

**THE EVALUATION OF THE APPLICABILITY OF USING GOLD PLATING
TO ENHANCE THE PREDICTABILITY AND STRENGTH OF BASE METAL
POSTCERAMIC GOLD SOLDER JOINTS WHEN USING A FLUX THAT
DOES NOT DISCOLOUR THE PORCELAIN**

by

JOHN NEIL WADDELL

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POSTCERAMIC GOLD SOLDER JOINTS WHEN USING A FLUX THAT
DOES NOT DISCOLOUR THE PORCELAIN

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I, John Neil Waddell, declare that this dissertation represents
my own work, both in conception and execution.

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DEDICATION

This study is dedicated to my saviour,
the
LORD JESUS CHRIST
through whom all things are possible.

ABSTRACT

The unpredictability of solder joints in dental base metal alloys constitute a major problem. This study aimed to establish whether gold plating the joint surfaces of a metal ceramic base metal alloy prior to postceramic soldering would enhance the predictability of the soldering method and joint strength.

150 standardised test specimens were prepared from 3 Degussa dental alloys, viz Resistal P (NiCr), Degulor M (AuPt) and Realor (PdAu). The Resistal P specimens were subjected to 6 porcelain firing cycles and solder joint surfaces of 40 specimens gold plated prior to soldering, using gold plating equipment and solutions found in the jewellery industry. Suitable fluxes were used and all soldering was done in a porcelain furnace.

First the soldering method was validated by soldering 20 Degulor M specimens and determining the tensile strength of the 10 joints in an Instron testing machine. The data were then compared with the ISO minimum standards. The soldering method proved sound and a control was thus established.

Secondly, using 3 alloy combinations without gold plating, 30 solder joints were made and their tensile strengths determined. This established the level of predictability and strength and parameters for comparison.

Thirdly, the above procedure was repeated, but the Resistal P specimen joint surfaces were gold plated prior to investing for soldering. The levels of predictability and strength were then compared with the parameters set.

The fracture sites of broken joints were examined and photographed using a metallurgical microscope.

The data were analysed using 4 statistical tests. The Degulor M control group solder joints were the strongest and the Resistal P joints the weakest. The success rates for the non-plated joints were; Degulor M to Degulor M = 100%, Resistal P to Degulor M = 90%, Resistal P to Realor = 80% and Resistal P to Resistal P = 60%. The success rates for the gold plated joints were; Resistal P to Degulor M = 60%, Resistal P to Realor = 40% and Resistal P to Resistal P = 80%.

This study found, under the circumstances of testing, that gold plating of the base metal alloy prior to investing for postceramic soldering did not enhance the strength, solderability and predictability of the solder joints to an extent that a flux that does not discolour porcelain can be used.

OPSOMMING

Die onvoorspelbaarheid van soldeerlasse in tandheelkundige onedel metaallegerings is 'n groot probleem. Hierdie studie is onderneem om vas te stel of die voorspelbaarheid van die soldeermetode en sterkte van die las verhoog kon word deur die lasoppervlak van 'n onedel metaallegering met goud te plateer voordat dit gesoldeer word.

150 gestandaardiseerde toetsmonsters is vervaardig uit 3 Degussa tandheelkundige legerings, naamlik Resistal P (NiCr), Degulor M (AuPt) en Realor (PdAu). Die Resistal P-toetsmonsters is onderwerp aan 6 porselein-baksiklusse. Die lasoppervlakke van 40 toetsmonsters is voor soldering met goud geplateer met behulp van goudplateringstoerusting wat in die juweliersbedryf gebruik word. Geskikte vloeimiddels is gebruik en al die soldeerwerk is in 'n porselein-hoogoond gedoen.

Eerstens is die soldeermetode getoets deur 20 Degulor M-toetsmonsters te soldeer en die treksterkte van 10 lasse in 'n Instron-toetsmasjien te bepaal. Die data is toe met die ISO minimum standarde vergelyk. Die soldeermetode is as doeltreffend bewys en 'n kontrole is sodoende daargestel.

Tweedens is 30 soldeerlasse uit 3 legeringskombinasies gemaak sonder om goudplatering te gebruik. Die treksterkte daarvan is bepaal. Op hierdie manier is die vlak van voorspelbaarheid en sterkte vasgestel, en die parameters vir vergelyking daargestel.

Derdens is bovermelde prosedure herhaal, maar die Resistal P-toetsmonsters se lasoppervlakke is met goud geplateer voordat dit gesoldeer is. Die vlakke van voorspelbaarheid en sterkte is toe met die gestelde parameters vergelyk.

Die breekplekke in die lasse is ondersoek en met behulp van 'n metallurgiese mikroskoop gefotografeer.

Die data is toe deur middel van 4 statistiese toetse ontleed. Die lasse van die Degulor M-kontrolegroep was die sterkste en die Resistal P-lasse die swakste. Die persentasie sukses vir die nie-geplateerde lasse was: Degulor M aan Degulor M = 100%; Resistal P aan Degulor M = 90%; Resistal P aan Realor = 80%; en Resistal P aan Resistal P = 60%. Die persentasie sukses vir die goudplateerde lasse was: Resistal P aan Degulor M = 60%; Resistal P aan Realor = 40% en Resistal P aan Resistal P = 80%.

Daar is in hierdie studie bevind dat, onder die toets-omstandighede, die goudplatering van onedel metaallegerings voor soldering nie die sterkte, soldeerbaarheid en voorspelbaarheid van die soldeerlasse in so 'n mate verhoog dat 'n vloeimiddel wat nie die porselein verkleur nie gebruik kan word nie.

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CHAPTER ONE

THE PROBLEM AND ITS SETTING

1.1 INTRODUCTION

The costs of production in metal ceramic dental laboratories have risen sharply in recent years. One of the main contributory factors has been the spiralling price of precious metals used for the metal ceramic substructure, but the use of base metal alloys has contributed significantly to reduce these escalating costs [Tuccillo and Cascone, 1983:367]. The practice of postceramic soldering, whether this involves soldering of metal ceramic alloys or soldering a metal ceramic alloy to a precious alloy all metal abutment, is common in the construction of multi-unit restorations. The postceramic soldering of precious metal alloys, while utilising a flux that does not discolour the porcelain, generally produces results that are satisfactory and predictable. However, the unpredictability of the solder joints in base metal alloys incorporates an element of risk. This is especially the case when utilising a flux that does not discolour the porcelain. Often the failure of the solder joint only manifests itself after cementation in the patient's mouth. This leads to costly remakes as well as considerable stress to the patient. The result is that many dental ceramists prefer not to use base metal alloys when constructing multi-unit

restorations, thus forfeiting the favourable qualities of high elastic moduli, high strength, high sag and creep resistance and low cost [Dudek, 1988:85].

It is common in the electronics industry to gold plate base metal connectors to enhance their solderability [Lee, 1971:997; and Metals Handbook Ninth Edition, Vol. 6, 1983:1075]. By applying the technique of gold plating the dental base metal joint parts prior to postceramic soldering, the dental technician should be able to use a potentially effective, time-saving alternative technique. Thus if the hypotheses in section 1.2.3 are supported, the metal-ceramist will have available to him a method that will produce a strong, predictable solder joint.

Dudek [1988:85-86] estimated that in the USA 50% to 70% of all fixed restorations were fabricated using base metal alloys. Based on a telephone survey of a leading dental supply company, it was found that the situation in South Africa is similar [Pert, 1992: Personal communication]. The cost-effective implications of the use of base metal alloys are extensive. The average cost per gram of a metal ceramic base metal alloy as at June 1992 was R1.60, while that of semi-precious metal ceramic alloys was R16.00 and precious metal ceramic alloys was R40.00. The fact that the ceramist can choose a less expensive alloy when faced with situations that require postceramic soldering will result in large savings for the industry and the patient.

It should be noted that this study is primarily concerned with establishing the effectiveness of the application of a well established soldering technique used in the electronics industry in order to solve a problem in the dental laboratory industry. Therefore the problem will be seen through the eyes of a dental technician and not from the point of view of an experienced metallurgist. The main aim will be to measure the strengths produced by the proposed soldering technique as compared to existing methods and at the same time to establish the predictability of the proposed technique.

1.2 THE PROBLEM AND ITS SETTING

1.2.1 PROBLEM STATEMENT

The purpose of this study is to evaluate the postceramic solderability of gold plated surfaces compared to non gold plated surfaces in the following selected alloy types commonly used in the dental industry:

- metal ceramic base metal alloys
- metal ceramic base metal to semi-precious alloys
- metal ceramic base metal to precious alloys.

1.2.2 SUBPROBLEMS

1.2.2.1 SUBPROBLEM ONE

The first subproblem is to establish the validity of the postceramic soldering technique when soldering precious to precious alloys in order to validate its use as a control for the selected alloys tests.

1.2.2.2 SUBPROBLEM TWO

The second subproblem is to establish the level of predictability and strength when soldering the alloy types without gold plating in order to identify parameters for comparison.

1.2.2.3 SUBPROBLEM THREE

The third subproblem is to establish the level of predictability and strength of the alloy types with gold plated joints for the purpose of comparing the results with the selected parameters.

1.2.2.4 SUBPROBLEM FOUR

The fourth subproblem is to integrate the results to establish whether gold plating the joint surfaces prior to investment for soldering will produce predictable, strong solder joints.

1.2.3 THE HYPOTHESES

1.2.3.1 HYPOTHESIS ONE

The soldering technique will produce predictable, strong solder joints when soldering the precious to precious alloys, thus validating the soldering method, and will establish a control.

