.5 mm/minute. Packing forces of 10N - 20N and rates of 120 - 200 thrusts/minute were used.

Basker and Wilson (1970), in agreement with one-hour compressive strength results presented by Wing (1965), found that an increase in packing force from 10N - 20N resulted in an increased strength of the order of 20%. An increased packing rate from 120 - 200 thrusts/minute increased the strength by 8% and an increase in packing time from one minute to two minutes showed a significant increase in specimen strength.

Turchyn and Youdelis (1970) considered that the diametral test results in a small but definite flattening of the cylindrical specimen due to plastic flow. Once this flattening occurs, constraining forces develop at the surface, which are transmitted into the specimen counter-acting the resolved tensile stresses that fail the specimen in tension. Consequently, a larger compressive load is required to fail the specimen, yielding a higher apparent tensile strength, particularly for the softer amalgams. Consequently, these workers used a three-point load
transverse test to determine the flexure strength of cylindrical 4 mm x 12 mm specimens. Their results showed that the flow characteristics of amalgam preclude the accurate determination of either tensile or flexure strength. In both tests, plastic deformation of the specimen decreases the actual tensile stresses and accordingly a higher load must be applied to achieve fracture resulting in higher apparent tensile and flexure strengths.

Recently, Forsten (1969, 1970 a, b; 1971, a,b,c,d; 1972, a, b) has devoted his research efforts almost entirely to the investigation of the transverse strength of dental amalgams. In his early papers, Forsten (1969, 1970, a, b) described a method for measuring transverse strength and observed the effect of delayed condensation on the transverse strength of various fully set amalgams. Forsten 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. Forsten (1971, a) investigated the influence of manipulation technique on the one-hour strength of different types of amalgam. A 'dry' method and Jørgensen's technique of excess initial
mercury, were used. When tested at a cross-head speed of 5.5 mm/minute, the 'dry' technique yielded slightly better one-hour strength than the wet technique.

Forsten (1971, b) investigated the influence of different amalgamators, pestles and trituration times on transverse strengths of different types of amalgams. The effect of initial minimum or excess mercury ratios was also studied.

Specimens 2 mm x 2 mm x 12 mm were used and transverse testing was carried out at seven days, using a cross-head speed of 0.05 mm/minute and a three-point testing jig with the supports set 10 mm apart.

Forsten gave five reasons for the choice of a transverse strength test:

1. The 'clinical' fracture of amalgam, and the breakage of 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 cross-head speed.

3. Specimens of 'clinical' size and shape may be used.

4. As the width and depth of the cavity mould are close to those of 'clinical' cavities, condensing
procedures very similar to clinical conditions may be used.

5. It may be expected that improper trituration, which often results from alloy particles remaining unwetted by mercury, from only partly broken down alloy pellets, or from microporosities or large internal voids in the filling, will show up better in this type of test than when a compressive test is used.

Forsten tested three alloys: a fine cut lathe (Caulk fine cut\textsuperscript{1}), a dispersion modified (Dispersalloy\textsuperscript{2}) and a spherical (Spheraloy\textsuperscript{3}), and achieved maximum strength values when a high-speed amalgamator was operated for 11-16 seconds and when an ultra-high-speed mixer was operated for 6-11 seconds. A medium sized cylindrical metal pestle was found to be the most efficient and when two different precondensation mercury ratios were used, the higher precondensation mercury content seemed to facilitate the mixing of the fine-cut alloy only, because of the higher surface area to volume ratio for this type of alloy.

1. L. D. Caulk Co., Milford, Delaware, U.S.A.
2. Unitek Co., U.S.A.
Forsten (1971, b) also noted that although Dispersalloy showed the lowest transverse strength of the amalgams tested, it was the least susceptible to manipulative variables. In a related paper, Forsten (1971, c) investigated the influence of trituration variables on the transverse strength of preamalgamated alloys in filing form and found that the alloys behaved in a similar manner to those tested in the earlier work (1971, b).