1.2.3.2 HYPOTHESIS TWO

The selected alloy types that are soldered without gold plating the joints will not produce the same level of predictability and strength as the control, and will thereby set parameters for comparison.

1.2.3.3 HYPOTHESIS THREE

The selected alloy types that are soldered after gold plating the joints, will produce levels of predictability and strength that are suitable for comparison with the identified parameters.

1.2.3.4 HYPOTHESIS FOUR

It will be possible to integrate the results and to show that gold plating the joint surfaces prior to investment for soldering will produce predictable strong solder joints.

1.2.4 ASSUMPTIONS

1. Cost and speed of production will always be important factors in the choice of alloy in the dental technology industry. Therefore it is assumed that due to low cost and continued improvement, metal ceramic base metal alloys are becoming more acceptable to the dental profession.
2. Due to insufficient occlusal - gingival space in long span posterior metal ceramic bridge abutments, the posterior abutments are often made as an all metal crown and then postceramic soldered to the completed porcelain veneered section of the bridge. It is therefore assumed that whenever this situation arises there will be a need for a soldering technique that provides a strong, predictable joint.

3. The researcher did not have access to a gold plating bath which was being used to plate electronic solder connectors. Due to the fact that the mechanisms and principles of gold plating are the same as that which is found in the jewellery industry, it was assumed that the gold plating bath housed in the Department of Jewellery Design of Technikon Natal would be suitable.
4. The gold plating bath and solutions housed in the Department of Jewellery Design of Technikon Natal are similar to those used by the jewellery manufacturing industry in Natal with regard to type and cost of the plating bath and plating solution, and therefore it can be assumed that this will be in the price range that the average commercial dental laboratory can afford.
5. It is assumed that the manufacturer's recommended gap width of 2 mm between the two surfaces to be soldered is suitable [Degussa AG, 1980: Working Instructions, Ceramic Alloys]. Therefore no test was carried out to establish this parameter for the study [ISO 5179, 1983].
6. It is assumed that the flux recommended by the alloy manufacturer for base metal alloys will not discolour the porcelain and will facilitate adequate wetting by the recommended solder.

7. It is assumed that the gold plating process will not discolour the porcelain.

1.2.5 DELIMITATIONS

1. This study will not evaluate the applicability of the method with beryllium containing metal ceramic base metal alloys. Several studies have been done on the occupational health hazards affecting dental laboratory technicians working with beryllium containing metal ceramic base metal alloys. Goldman, Hartman and Messite [1984:12], stated that the melting, grinding, buffing and general lathing operations of beryllium containing base metal alloys can result in significant exposure to this highly toxic metal. Studies done by Covington, McBride, Slagle and Disney [1985:128], concluded that "nickel-beryllium dental casting alloys possess the potential to be a significant hazard to the laboratory technician, dentist, and patient", while Dudek [1988:92] predicted that beryllium containing alloys will eventually be phased or legislated out of use. At present, certain European countries (e.g. Germany) do not allow the sale of these alloys.

The position in South Africa at present is that there is no restriction on their sale. However, this could change in the future in line with international trends. For the foregoing reasons this study excluded beryllium containing metal ceramic alloys so as not to invalidate the long term applicability of its findings.

2. This study will not evaluate the applicability of the method for the soldering of metal ceramic precious and semi-precious alloys to metal ceramic base metal alloys although the findings of the study will have application.

1.2.6 DEFINITIONS OF TERMS AND CLARIFICATION OF CONCEPTS

1.2.6.1 METAL CERAMIC

Metal ceramic refers to the process of fusing porcelain directly onto a cast alloy [Phillips, 1982:369].

1.2.6.2 BASE METAL ALLOY

For the purpose of this study, base metal alloy refers to nickel-chromium metal ceramic alloys that do not contain beryllium. (Beryllium has the effect of improving the oxide scale adherence in the metal ceramic bond [Bertolotti, 1988], but the occupational health hazards associated with it, [Covington et. al., 1985], have resulted in it being outlawed in certain European countries).

1.2.6.3 SEMI-PRECIOUS ALLOYS

For the purpose of this study, semi-precious alloys refer to silver-palladium based alloys used for metal crowns, bridges and inlays. It must not be confused with palladium based metal ceramic alloys that contain silver.

1.2.6.4 PRECIOUS ALLOYS

For the purpose of this study, precious alloys refer to gold-platinum based alloys used for metal crowns, bridges and inlays. It must not be confused with gold-platinum metal ceramic alloys.

1.2.6.5 SOLDERING

Soldering is the joining of metals by the use of a filler metal which has a substantially lower fusion temperature than that of the metal parts being joined [Phillips, 1982:534].

Since 1990 this definition has been superseded by two definitions, namely *brazing* and *soldering*.

During brazing, metal parts are joined together by melting a filler metal between them at a temperature below the solidus temperature of the metal being joined and above 450°C [Phillips, 1991:529].

During soldering, metal parts are joined together by melting a filler metal between them at a temperature below the solidus temperature of the metal being joined and below 450°C [Phillips, 1991:529].

Due to the recent change in definition the terms soldering and brazing should be regarded as having the same meaning and as being interchangeable.

1.2.6.6 PRECERAMIC AND POSTCERAMIC SOLDERING

Preceramic soldering is the soldering of the metal ceramic substructure prior to the porcelain application (sometimes called presoldering), while postceramic soldering is the soldering of the metal ceramic substructure after the porcelain has been applied (sometimes called postsoldering) [Phillips, 1982:542].

1.2.6.7 SOLDERABILITY

Solderability refers to the ability of the solder to completely wet the alloy [Waclawsky, 1985:82].

1.2.6.8 WETTING

Wetting refers to the metallurgical process resulting in a uniform layer of solder which is firmly adherent to the base metal [Waclawsky, 1985:82].

1.2.6.9 TENSILE STRENGTH

Tensile strength is calculated by dividing the maximum load carried by the specimen during a tension test by the original cross-sectional area of the specimen [ASTM E 8M - 86a, 1984:206].

The minimum acceptable tensile strength is based on the ISO definition:

The maximum stress of the specimen shall exceed 350 MPa or the 0,2% proof stress of the weakest of the metallic parts [ISO 9333:1990(E):1].

CHAPTER TWO

REVIEW OF THE RELATED LITERATURE

2.1 INTRODUCTION

The review of the related literature will firstly describe the increasing use of base metal alloys in the dental technology industry and then look at the advantageous properties of base metal alloys as compared to the precious and semi-precious alloys.

This will be followed by an overview of the present position in the industry with regard to postceramic soldering showing that the postceramic soldering of precious and semi-precious alloys is well established with reliable predictable results. When the same soldering method is used with base metal alloys using a flux that does not discolour the porcelain the results are unreliable. This problem can only be overcome by using a flux that does discolour the porcelain or a soldering method that requires the preceramic wetting of the connector area with high heat solder prior to porcelain application.

The section on soldering in the electronics industry describes how the solderability of base metal alloys is enhanced by gold plating the surfaces prior to soldering.

The section on fluxes describes the fluxes used in the dental industry and the electronics industry and it also looks at how this very important part of the soldering process affects the ability of the solder to wet the joint surface.

2.2 BASE METAL ALLOYS

2.2.1 USE OF BASE METAL ALLOYS IN DENTISTRY

The costs of production in a metal ceramic dental laboratory have risen sharply in recent years. One of the main contributory factors has been the spiralling price of precious metals used for the metal ceramic substructure. The use of base metal alloys has gone a long way to reduce this cost. [Tuccillo and Cascone, 1983:367]. According to Dudek [1988:85], base metal alloys are the single most popular class of alloy for ceramic bonding used today. He estimates that currently 50% to 70% of all crowns and bridges in the USA are fabricated using base metal alloys. Although the primary advantage is the low cost of these alloys compared to the noble metal ceramic alloys, the widespread use of base metal alloys is supported by the current high degree of success experienced clinically and during dental laboratory processing. In recent years, alloys with improved properties and more predictable dental laboratory handling characteristics have come onto the market. An example of this is the Wiron alloys produced by Bego; the original Wiron 77 had a ductile yield of 4.5% and a Vickers hardness of 270. This was later followed by the development of Wiron 88 with the improved

ductile yield of 15% and a Vickers hardness of 200. At present their latest base metal alloy is Wiron 99 with a ductile yield of 25% and a Vickers hardness of 180 [Bego, Germany 1992].

2.2.2 PROPERTIES OF BASE METAL ALLOYS

Base metal alloys offer many superior properties that give them an advantage in selection over the metal ceramic precious and semi-precious alloys [Bertolotti, 1988:81]. Examples include high tensile strength (up to 827.4 MPa) and high elastic modulus (about 206850 MPa). The high tensile strength permits use of thinner metal sections than would be possible if metal ceramic precious and semi-precious alloys were used. This was demonstrated by Weiss [1983:232] who reported a reduction in coping thickness from 0.3 mm to 0.1 - 0.2 mm and a reduction in the cross section of interproximal connectors from 4 - 8 mm to 1 - 2 mm. The base metal alloys have the highest elastic moduli of all dental alloys, and this decreases flexibility to a significant degree. The flexibility of a bridge framework constructed of base metal alloy is less than half that of a framework of the same dimensions made of metal ceramic precious alloy [Bertolotti, 1988:82].

Base metal alloys show sag resistance that is uniformly superior to all metal ceramic precious and semi-precious alloys. This characteristic allows for a reduction in the width of lingual collars to less than 1 mm, resulting in superior aesthetics.

This quality also allows for the reduction in the thickness of copings without deformation and creep when using high firing cycles [Weiss, 1983:233].

In addition to the foregoing, base metal alloys have qualities of low density, excellent castability and most significantly, low cost [Bellagamba, 1985:355].

2.3 OVERVIEW OF THE PRESENT POSITION IN THE INDUSTRY WITH REGARD TO POSTCERAMIC SOLDERING

2.3.1 POSTSOLDERING OF METAL CERAMIC BRIDGES

The technique of postsoldering two or more units of a bridge after the porcelain has been fired and glazed is well documented and described, [Friedman, 1978:31-34 ; McLean, 1980:410-412]. The value of this technique can be seen in the following situations :

1. It can be used to attach an all-metal crown to a metal ceramic crown or bridge. This is especially the case when there is insufficient space for metal and porcelain in the distal abutment of a posterior metal ceramic bridge [McLean, 1980:410].

2. It is also common practice to postceramic solder the individual units of a metal ceramic bridge in order to give the crowns a more natural axial contour with open embrasures [Friedman, 1978:31].

In addition the technique can be used to solder a precision attachment or to repair a metal ceramic bridge.

2.3.2 SOLDERING OF PRECIOUS AND SEMI-PRECIOUS ALLOYS

The solderability of precious and semi-precious alloys is well documented [Phillips, 1982], and much of the literature published on postceramic soldering of these alloys centres around the effect of:

- various gap distances on strength [Stade, Reisbick and Preston 1975:527],
- types of solder used [Nicholls, 1985:477], and the effect of various combinations of alloy when soldered together [Sloan, Reisbick and Preston, 1982:686].

All the above authors found that the tensile strengths produced when soldering these alloys with gold solders were clinically acceptable. The tensile strength of gold alloy solders is similar to those of a Type II or Type III cast gold alloys [Phillips, 1982:538].

2.3.3 SOLDERING OF BASE METAL ALLOYS

The inability to postceramic solder base metal alloys successfully when using the same soldering method as for precious and semi-precious alloys, has been a negative factor in the selection of these alloys for multi unit restorations. This is especially the case when full gold restorations need to be soldered to base metal alloy metal ceramic restorations. In general, soldering has been unreliable and technique-related problems appear to be a major contributor [Kaylakie and Brukl, 1985:461]. The most important consideration with the base metal alloys was the control of surface oxide formation between the base metal and the solder when an attempt was made to solder base metal to a precious, high-gold alloy [Bellagamba, 1985:355]. Other research has shown that base metal alloys formed oxide layers approximately three times thicker when heated in air rather than in a furnace under vacuum [Baran, 1984:80]. Although soldered joints involving base metals were reported to possess adequate strength despite the presence of oxide layers, the oxide layers and the corrosion potential were considered detrimental [Staffanou, Radke and Jendresen, 1980:34]. It was also reported that successful solder joints were not achieved consistently when base metal alloys were soldered to each other or to semi-precious alloys. However, precious alloys soldered to semi-precious or precious alloys were successful and predictable [Sloan et. al., 1982:689].

2.3.4 EXISTING SOLUTIONS TO THE PROBLEMS OF SOLDERING BASE METAL ALLOYS

An analysis of the literature shows that there are a number of existing solutions to the problems of soldering these alloys:

1. The use of argon gas

One of the reported methods to overcome the problem of the oxide formation has been to carry out the soldering procedure in a argon-protected atmosphere [Prasad, Day and Best, 1982]. However this technique has only been perfected for preceramic soldering and further research has shown that the use of an argon atmosphere does not contribute to an improvement in the flexural strength or solderability of preceramic soldered base metal alloys [Anusavice and Shafagh, 1986:317]. This result combined with the difficulty of converting the porcelain furnace to take argon into the firing chamber while still retaining vacuum is probably the reason for it not being tried for postceramic soldering.

2. The method of preceramic wetting of the joint surfaces

Another successful technique, described by Bellagamba [1985:356], of prewetting the base metal alloy with a high temperature solder prior to investing for postsoldering in the porcelain furnace is the present solution to the problem. The method consists of the following steps:

1. The parts to be soldered are firstly invested so that the joint area is horizontal. (This can be difficult in long span bridges but is necessary to prevent potential distortion.)
2. Firing in the porcelain furnace or heating with a gas/oxygen torch to prewet the joint with solder.
3. Cooling, divesting, trimming up the joint area.
4. Porcelain application and firing.
5. Re-positioning and investing the parts and then soldering the parts together in the porcelain furnace.

The drawback of this technique is the time and extra work involved in having to invest twice.

2.4 BASE METAL SOLDERING TECHNIQUE USED IN ELECTRONICS INDUSTRY

The use of gold plating connectors to enhance the solderability and tensile strength of electronic components has been successful in the electronics and aerospace industry for some time [Lee, 1971:997; Metals Handbook Ninth Edition, vol 6, 1983:1075]. The effect of the gold plating is to prevent the oxide build-up on the base metal alloy, thus allowing the solder to properly wet the surfaces to be soldered. In addition the wetting speed requirements of mass soldering, as used in

electronics soldering, often necessitate plating to improve the solderability of metals such as nickel, nickel-chromium, nickel-copper or beryllium-copper. [Metals Handbook Ninth Edition, vol 6, 1983:1075].

There is no reason why this technique should not be applied to the soldering of dental base metal alloys.

2.5 FLUXES

2.5.1 GENERAL DISCUSSION OF FLUXES USED IN DENTISTRY

The purpose of flux is twofold: firstly to remove any oxide coating on the parent metal surface and/or secondly, to prevent oxide from forming so that when the filler metal or solder is fluid it will wet the parent metal surface and flow into place. The filler metal or solder will not wet an oxide surface [Phillips, 1991:530], and if wetting does not take place, or is only partial in nature, the strength of the joint will be compromised.

Fluxes may be divided into three activity types:

Type 1. Protective. This type covers the metal surface and prevents access to oxygen so no oxide can form.

Type 2. Reducing. This type reduces any oxide present to free metal and oxygen.

Type 3. Solvent. This type dissolves any oxide present and carries it away.

Most fluxes combine two or more of these activity types [Phillips, 1991:530].

Fluxes used for precious and semi-precious alloys are usually a combination of type 1 and 2 fluxes. They are usually based on boric or borate compounds such as boric acid, boric anhydride, and borax. They act as protective fluxes by forming low-temperature glass and reducing fluxes for low-stability oxides such as copper oxide [Phillips, 1991:530].

With regard to base metal alloys, the oxides that form on these alloys are more stable in nature and therefore the fluxes used for these alloys are a combination of a type 1 flux and type 3 flux. They usually contain borates as glass formers to act as the protective element and fluorides to act as a solvent and to dissolve the chromium and nickel oxides [Phillips, 1991:530]. These types of fluxes are used for preceramic soldering of base metal alloys, but are not suitable for postceramic soldering due to the fact that the type 3 element attacks and discolours the porcelain.

2.5.2 FLUXES USED IN THE ELECTRONICS INDUSTRY

It is well accepted that alloys such as nickel-chromium, stainless steel, nickel-copper and chromium are considered very difficult to solder. The recommended fluxes to solder these alloys are inorganic fluxes usually composed of a combination of the following:

Acids - (hydrochloric, hydrofluoric, or orthophosphoric) and
Salts - (zinc, chloride, ammonium chloride, tin chloride)
[Metals Handbook Ninth Edition, vol 6, Welding, Brazing and
Soldering, 1983:1075]. This implies that the foregoing
components in these fluxes make them similar to the type 3
fluxes used in dentistry as outlined in section 2.5.1

The discolouration of the porcelain and the corrosive potential of these fluxes are high, and while these may not be factors in the electronics industry, they would not be suitable for postceramic soldering of dental alloys.

2.5.3 FLUXES USED IN POSTCERAMIC SOLDERING

A flux used for postceramic soldering must not discolour or attack the porcelain. The cause of discolouration is high concentrations of type 3 elements, usually fluorides, contained in fluxes made for use with base metal alloys.

Precious and semi-precious metal ceramic alloys contain small amounts of base metal elements, usually less than 2% of elements such as tin, iron and indium, in order to provide a suitable oxide for porcelain bonding [Phillips, 1991:365]. The fact that these base metal elements produce an oxide, requires the inclusion of small amounts of the type 3 elements in the fluxes recommended for postceramic soldering. The concentration of the type 3 elements is sufficiently low so that they do not discolour or significantly attack the porcelain but are able to cope with the small amount of base metal oxides.

In the case of the metal ceramic base metal alloys, the concentration of the type 3 elements in a flux suitable for precious or semi-precious metal ceramic alloys, is insufficient to cope with the higher levels of oxide production. It is only by increasing the concentration of the type 3 element in the flux that wetting can be achieved. This however results in discolouration of the porcelain making the fluxes unsuitable when conventional soldering methods are used [Gundlach, 1992:Personal communication, and Phillips, 1991:530].

2.5.4 DISCUSSION

It is probable that one of the major factors leading to the technique sensitivity of previous studies into the solderability of metal ceramic base metal alloys for postceramic soldering, was that the efficiency of the fluxes recommended by the manufacturer, was insufficient to cope with the oxide formation.

This is probably because the fluxes that are available on the market and that will cope with base metal oxides attack and discolour the porcelain, thus the fluxes that dental suppliers and manufacturers are recommending are of the type 1 and 2 composition with small concentrations of type 3 elements, which don't discolour and attack the porcelain. With the exception of Sobieralski, Brukl and Smith [1987:40 & 42] , very little attention has been given in articles published in the dental literature to the influence of the flux on the success or lack of success of the studies done. All that is usually mentioned is that the flux recommended by the solder manufacturer was used. This indicates that the authors assumed that the flux recommended by the manufacturer was efficient, and therefore insufficient attention was given to its importance as a factor. When authors changed to a more efficient flux [Sobieralski et. al., 1987:40, Sloan et. al., 1982:689], they did not indicate whether or not the flux used would discolour the porcelain. In all probability the flux that they used to produce strong joints did discolour the porcelain and therefore it would be unwise for a commercial dental technician to assume that he can achieve similar results without affecting the porcelain.