Forsten (1972, a) investigated the influence of changing the precondensation mercury on the early and final transverse strength of several amalgams of different composition. Initial mercury contents of 50%, 54%, 59% were used to prepare 2 mm x 2 mm x 12 mm transverse specimens which were tested at one hour or one week. Overall he concluded that it was best to use a moderate excess of initial mercury in clinical work than to strive for an initial minimal mercury content because, when a 'dry' mix is used, the amalgam is more sensitive to human variables during condensation.

Forsten (1972, a) considered that a moderate excess of precondensation mercury does not seem to decrease the early strength of amalgams, but does tend to increase the final strength. He suggests that, clinically, excess precondensation mercury should be used.
Forsten (1972, b) studied the influence of precondensation mercury content and mulling on the final transverse strength of amalgams condensed after a delay. He found that following a precondensation delay of five minutes, most specimens tested at one week, showed a diminished transverse strength of from 1% to 42%, depending on the alloy used. This is in agreement with earlier work by Phillips et al (1965). The effect of increasing the precondensation mercury was to decrease the effect of the five minute delay on the final strength. "Mulling" the amalgam mix also tended to diminish the effect of the precondensation delay. Again, Dispersalloy tended to be the amalgam least sensitive to manipulative variables, being little effected by a precondensation delay or mercury content variation.

In closing, Forsten (1972, b) concluded that during periods where long condensation time is required, the use of fast setting amalgams with an initial mercury content exceeding 60%, or even preamalgamated amalgams with more than 55% initial mercury content may be advisable. Routine use of too large an excess of initial mercury in clinical practice should be avoided due to the inconvenience of handling a "sloppy" mix and because of the possibility of mercury contamination of the office.
Forsten's concept of excess precondensation mercury somewhat parallels that of Wing (1971) who, in stressing the need for adequate mercury removal, said "irrespective of the technique employed, amalgam alloy chosen, original mercury:alloy ratio used, trituration carried out, and condensation performed, a satisfactory clinical restoration cannot be placed unless mercury removal is conscientiously carried out during the building of the restoration and after completion of condensation".

Younis, Asgar and Powers (1975) studied the force required to initiate a crack in dental amalgams and related this to microstructure and specimen age. These workers used a composite beam attached to a Flinn Strain Viewer and tested specimens of Optaloy,\textsuperscript{1} Spher-A-Cap,\textsuperscript{2} Dispersalloy\textsuperscript{3} and an experimental alloy\textsuperscript{4} at two days and four months, by a method described previously (Asgar and Sutfin, 1965; Allen, Asgar, Peyton, 1965). The gamma-2 phase was found to be the least resistant to crack initiation, followed by the gamma-1 phase and gamma phase with a relatively higher percentage of gamma-1 and gamma phases present.

1. L. D. Caulk Co., Milford, Delaware, U.S.A.

2. Kerr Manufacturing Co., Romulus, Michigan, U.S.A.


4. All spherical alloys were composed of the following: Ag-Cu spherical particle: Ag, 72%, Cu 28%; Ag-Sn spherical particle: Ag 70%, Sn 27.5%, and Cu 2.5% proportioned to one-third Ag-Cu to two-thirds Ag-Sn.
In some cases in the work of Younis et al, using the gamma-2 containing amalgams, two cracks initiated, one in the gamma-2 phase and the other in the gamma-1 phase. The gamma-2 initiated crack ceased when encountering a gamma particle, indicating the greater strength of the gamma phase. It was suggested that although the gamma-2 phase is weaker than the gamma-1 phase, the difference in strength between them may not be great. However, as no indication is given in this paper as to which crack initiated first, it might be that the gamma-2 phase crack initiated first, encountered the gamma particle which halted that crack, then as the tensile stress increased, the gamma-1 phase crack initiated and continued because of the relatively high percentage of this phase in the amalgam. Therefore, it seems unreasonable to assume that the gamma-2 and gamma-1 phases are of comparable strength, particularly as the gamma-2 phase occurs in low percentage and usually in islands.