In contrast the literature in the electronics industry attaches great importance to the efficiency of the flux used. It can only be assumed that the demands of large scale production of soldered joints in this industry have necessitated greater study in this area.

The important point here is whether gold plating will result in predictable strong solder joints when using a flux that does not discolour or attack the porcelain.

2.6 SUMMARY

The foregoing literature review has explained the problems associated with the soldering of base metal alloys (specifically the oxide layer preventing the gold solder from wetting the joint surface), and outlined the present methods used to overcome them. However these methods are time consuming. One of the successful methods used in the electronics industry is to gold plate the surfaces of the soldered joints prior to them being soldered. It was the initial purpose of this study to apply this same technique to the soldering of base metal alloys used in dentistry. At the same time, the study attempted to evaluate the applicability of a gold soldering method where a flux that did not discolour or attack the porcelain was used. This was done in order to produce predictable, strong joints which would be clinically and commercially acceptable.

CHAPTER THREE

METHOD

3.1 THE DATA TO BE USED

3.1.1 PRIMARY DATA NEEDED

The following primary data were needed to address subproblems one, two and three:

The mean tensile strength of the samples when tensile fracture occurs in MPa.

The level of predictability of the soldering technique based on detailed records kept of the number of soldered joints attempted compared to the number of successful joints produced in terms of their tensile strength, (exceeding the ISO minimum of 350 Mpa, or the 0,2% proof stress of the weakest of the metallic parts [ISO 9333, 1990(E) : 1]). These data are expressed in a non-parametric X^2 statistic.

3.1.2 SECONDARY DATA NEEDED

In order to compare tensile strengths to the minimum specifications, the ISO 9333 [1990:1] [Mechanical strength of brazed joint] was used:

The maximum stress of the specimen shall exceed 350 MPa or the 0,2% proof stress of the weakest of the metallic parts [ISO 9333, 1990(E) : 1].

The minimum specifications in MPa for the groups are:

precious to precious (control group)	400
base metal to base metal no gold plating	360
base metal to base metal with gold plating	360
base metal to semi-precious no gold plating	350
base metal to semi-precious with gold plating	350
base metal to precious no gold plating	400
base metal to precious with gold plating	400

The above specifications are based on the technical data supplied by the manufacturer [Degussa AG, Germany, 1987].

The alloys used in this study are:

Base metal - Resistal P, 0,2%-Proof stress = 360 MPa (as cast).

Semi-precious - Realor, 0,2%-Proof stress = 240 MPa (soft).

Because this is below the minimum, the higher ISO minimum strength of 350 MPa was used.

Precious - Degulor M, 0,2%-Proof stress = 400 MPa (soft).

3.1.3 CRITERIA FOR ACCEPTABILITY OF DATA

Only data produced by the researcher were used, except in the case where scanning electron microscopy was done on selected specimens by Huletts Aluminium, Pietermaritzburg.

3.1.4 METHODOLOGY

The study used the pre-test post-test experimental method. First the soldering technique was validated by soldering precious to precious alloy. The data produced were then compared with existing published ISO minimum standards, thereby showing that the soldering method and technique were sound and a control was established. In the second part of the study the selected alloy groups were soldered without gold plating the joints. This established the level of predictability and strength, and parameters were established so that comparisons could be made. In the last part of the experiment the above process was repeated, but the joints were gold plated prior to investing for soldering, after which the levels of predictability and strength were compared with the parameters set.

3.1.5 SAMPLE SIZE

The central limit theorem states that for sample sizes greater than 30 the sampling distribution of means has a normal distribution and hence statistically a sample greater or equal to 30 is recommended.

Due to budget constraints with regard to the price of the precious and semi-precious alloys required for the fabrication of the specimens in order to address subproblems one, two and three, the number of specimens for each group within the subproblems had to be limited.

On reviewing the related literature, it was found that the size of the sample used for tensile testing in the studies listed below ranged from $n = 5$ to $n = 10$, while the sample size recommended by ISO 6871 for tensile testing base metal casting alloys, is $n = 6$ and that of ISO 9333 for dental brazing materials, is $n = 3$.

◦ ISO 9333:1990(E)	$n = 3$
◦ Kaylakie et., al., [1985]	$n = 5$
◦ ISO 6871:1987(E)	$n = 6$
◦ Staffanou et., al., [1980]	$n = 6$
◦ Sobieralski et., al., [1990]	$n = 10$
◦ Mashall et., al., [1984]	$n = 10$
◦ Sloan, et., al., [1982]	$n = 10$
Mean	$n = 7$

It was therefore decided to use the maximum affordable sample size of $n = 10$. Because the study also attempted to measure the predictability of the soldering process, ten specimens were prepared for soldering and the sample size for tensile testing was based on the number of successful solder joints produced. A lower limit cutoff of $n = 6$ for the tensile testing sample was set, in which case the soldering method would be considered to

be unpredictable and would require a modification. The relevance of the results when compared to previous studies would therefore be acceptable.

3.2. GENERAL DESCRIPTION OF THE MATERIALS AND METHOD

3.2.1 INTRODUCTION

The following description of the materials and method is a step by step outline of the critical steps that were followed in this study. By following these steps and methods any subsequent researcher should be able to duplicate the techniques and results. A detailed discussion as to why a particular material or method was used, or what could or did influence the steps outlined, is provided in section 3.3. so as not to detract the reader from following the main thread of the method used.

3.2.2 WAX PATTERN PRODUCTION

Half of a cast tensile test specimen, in accordance with the dimensions shown in section 3.3.2.2 (Figure 3.1 A), was turned on a lathe in brass. In addition, a sprue was turned to allow for the wax injection and later for the attachment to the sprue former for investment for casting. A HTV (high temperature vulcanising) rubber mould was made of the specimen (Figure 3.1 B). The mould was separated using a number 11 scalpel blade. The wax patterns were made by wax injection using a vacuum-pressure jewellery wax injection machine.

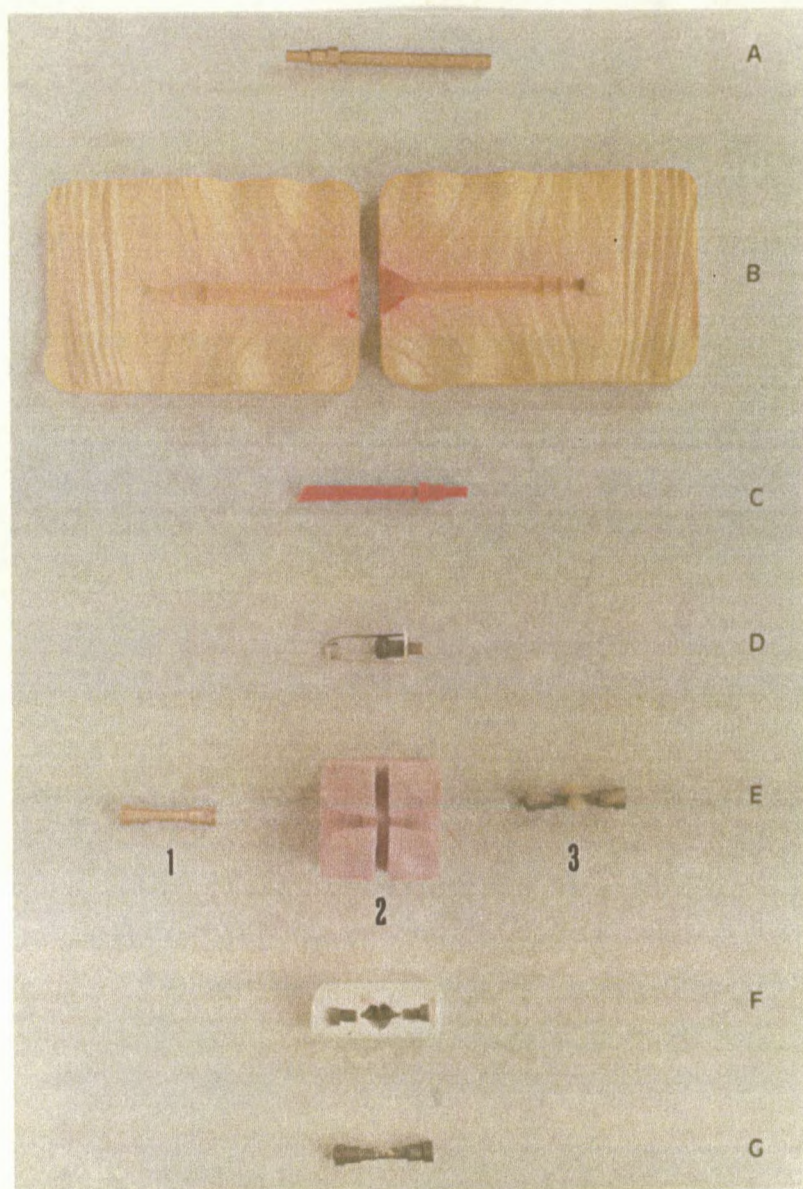


FIGURE 3.1: SEQUENCE OF TENSILE TEST SPECIMEN FABRICATION SHOWING:

- A - Half of a tensile test specimen turned in brass.
- B - An HTV rubber mould of the brass specimen.
- C - A wax pattern made in the mould by wax injection.
- D - Half of a cast tensile test specimen with gold plated end, held in a wire clip used to suspend specimen in gold plating bath.
- E1- Brass test specimen machined to 5% less than specification.
- E2- Silicon soldering specimen assembly jig.
- E3- Specimens invested ready for soldering.
- F - Specimen invested ready for soldering.
- G - Soldered specimen ready for tensile testing after being machined to diameter of $2.5 \text{ mm} \pm 0.01 \text{ mm}$.

3.2.3 CASTING OF SPECIMENS

Batches of 10 wax patterns produced by the method described in section 3.2.2 were attached to a 3X sprue former [Degussa AG, Germany]. The amount of alloy required to cast the patterns was

calculated in the following way:

$$A \times B = C$$

A = weight of sprue former with the attached patterns
minus the weight of the sprue former in grams

B = specific gravity of the alloy

C = weight in grams of the alloy required to cast the
patterns

The following brands of alloy were used:

Base metal alloy - Resistal P [Degussa AG, Germany]

Semi-precious alloy - Realor [Degussa AG, Germany]

Precious alloy - Degulor M [Degussa AG, Germany]

A 3X ring which was lined with one layer of ring liner (this was to facilitate easy removal of the investment from the ring after casting), was placed over the sprue former ready for investment. The ring was then invested with a phosphate bonded investment, [Belavest T, Bego, Germany] using 100% water and no silica

solution (this was to facilitate easier recovery of the casting from the investment). The investment and the water were mixed for 60 seconds, poured into the ring and allowed to bench set for 1 hour. The ring was then placed in a burnout furnace [KWO/EWL, Type 5645, Germany], and the following staged burnout cycle for each alloy was used:

Resistal P alloy - room temperature to 250°C, maintained at 250°C for 60 minutes, raised to 900°C, and maintained at this temperature for 60 minutes prior to casting.

Degulor M and Realor alloys - room temperature to 250°C, maintained at 250°C for 60 minutes, raised to 650°C, and maintained at this temperature for 60 minutes prior to casting.

A centrifugal casting machine was used [Kerr, UK]. The casting machine was carefully balanced prior to the rings being placed in the furnace. The alloys were melted with a gas/oxygen torch [Degussa AG, Germany, model 2350 0042].

3.2.4 RECOVERY AND CLEANING OF CASTINGS

After casting, the rings were allowed to bench cool till they could be handled and then divested. After divesting the castings were sandblasted with 50 micron alumina oxide at a pressure of 3 bars to remove all traces of investment and oxide. The specimens were then examined for any casting defects and any nodules were removed. At this stage the Degulor M and Realor specimens were separated from their sprues using a 0,3 mm cutting disk mounted in a high speed grinder [KWO EWL 4422/740, Germany].

3.2.5 PORCELAIN FIRING CYCLE FOR BASE METAL SPECIMENS

The Resistal P specimens (still attached to the sprues to facilitate easy handling), were then placed in a porcelain furnace [Vita Vacuumat 300], and subjected to the following porcelain firing cycles:

One wash bake cycle:

- preheat at 500°C for 1 minute
- heating to 990°C for 3 minute
- hold at 990°C for 1 minute
- vacuum for 3 minutes

Two opaque bake cycles:

- preheat at 500°C for 3 minute
- heating to 930°C for 6 minute
- hold at 930°C for 1 minute
- vacuum for 6 minutes

Two dentine bake cycles:

preheat at 500°C for 6 minute

heating to 920°C for 6 minute

hold at 920°C for 1 minute

vacuum for 6 minutes

One glaze cycle:

preheat at 500°C for 4 minute

heating to 920°C for 3 minute

hold at 920°C for 2 minute

no vacuum

The specimens were then separated from their sprues using a 0,3 mm cutting disk mounted in a high speed grinder.

3.2.6 GOLD PLATING OF THE SPECIMENS

The Resistal P base metal specimens that were required for gold plating for subproblem three were separated from those required for subproblem two.

The surfaces to be soldered were ground with a straight cut carbide bur. A slight bevel was also ground at the edge. This was done to channel the molten solder to flow into the prepared solder gap and not to flow back along the shaft of the specimen. In addition the sides of the specimens were also ground to allow a current to flow to the clip holders to facilitate gold

plating. Holding clips were made from 0,7 mm stainless steel orthodontic wire (Figure 3.1 D), so that the samples could then be suspended in the plating solutions.

The following method was used to gold plate the specimens:

1. Ten specimens at a time were mounted on a copper wire holder that was bent up to hold the samples in the centre of the containers that were holding the solutions.
2. The specimens were then placed into an electrolytic cleaning solution for 1 minute at 6 volts, temperature of the bath 65°C [Balco Electrolytic Cleaning Salts, Balco, England]. The specimens were then rinsed under running tap water for 15 seconds.
3. The lower half of the specimens were then placed into the gold plating solution [Balco Gilding Salt, Balco, England], at 4 volts, temperature of the bath 60°C for 15 minutes, using a 24 carat gold anode. On removal the specimens were rinsed under running tap water for 15 seconds to remove any residue of the plating solution.

[The gold plating bath was manufactured by Jewellery Machine Supplies, Durban, S.A.]

3.2.7 NUMBERING AND IDENTIFICATION OF SPECIMENS

The specimens were numbered in a way that would allow for the specific identification of each individual half of a tensile test specimen. Table 3.1 outlines the method used in tabular form. It was important that each specimen could be specifically identified so that there could be no confusion during photomicroscopy and when relating the results from the tensile testing to the observations made.

TABLE 3.1 SYSTEM USED FOR NUMBERING AND IDENTIFICATION OF SPECIMENS

	Precious to Precious No G.P.	Base metal to Base metal No G.P.	Base metal to Semi-precious No G.P.	Base metal to Precious No G.P.	Base metal to Base metal With G.P.	Base metal to Semi-precious With G.P.	Base metal to Precious With G.P.
Subproblem One	Numbered 1-10 Left side marked A. Right side marked B.						
Subproblem Two		Marked A. Numbered A-J Left side marked 1. Right side marked 2.	Marked B. Numbered A-J Left side marked 1. Right side marked 2.	Marked C. Numbered A-J Left side marked 1. Right side marked 2.			
Subproblem Three					Marked D. Numbered A-J Left side marked 1. Right side marked 2.	Marked E. Numbered A-J Left side marked 1. Right side marked 2.	Marked F. Numbered A-J Left side marked 1. Right side marked 2.

3.2.8 SOLDER JOINT SURFACE PREPARATION

The surfaces to be soldered were ground with a straight cut carbide bur (section 5.2.6). A slight bevel was also ground at the edge. This was done to channel the molten solder to flow into the prepared solder gap and not to flow back along the shaft of the specimen. Once ground, particular care was taken not to touch the prepared joint or to allow the surface to be contaminated in any way prior to the specimen being mounted in the soldering jig ready for waxing together.

3.2.9 JOINING SPECIMENS IN SOLDERING ASSEMBLY JIG

A silicone rubber soldering assembly jig (section 3.3.9) was used to align the cast samples. A 0,2 mm spacer was used to establish the soldering gap. Then the specimens were sticky waxed together and a 1,1 mm stainless steel wire rod was sticky waxed along the length of the specimens to add support and rigidity to the assembly. Once the wax had cooled, the assembled specimens were removed and the area around the joint was waxed so as to prevent any investment from coming into contact with the joint area and to create a space in the investment after the wax had been boiled off to allow for fluxing and solder flow (Figure 3.1 E2 & E3).

3.2.10 INVESTING SPECIMENS FOR SOLDERING

The specimens were invested in a soldering investment [Deguvest L, Degussa AG, Germany] mixed according to the manufacturer's instructions. The investment blocks were allowed to bench set for 2 hours and were then trimmed on a model grinder to the minimum shape possible without compromising the strength of the soldering block. This was to allow for a more efficient heating of the block when in the furnace. Excess investment dissipates the heat away from the parts to be soldered and requires a longer heating time which can be detrimental to the coefficient of thermal expansion of the porcelain [Yamamoto 1985:190].

The sticky wax and stainless steel bar were then melted off by pouring boiling water over the invested specimens till no residue of wax was left. The investment blocks were then allowed to dry for 30 minutes before fluxing and placement of the solder.

3.2.11 FLUXING OF SPECIMENS AND PLACEMENT OF GOLD SOLDER

The solder for subproblems one, two and three was prepared in the following manner:

1. The strip solder, Degulor lot 2, was dipped into the flux to the depth of approximately 3 cm, and then held over a bunsen burner to melt the solder so that it formed a small ball. While still hot it was dipped back into the flux to remove any oxide that may have formed.

The fluxes used were:

Subproblem one - T flux [Degussa AG, Germany]

Subproblem two and three - Minoxyd flux, [Bego, Germany].

2. The ball of solder was cut off from the strip leaving a short tag or tail of approximately 5 mm. This was to act as a support for the solder when positioning it on the solder joint gap.
3. Flux was placed into the soldering gap, sufficient to wet all the surfaces prepared for soldering.
4. The solder ball was then placed over the gap with the tail resting on the side of the soldering investment block for stability and support. A small amount of runny soldering investment was placed over the tail to secure the solder in position. This was to prevent the solder from slipping off the specimens when the flux became molten during the firing procedure.
5. The prepared soldering block and specimens were then placed into a drying cabinet set at 100°C for 1 hour to dry off any moisture that remained in the soldering investment. This is very important for, if there is excessive moisture, it will, upon being placed on the firing muffle of the porcelain furnace at 500°C, boil and produce steam. This will shatter the investment block, or in the case of crowns, cause them to pop off the investment.

The above procedure was carried out for each of the specimens (Figure 3.1 F).