In the Dispersalloy and experimental amalgams tested in this study, the crack initiated in the gamma-1 (Ag-Hg) phase in all but one case, in which the crack initiated through the gamma (Ag-Sn) phase because this was the only choice according to these workers. The possibility
of crack initiation in the dispersed phase (Ag-Cu or Cu-Sn) was not considered.

It seems difficult to comprehend the reason given for this isolated case of gamma phase initiation because it is unreasonable to assume that the tensile surface consists entirely of gamma (Ag-Sn) particles, or Ag-Cu or Cu-Sn phases, without any interconnecting gamma-1 matrix phase. It was later indicated that the difference in strength between the gamma (Ag-Sn), Ag-Cu, Cu-Sn and Ag-Hg phases is great, with the Ag-Hg phase being the weakest.
CHAPTER 5

MICROSTRUCTURE OF DENTAL AMALGAM

The relationship between microstructure and mechanical properties has generally been assumed to be of great significance. The properties of individual phases within amalgam, and the relationship between these phases, will have an effect on the mechanical properties of amalgam. Amalgams which contain a high percentage of the strong phases may be assumed to be stronger than those containing lower percentages of the strong phase.

Until very recently research into the microstructure of dental amalgam has largely been concerned with amalgams produced from alloys containing Silver-Tin-Copper-Zinc in a formulation similar to the "Balanced Alloy" of G. V. Black (1896, 1908). These alloys are based principally on the intermetallic compound Ag₃Sn with small inclusions of copper containing compounds. The position occupied by zinc in the original amalgam alloy has not been well defined. More recently with the introduction of the "Dispersion Modified" amalgams, following the work of Innes and Youdelis (1963) and the introduction of ternary single melt high copper alloys (e.g. Sybraloy,¹ Tytin,²), the knowledge of

1. Sybron-Kerr, Romulus, Ann Arbor, Michigan, U.S.A.
microstructure of dental amalgam based on three-quarters of a century of research is suddenly inadequate for the needs of other materials. With the introduction of the newer materials, new phases with different mechanical properties have become an important consideration in the behaviour of dental amalgams.

"Ag₃Sn" Amalgams – Structure and Setting Reaction

Earlier workers, prior to 1960, in attempting to demonstrate the nature of set amalgam and explain the setting reactions of this material produced micro-structural pictures which depended largely on conjecture for interpretation. Rosenhain (1926) showed what appeared to be a two-phased structure, with one phase completely etched out in his microstructural pictures. Ryge et al (1952) and Smith et al (1953) showed amalgams to consist of a matrix and original alloy particles. No positive identification of phases present was possible using the metallographic technique employed by these workers. Schmitt (1960) claimed to be able to distinguish phases present in the matrix of set amalgam but again a good deal of interpretational conjecture was necessary.
Gayler (1937) and Troiano (1938) had earlier advanced theories of the setting reactions in Ag₃Sn amalgams and had concluded that set amalgam consisted of a mixture of three phases:

- Beta-1 - Ag-Hg
- Gamma-1 - Ag-Hg
- Gamma-2 - Sn-Hg

Further researchers (Frankel and Fankuchen, 1952; Ryge, Moffett and Barkow, 1953; Taylor, 1963; Fairhurst and Ryge, 1963) using x-ray diffraction as the major research tool, have established the composition of the gamma-1 and gamma-2 phases with reasonable certainty. The compositions now accepted are gamma-1 (Ag₂Hg₃), and gamma-2 (Sn₇Hg or Sn₈Hg). None of these workers demonstrated the presence of the beta-1, Ag-Hg phase.

The introduction of the use of diamond polishing pastes for metallurgical specimen preparation of set dental amalgams, and the use of a double etch technique using iodine and potassium cyanide, followed by potassium ferricyanide (Wing, 1961, 1962, 1965) led to the demonstration of grain boundaries within the matrix of the set amalgam, the differentiation between original alloy and matrix and the recognition of two phases within the matrix.
Differentiation between the silver-mercury (gamma-1) and tin-mercury (gamma-2) phases was difficult in the older lathe cut amalgams. Allan et al (1965) and Koran and Asgar (1967), using a different etching technique,

<table>
<thead>
<tr>
<th>Solution A</th>
<th>Solution B</th>
<th>Hypo Rinse</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 gm K$_2$Cr$_2$O$_7$</td>
<td>4 gm I</td>
<td>Na$_2$S$_2$O$_3$ 5H$_2$O</td>
</tr>
<tr>
<td>1 gm KI</td>
<td>96 ml ethyl alcohol</td>
<td>in H$_2$O</td>
</tr>
<tr>
<td>100 ml H$_2$O</td>
<td></td>
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</tbody>
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were able to demonstrate a difference between original alloy and matrix but assumed that voids in their microstructural pictures were the result of etching out of the gamma-2 phase.

The structure of dental amalgams has been studied by Jørgensen et al (1967, a & b, 1970) and conclusions reached concerning the nature of the gamma-2 (tin-mercury) phase in lathe cut amalgams. Jørgensen and Saito (1970), following theoretical reasoning, serial sectioning and chemical corrosion using sodium chloride and sodium citrate solutions, have concluded that the tin-mercury phase forms a continuous network throughout the body of dental amalgams. In considering the very extensive work of Jørgensen and his fellow workers, it must be realised that in general the method of amalgam preparation is such that the
proportion of the gamma-2 phase present in their photomicrographs is very much higher than that observed by most other workers.

The development of spherical amalgams by Demaree and Taylor (1962) and the use of these alloys for research has assisted in the more positive identification of the phases present. In a series of publications, Wing and Ryge (1965), Wing (1966, a & b, 1969, 1970, b, 1971,a, 1971, b) have shown clearly that voids may be clearly differentiated from the gamma-2 phase which is not removed during etching as claimed by Allan et al (1965) and Koran et al (1967). The presence of the three principal phases in dental amalgams has been confirmed by use of the electron probe microanalyser for qualitative analysis (Wing, 1966, a; Koran and Asgar, 1967; Johnson, 1967, a & b). The use of the electron probe analyser for quantitative analysis of dental amalgams (Mahler, 1975; Wing, 1975) has confirmed the presence and composition of these major phases of dental amalgams. Both Mahler and Wing found the presence of very small percentages of tin (less than 2%) within the Ag-Hg (gamma-1) phase.
Using x-ray diffraction methods, no evidence of a phase which could be designated as a beta-1 (Ag-Hg) phase has been detected in a wide range of commercial spherical or lathe cut amalgams or experimental silver-tin alloys reacted with mercury (Frankel and Fankuchen, 1952; Ryge et al, 1953; Wing and Ryge, 1965, a; 1965, b).

Johnson (1967, a & b) has claimed to demonstrate the conversion of the gamma-1 (Ag-Hg) phase to the beta-1 (Ag-Hg) phase at temperatures of 60°C and, to a lesser extent, with time at 37°C (Johnson, 1967, b). The evidence presented by Johnson in these two papers is somewhat inconclusive despite the use of metallography, x-ray diffraction and electron probe microanalysis. Although Mahler et al (1973) tended to agree with Johnson's findings regarding the transformation of gamma-1 to beta-1 in clinical amalgams, the same workers (Mahler et al, 1975) in their detailed quantitative analysis, do not designate a phase in the set amalgam as beta-1 (Ag-Hg).

Copper which is present in small quantities in most commercial amalgams, plays little part in the setting reaction of Ag₃Sn type amalgams. In lathe cut amalgams, where the particles are produced following homogenization of the bar, before cutting on a lathe and ball milling,
the presence of copper rich areas within the original lathe cut particles and matrix of set amalgams was suggested by Wing (1961, 1962, 1965, a) following optical microscopy. This has been confirmed qualitatively where use was made of an electron probe (Johnson et al, 1969; Wing, 1970, a; Johnson, 1972; Wirjosumarto et al, 1973, Bryant, 1979). The composition of this copper rich phase has now been determined to an almost universal level of acceptance as Cu₃Sn, following quantitative analysis, using different electron microanalyser techniques (Mahler, 1975; Wing, 1975; Marshall et al, 1977).