3.2.12 IN FURNACE SOLDERING

The porcelain furnace used to solder the samples was a Vita Vacumat 300 [Vita Zahnfabrik, Germany].

The following firing cycle was used to solder the samples:

Preheating - 500°C for 8 minutes

Temperature climb - 500 - 800°C over 4 minutes

Hold temperature - 800°C for 3 minutes

Vacuum - 7 minutes.

The specimens were put through the above firing cycle individually. Upon completion of the cycle the specimens were removed from the muffle and allowed to cool to room temperature.

3.2.13 RECOVERY AND CLEANING

When the soldered specimens had cooled the investment was removed and the specimens were sandblasted with 50 micron alumina oxide (pressure of 3 bars) to remove all investment, oxide and flux. A visual inspection was carried out to establish any defects which were noted.

3.2.14 MACHINING ON LATHE TO SIZE SPECIMENS

After being recovered and cleaned, the specimens were turned on a centre lathe [Maximat V13, Austria, supplied by INTERNATIO (PTY), LTD, Johannesburg] using a collet to hold the grip end of

the specimen. The specimens were reduced to a diameter of $2,5 \text{ mm} \pm 0,01 \text{ mm}$, to $\pm 1,5 \text{ mm}$ either side of the solder joint along the guage length (Figure 3.1 G).

3.2.15 TENSILE TESTING OF SPECIMENS

Tensile testing was performed on an Instron Universal Testing Machine [model 4301, Instron Corp., High Wycombe, Bucks, U.K.]. The grips used for the tests were " Instron Wedge-Action Grips " [Instron Ltd, High Wycombe, Bucks, U.K.], the upper member being connected to the load cell by a universal joint to avoid torsional forces on the samples during loading. Each specimen was loaded to fracture using a cross head speed of 1 mm/min. By entering the area of the cross section of the diameter of the specimen at the solder joint into the microprocessor of the testing machine, the ultimate tensile strength for each specimen was automatically calculated and given in MPa (Table 4.1).

3.2.16 MICROSCOPY

All the specimens were photographed end-on using an inverted metallurgical microscope [Nikon Epiphot, Japan]. Selected specimens were mounted for side-on photographs using the following preparation method:

1. The specimens were pressed into a flat piece of sheet wax [Toughened no. 4, Kemdent, UK] at an angle of 50 degrees. A circle of wax was then placed around the specimens and waxed to the flat sheet. Clear self curing acrylic [Sankin, Japan] was poured into the circle of wax while still liquid and allowed to set.

2. The wax was boiled off and a shallow hole was ground into the top of the acrylic to allow for the positioning of the point of the rotating arm of the polishing machine. This produced an acrylic block with the fracture end of the specimens slightly protruding below the base by approximately 50%.

3. Each acrylic block was then polished in a polishing machine [Buehler Minimet Polisher] with the following grit water paper:

180 grit - 5 minutes

400 grit - 10 minutes

600 grit - 10 minutes

Thereafter, each acrylic block was polished with diamond polishing pastes:

8 micron - 10 minutes

3 micron - 10 minutes

1 micron - 10 minutes

After each grit or diamond paste polishing stage, the acrylic block was rinsed off with boiling water to remove any grit or residue that could remain to contaminate the next stage of the polishing cycle.

3.2.17 ELECTRON SCANNING MICROSCOPY

Selective specimens were examined using qualitative analysis of an energy dispersive x-ray spectrum [Series II X-Ray Analyser, Noran Instruments, Middleton] which was attached to a scanning electron microscope [Model S-450, Hitachi, Ltd, Tokyo, Japan]. The results are shown in section 4.13.

3.2.18 STATISTICAL ANALYSIS

The following statistical tests were applied to address the subproblems:

Subproblem One:

Tensile strengths

Although previous authors used the t-test to establish whether the sample mean differed from published specifications, it is argued that this test is inappropriate due to the upper tensile strength limit of the parent metal of the parts to be soldered which causes a skewing in the distribution of the population. (Note: The t-test is based upon the assumption that the sample

is drawn from a population that has a normal distribution.) Therefore the analogous non-parametric testing technique, namely the one-sample sign test was applied.

Predictability

In order to measure the success rate of the soldering procedure against a specified proportion (100%), a binomial test for small samples ($n = 10$) was used.

Subproblem Four:

Tensile strengths

Previous authors used the unpaired t-test to establish whether there was a significant difference between the means of the sample groups. It is argued that this test is inappropriate due to the upper tensile strength limit of the parent metal of the parts to be soldered which causes a skewing in the distribution of the population. (Note: The unpaired t-test is based upon the assumption that the sample is drawn from a population that has a normal distribution.)

Therefore the analogous non-parametric testing technique, namely the Mann-Whitney U test was applied to the means of the

sample groups:

- base metal to base metal no gold plating
base metal to base metal with gold plating
- base metal to semi-precious no gold plating
base metal to semi-precious with gold plating
- base metal to precious no gold plating
base metal to precious with gold plating

In order to establish if each sample met the minimum specification, the analogous non-parametric testing technique, namely the one-sample sign test was applied to each of the following sample groups:

- base metal to base metal no gold plating
base metal to base metal with gold plating
- base metal to semi-precious no gold plating
base metal to semi-precious with gold plating
- base metal to precious no gold plating
base metal to precious with gold plating

Predictability

The non-parametric X^2 statistic was used to determine if there was a dependence between two sets of categorical variables .

Gold plating versus no gold plating:

The first variable: a dichotomous yes / no response, i.e. did soldering produce a strong solder joint (equal to or greater than the minimum specification) or not.

The second variable: to identify whether or not gold plating was used.

Control group versus all base metal alloy groups:

The first variable: a dichotomous yes / no response, i.e. did soldering produce a strong solder joint (equal to or greater than the minimum specification) or not.

The second variable: to identify whether or not the control group or base metal alloy groups were used.

3.3 DISCUSSION OF IMPORTANT FACTORS THAT AFFECT THE MATERIALS AND METHOD

3.3.1 INTRODUCTION

The following section is a discussion to substantiate why a particular method, technique, material or item of equipment was used. It also offers explanations of factors that may have had bearing on or may have affected the method that was outlined in the previous section (3.2.). Also, in certain instances, this section attempts to highlight areas where the results produced

by the method followed may have been influenced and to explain to what extent this influence may have occurred.

3.3.2 TENSILE TESTING

3.3.2.1 METHOD SELECTION

An analysis of the stresses that a soldered connector is subjected to in the mouth, especially when it is part of a long span bridge, shows that the stresses present can be either tensile, compressive or shear, or a combination of all or two of these at the same time. However, an attempt to create such stresses in a laboratory and to standardise the procedure so that it could be accurately repeated and compared, would have been too complicated and time consuming in terms of the allowed budget for the study.

Previous studies on the strength of soldered joints have all used tensile strength as a measure of their strength [Kaylakie, et. al., 1985; Marshall, et. al., 1984; Nicholls, 1985; Sloan, et. al., 1982]. The main reason for choosing tensile testing is the confidence placed in the value and significance of the test procedure. The chief advantages of the test are that the stress state is well established, the procedure has been carefully standardised, and it is relatively easy and inexpensive to perform. The data obtained can thus serve as a measure of the quality of the solder joint in comparison with data obtained previously or from other sources. [Metals Handbook Ninth Edition, Vol 8, Tension Testing, 1985.]

Although the ISO method [ISO 9333, Dental Brazing Materials] was available, it was decided to use the ASTM Standard Methods for Tension Testing of Metallic Materials [Metric], E 8M-86a, as the method [ASTM E 8M - 86a, 1984], due to the large number of published authors who have used the ASTM method.

3.3.2.2 SPECIMEN PREPARATION: ASTM E 8M - 86a

The specimen preparation was carried out in accordance with ASTM E 8M - 86a, with special reference to:

- section 6.4.2
- Figure 9: Various Types of Ends for Standard Round Tension Test Specimens, Specimen 4
- reduced in accordance with Figure 8: Standard 12.5 mm Round Tension Test Specimen with Gauge Lengths Five Times the Diameters (5D), and Examples of Small-Size Specimens Proportional to the Standard Specimen, diameter 2.5 mm [ASTM E 8M - 86a, 1984:201], (Figures 3.2 & 3.3).

The diameter of the grip end section of the test specimen was increased to 5 mm to facilitate better retention in the grips of the testing machine. This is in accordance with section 6.4.3. of ASTM E 8M - 86a page 201.

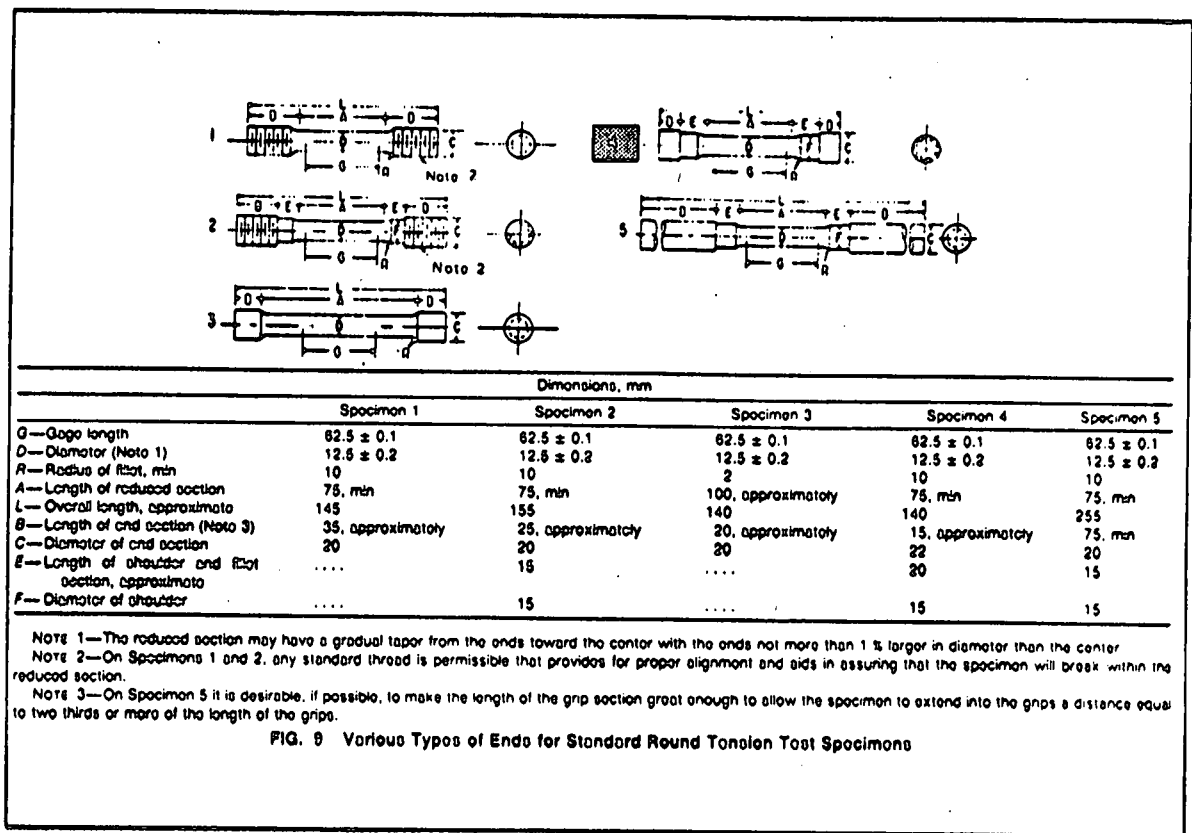


FIGURE 3.2: DIAGRAM SHOWING DIMENSIONS FOR TENSILE TEST SPECIMEN BEFORE BEING REDUCED TO 2.5 mm.
(FROM ASTM E 8M - 86a, 1984:214)

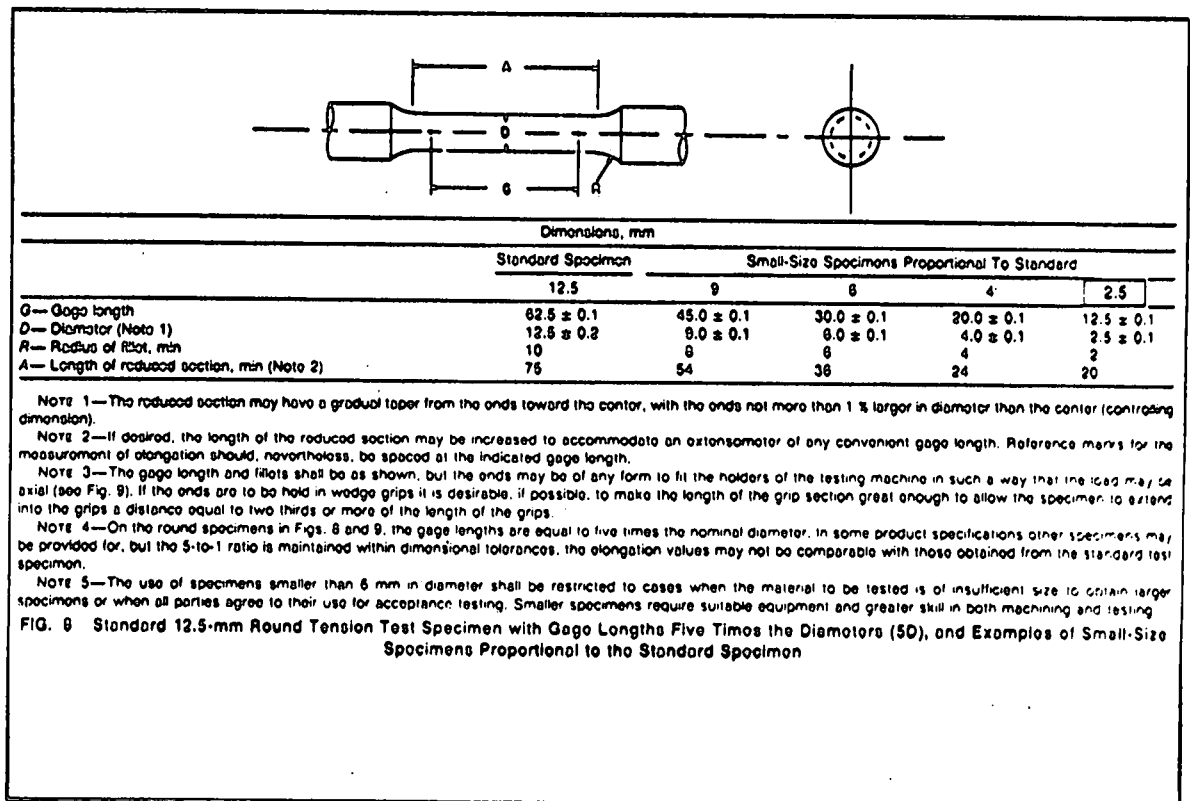


FIGURE 3.3: DIAGRAM SHOWING DIMENSIONS FOR REDUCING SPECIMEN SIZE TO 2.5 mm (FROM ASTM E 8M - 86a, 1984:213)

3.3.2.3 TEST SPECIMEN WAX PATTERN FABRICATION

The minimum number of wax patterns of the specimens required for casting in the various alloys was 140. In order to facilitate the uniform production of these, the following jewellery manufacturing procedure was used:

1. Half of a cast tensile test specimen, in accordance with the dimensions shown in section 3.3.2.2, was turned on a lathe in brass. In addition, a sprue was turned to allow for the wax injection and later for the attachment to the sprue former for investment for casting. The diameters of the specimen turned on the lathe were increased by 10% to allow for the shrinkage of the wax during the wax injection process (Figure 3.1 A).
2. An HTV rubber mould was made of the specimen fabricated in 1. using "castaldo" rubber, type "white label", heated at 153°C in a jewellery vulcanising machine supplied by Durban Wholesale Jewellery Supplies. The mould was separated using a number 11 scalpel blade (Figure 3.1 B).
3. The wax patterns were made by wax injection using a vacuum-pressure jewellery wax injection machine supplied by Durban Wholesale Jewellery Supplies (Figure 3.1 C).

The wax patterns produced by this method were then cast in the various alloys as specified by the subproblems.

The decision to use a jewellery manufacturing method was to facilitate the accuracy that was required with regard to dimensions, and because of the speed of production that this technique allows compared to the system in a dental laboratory where each pattern is made by hand, even if a mould is used.

3.3.3 TESTING MACHINE CONSIDERATIONS

3.3.3.1 DESCRIPTION OF TESTING MACHINE

The testing machine used for the tensile tests was a Instron Universal Testing Machine model 4302 (Annexure A). This machine more than complies with the minimum requirements as laid down by ASTM E8M-86a SEC 5.1 Testing machines., p200., ASTM E4 AND ASTM E83. It also fulfils the criteria described by Martin [1985:50], which state that the following factors need to be met when selecting a testing machine so as to ensure accuracy and reproducibility of test results:

1. Stiffness

The loading frame of a tension testing machine must be extremely stiff to avoid deflections that can cause testing error.

2. Precision

For reliable data, the testing machine must be accurate and provide reproducible results. Effects of mechanical inertia of offsets could be devastating to a testing program. A highly accurate, responsive load and strain measuring system should be employed that maintains the same accuracy whether a given load cell is operating on a low load range or a high load range. In addition, the testing speed must be controlled within close limits, as specified by the appropriate test method.

3. Flexibility

A testing machine must cover a variety of uses that typically require different speeds and load ranges. Modular transducers, specimen grips and accessories for machine control and data read-out greatly extend the useful life of a testing machine.

3.3.3.2 GRIPPING TECHNIQUE

The grips used for the tests were "Instron Wedge-Action Grips" [Instron Ltd, High Wycombe, Bucks, U.K.].

According to Martin [1985:50], the use of proper grips and faces for testing materials in tension is critical in obtaining meaningful results. Tension testing of most round specimens can be accommodated with wedge-type grips.

In choosing this instrument the requirements as laid down by ASTM E8M-86a SEC 5.2.2 Wedge Grips, p. 200 were met.

3.3.3.3 STRAIN RATE

According to Gillis and Gross [1985:38], the rate at which a specimen is deformed (strain rate) is an important consideration in the testing of a material as the strength properties tend to increase at higher rates of deformation.

The following factors were therefore considered in determining the cross head speed of the testing machine:

1. Speed of testing

ASTM E8M-86a Sec., 7.2.1(d) defines the speed of testing in terms of rate of separation of the two heads of the testing machine during the test. This method has been used in most previous studies on the mechanical strength of soldered joints.

ISO 9333:1990(e) specify that the specimens should be loaded at a cross head speed of $1,5 \pm 0,5$ mm/min up to the fracture point of the specimen.

2. Cross head speed used in previous studies

Sobieralski et. al., [1990:755]	1	mm/min
Kaylakie et. al., [1985:457]	1	mm/min
Bellagama [1985:357]	2	mm/min
Sloan et. al., [1982:687]	1,25	mm/min

It was decided for this study that a cross head speed of 1 mm/min would be used so that the results could be best compared with those strengths established by Kaylakie et al [1985:457].