Because they have not been subjected to homogenization heat treatments after atomization, spherical amalgams contain copper widely spread through the whole of the spherical particles, rather than being present in discrete areas of Cu₃Sn as in lathe cut amalgams. Spherical particle alloys are similar to the "as cast" condition of the ingot for the formation of lathe cut alloys.

"Ag₃Sn" type amalgams have now come to be generally accepted as consisting of a mixture of –

(i) silver-tin (gamma) original alloy particles;
(ii) silver mercury (gamma-1) matrix and a
(iii) tin-mercury (gamma-2) phase with the presence of
(iv) copper-tin (Cu₃Sn) inclusions within the original
alloy particles of lathe cut amalgams and within the matrix of these amalgams following trituration and condensation which together bring about fracture of the original alloy particles.

Dispersion Modified Amalgams - Structure and Setting Reactions

These amalgams developed by Innes and Youdelis (1963) consist of approximately 70% by weight of "Ag₃Sn" type particles and 30% by weight of silver copper eutectic as a dispersed phase. Although the original inventors introduced the material for reasons of "dispersion strengthening" and, in particular, increasing the compressive strength, this type of amalgam has not been shown to exhibit significantly increased strength. The mechanical property of creep is significantly improved compared with "Ag₃Sn" type amalgams. The principal effect produced by the dispersion modification, lies in the structure modifying effects of the dispersed material, particularly from the point of view of removal of the tin-mercury (gamma-2) phase.

The structure and setting reactions of Dispersion Modified amalgams have been extensively studied by a number of workers and the nature of these amalgams when set is
generally accepted. Wing (1970, 1975, a & b), Mahler et al (1975), Wing et al (1976), Wing (1977), Marshall et al (1977), Okabe et al (1977), Takatsu et al (1977), Wing (1978) and Bryant (1979), using optical microscopy and electron probe microanalysis, have established that following trituration of this type of amalgam, the reaction of the \( \text{Ag}_3\text{Sn} \) component with mercury, produces the gamma-2 (tin-mercury) phase but at some stage during setting, the tin from the gamma-2 phase diffuses into the spherical dispersed silver-copper eutectic to produce a peripheral zone of these spheres of a mainly tin-copper compound at the composition \( \text{Cu}_6\text{Sn}_5 \). The setting reaction is thus a two-stage reaction and the final structure following the second stage consists of:

(i) a silver-mercury (gamma-1) matrix with

(ii) particles of silver-tin (gamma Ag\( _3\)Sn) original alloy,

(iii) \( \text{Cu}_3\text{Sn} \) from the original \( \text{Ag}_3\text{Sn} \) alloy, and a

(iv) halo of the newly formed copper-tin (Cu\( _6\)Sn\( _5 \)) phase surrounding

(v) the original silver-copper (eutectic) spheres and within those spheres.

The tin-mercury (gamma-2) phase is almost entirely absent from this type of amalgam although Sarkar and Greener (1972, a, 1972, b) claimed that it is present in very small quantities following electrochemical studies.
Ternary High Copper Amalgams

The original alloys:

The alloys from which these amalgams are produced consist of spherical particles, which have been formed by an atomization process and which generally contain a mixture of silver, tin and copper (and sometimes zinc) as a result of a single melt process. Although a large number of this type of alloy are now available, Sybraloy$^1$ and Tytin$^2$ provide the basis for most commercially available alloys.

The compositions of these original alloys are approximately:

Sybraloy
- $\text{Ag} - 40\%$
- $\text{Sn} - 30\%$
- $\text{Cu} - 30\%$ by weight

Tytin
- $\text{Ag} - 60\%$
- $\text{Sn} - 25\%$
- $\text{Cu} - 15\%$ by weight

An alloy which represents a departure from Sybraloy and Tytin is Indiloy$^3$, which has a composition basically similar to that of Tytin, but containing 4% Indium at the expense of each of the other metals.

Indiloy
- $\text{Ag} - 59\%$
- $\text{Sn} - 24\%$
- $\text{Cu} - 13\%$
- $\text{In} - 4\%$

by weight.

1. Sybron-Kerr, Romulus, Ann Arbor, Michigan, U.S.A.
Structure and Setting Reaction:

The single phase high copper ternary amalgams are generally regarded as being of the non gamma-2 type although the setting reaction has not at this stage been well defined. Because of the nature of the original alloy, i.e. a single phase ternary alloy, it is unlikely that the setting reaction is of the same type as that in the Dispersion Modified type of amalgam where the reaction occurs in two stages to reach the final structure.

In S.E.M. pictures and optical micrographs (Okabe et al, 1978; Marshall et al, 1977; Wing et al, 1976; Wing, 1977 and Bryant, 1979), the structure of the set amalgams may be seen to consist of original spherical particles in which zones of reaction may be observed, a matrix with an appearance similar to that of the gamma-1 (silver-mercury) matrix of \( \text{AgSn} \) and dispersion modified type amalgams, and a phase within the matrix with a similar appearance to the gamma-2 (tin-mercury) phase of \( \text{Ag}_3\text{Sn} \) type amalgams. Electron probe analysis using an energy dispersive x-ray spectrometer system (Okabe et al, 1978; Marshall et al, 1977) have confirmed the presence of some unreacted original alloy at the original ternary composition and a gamma-1 matrix composition similar to that found in \( \text{Ag}_3\text{Sn} \) and Dispersion type amalgams.
The other reaction phase was analysed at a composition of approximately Cu$_6$Sn$_5$, similar to the reaction zone found in Dispersalloy. Using a wave length dispersive electron probe microanalyser, Mahler and Adey (1977, 1978) came to the conclusion that the reaction phase is a mixture of Cu$_6$Sn$_5$ and gamma-1 and possibly original alloy which could not be separated by optical or electron microscopy. These workers used a technique of setting aside amounts of silver and mercury in an assumed ratio and an allowance for artefact production. The high copper alloy investigated by Mahler et al was similar in composition to Tytin. Wing (1977, 1978, 1979), by combining optical microscopy and electron probe microanalysis using a wave length dispersive electron probe microanalyser for quantitative analysis, claims that amalgams produced from Sybraloy contain considerably more of the phase than is present in Tytin amalgams. This is consistent with the microstructural pictures of Okabe et al (1978). Wing fixes the composition of this phase, which is similar in appearance to gamma-2, at approximately:

- silver: 2 per cent
- tin: 10 per cent
- copper: 32 per cent
- mercury: 16 per cent.

After allowing for the possible presence of some gamma-1
(silver-mercury) phase he suggests that this may be a ternary tin-copper-mercury compound. Wing and Blackler (1978) showed that the mechanical properties of this phase are different from those of gamma-2. An investigation of microhardness of the phases present showed that the tin-copper-mercury areas have a D.P.N. hardness of 60-80 compared with the hardness of gamma-2 of D.P.N. 15. Wing (1979) showed that the gamma-1 matrix of the ternary high copper amalgams is similar to the matrix of Ag₃Sn and Dispersion amalgams but with a slightly higher copper content of approximately 1.5 - 2.0 per cent. The copper concentration in the gamma-matrix of Sybraloy amalgams has been found to be slightly higher than in Tytin amalgams.

In summary, the final structure of ternary high copper amalgams may be considered to be made up of:

(i) original ternary alloy

(ii) a gamma-1 type matrix

(iii) reaction zones closely related to the original alloy at a composition Cu₆Sn₅

(iv) a reaction phase containing tin, copper, mercury and very small amounts of silver, the exact form of which is not determinable but which almost certainly is formed as part of the primary setting reaction.

The setting reaction is almost certainly a single stage reaction.