3.3.3.4 SOURCES AND EFFECTS OF MISALIGNMENT UNDER TENSILE LOADING

1. Sources of Misalignment

In the case of ideal alignments, the top and bottom grip centerlines are precisely in line with one another and with the centerlines of other components of the loading train. In addition they are precisely in line with the specimen centerline. Also, the specimen is symmetric about its centerline. Departures from this ideal situation are caused by poor alignment of the top and bottom grip centerline, poor conformance centerline to top and bottom grip centerlines, and asymmetric machining or casting of the specimen itself. A combination of these three sources of misalignment always operates in any test under tensile loading [ASTM E1012 APPENDIX X1, 1984:X1.1].

In this study an attempt was made to alleviate this problem by the use of a universal coupling joint to connect the grips to the load cell in the load train [ASTM E1012 APPENDIX X1, 1984:X1.1.5].

2. Effects of misalignment on test results

It is acknowledged that bending stresses which are associated with misalignment between the load and the specimen axes in tension tests can affect the results. However, in routine tension tests on most metals and alloys, bending stresses will be insignificant if there is sufficient plastic flow occurring during the test to eliminate the bending loads [ASTM E1012 APPENDIX X1, 1984:X1.2].

3.3.4 THEORETICAL BASIS FOR STATISTICAL ANALYSIS

Predictability - subproblem one: control group

It was important to establish a soldering method that would produce a successful solder joint each time it was used. In this way the method would not be a variable when using it to solder the base metal alloys if it had been previously established that it was 100% predictable when soldering the control group sample.

In order to measure the success rate of the soldering procedure against a specified proportion (100%), a binomial test for small samples ($n = 10$) was used.

Predictability - subproblem four: gold plating versus no gold plating - non-parametric χ^2 statistic.

In the busy commercial dental laboratory environment, the dental ceramist wants to know how predictable a soldering method is because he cannot afford costly remakes or the time it takes to redo a postceramic solder joint. In the previous studies referred to in the review of the related literature, the authors did not attempt to quantify the level of predictability of the methods they used. Yet most of the authors referred to the difficulty of obtaining consistent, reliable specimens despite following the manufacturer's instructions precisely [Kaylakie, et. al., 1985:458; Sloan, et. al., 1982:689; Marshall, et. al., 1984:671]. They therefore had to solder additional specimens in order to reach their sample size. A potential mistake could be made by the dental ceramist reading the above articles and thinking that, due to the acceptable strengths that the base metal solder joints exhibited, these solder joints are routine and predictable.

It is the intention in this study to attempt to investigate significant relationships which would indicate predictors of success. No attempt was made to provide a prediction model. This is a recommendation for later research based on a larger sample size.

Therefore ten specimens were prepared for soldering and the sample size was based on the number of successful solder joints produced. A lower limit cutoff of $n = 6$ for the sample was set in which case the soldering method would be considered to be too unpredictable and require a modification.

By attempting to test for significant relationships which would indicate predictors of success, it is hoped to make the findings of this study more relevant to the commercial dental ceramist.

3.3.5 PORCELAIN FIRING CYCLE FOR BASE METAL SAMPLES

The reason for subjecting the base metal samples to the porcelain firing cycles as described in section 3.2.5 was to simulate the same heating conditions and oxide production that the alloys would go through prior to the stage where the postceramic soldering process would begin. In this way the alloy should be in a similar condition to that which would be found in a metal ceramic bridge in a commercial dental laboratory [Sloan, et. al., 1982:687; Bellagamba, 1985:356].

3.3.6 CLEANING OF SURFACES TO BE SOLDERED

According to the ASM Committee on Soldering [Metals Handbook Ninth Edition, Welding, Brazing, and Soldering, 1983: Soldering 1079], oil, film, grease, tarnish, paint, pencil markings,

cutting lubricants, cutting swarf, and general atmospheric dirt, can all interfere with the soldering process. They also state that a clean surface is imperative to ensure a sound and uniform quality soldered joint. Fluxing alone cannot substitute for adequate cleaning.

Taking the above into account, it was decided to prepare all surfaces for soldering without gold plating by grinding them with a straight cut carbide bur. Surfaces that were to be gold plated were also ground with a straight cut carbide bur. The choice of bur and method was based on Yamamoto's findings, viz. that the method that would produce the least amount of bubbles as a result of microscopic contamination when grinding and preparing an alloy surface for porcelain application, was the use of a straight cut carbide bur [Yamamoto 1985:124-132].

In his study, Yamamoto ground the surfaces of precious metal cast plates with various abrasive materials:

silicone points, carborundum points, ceramic points, fissure burs, carbide burs both straight cut and cross cut, sandblasting with glass beads, and sandblasting with alumina oxide. He then applied clear porcelain to the specimens and fired them. The method that produced the least bubbles in the porcelain as a result of microscopic contamination was the straight cut carbide bur. He suggested that the use of a diamond bur was not recommended as the diamonds are fixed to the shaft by means of plating metal such as nickel, copper or

silver. These binder metals will attach to the precious metal and contaminate the metal structure at the porcelain interface.

By taking cognisance of the above information when preparing the surface of the solder joint, it can be assumed that the potential for porosity in the soldered joint will be similar to the findings of Yamamoto if a grinding stone or method is used that does not leave the surface to be soldered free of contamination. This was in fact borne out in the findings of the first attempt at subproblem one (section 3.4).

3.3.7 GOLD PLATING

Due to budget constraints, it was decided to use the gold plating bath and solutions housed within the Jewellery Design Department of Technikon Natal, as opposed to purchasing a new gold plating bath. Because a gold plating bath from a jewellery workshop was used, cognisance was taken of the following potential factors that could in some way affect the quality of the gold plate on the specimens [Nobel, Metals Handbook Ninth Edition; Surface Cleaning, Finishing, and Coating, 1982:Gold Plating]:

1. Potential metallic impurities

Because the gold plating machine and solution had been used by members of the Jewellery Design Department, the potential for metallic impurities that may have contaminated the solution to some degree or other existed. The potential contaminants were:

- Copper - contamination can cause dullness, reduced corrosion resistance and increased contact resistance. Copper wire is routinely used to suspend items in the solution while plating.
- Iron, nickel, and cobalt - contamination can result in poor solderability and brittleness. It is unlikely that this type of contamination occurred but the potential did exist.
- Silver - excess silver can harden gold and change its metallurgical properties. Infrared spectrum analysis of the gold plating solution [Balco Gilding Salt, Balco, England], showed that it contains 6% silver. In addition the most common items being plated in the bath were silver.

2. Metal distribution.

The following important variables affect good, uniform metal distribution during the plating process. This is especially the case when one wants to achieve heavier deposit thickness.

- Plating at current densities that are as low as possible. This requirement was fulfilled during the plating process.
- Proper anode area. This requirement was met.
- Proper agitation of specimens in plating solution. There was no automatic agitation system attached to the plating bath and only sporadic agitation was done by hand.
- Proper rack design. The rack that was used to suspend the specimens in the solution was designed like an inverted steering wheel to match the round shape of the plating bath. This kept the specimens equidistant from one another and positioned the specimens half the radius of the bath from its side.

3.3.8 SOLDER SELECTION

The choice of solder was based on the manufacturer's recommendations. The recommended solder for Degulor M and Realor was the solder Degulor-Lot 2 (745°C)[Degussa AG].

3.3.9 SOLDERING SPECIMEN ASSEMBLY JIG

In order to ensure that the half tensile test specimens were joined together end to end so that their alignment was as true as possible, a specimen assembly jig was manufactured in the following way:

A test specimen was machined in brass at 5% less than the ideal dimensions referred to in section 3.3.2.3 (Figure 3.1 E1). The specimen was then embedded in an addition silicone heavy bodied rubber impression material [Lab Putty, Coltene AG, Switzerland], which was then pressed into a square container with a flat bottom prior to setting. After setting the putty was cut in such a way that sufficient undercut was left so that the specimen had to be prised out (Figure 3.1 E1). A section of the putty was then cut out at right angles to the axis of the specimen to allow for the positioning of the 0,2 mm spacer.

By fabricating the jig in the above manner, the specimens were firmly held in position by friction along their full length with no room for lateral or horizontal movement. The flat base of the jig ensured that there was no distortion of the jig while resting on a flat surface.

The firmness with which the specimens was held was sufficient to keep them stable after removal of the spacer and during the process of waxing them together with sticky wax [Sticky Wax, P. Grant Smith, Johannesburg]. A 1,1 mm stainless steel wire bar was also sticky waxed along the length of the assembled specimens to add further support and rigidity during removal from the jig. The use of the rubber jig allowed for the removal of the assembled parts without distorting them or causing the wax to break (Figure 3.1 E3).

3.3.10 FLUX SELECTION

According to Phillips [1991:530], it is the responsibility of the alloy manufacturer to recommend the type of flux and solder that will be suitable when carrying out soldering procedures. This is because it is difficult to estimate the melting range and wettability of the alloy just from its composition. For subproblem one, the recommended flux was T flux [Degussa AG, Germany]. This flux produced successful solder joints with the precious to precious alloy Degulor M [Degussa AG, Germany].

For subproblems two and three the recommended flux was Resistal flux [Degussa AG, Germany], and in subproblem three the recommended flux on the gold plated surfaces was T flux. These fluxes did not produce successful solder joints (section 3.4 and 3.5). As a result of this the following wetting tests were carried out to establish a suitable flux to produce a successful solder joint:

Subproblem two

The test consisted of using the following brands of flux on cast square sheets of the base metal alloy Resistal P:

Fluxes that do not discolour porcelain

1. Resistal flux [Degussa AG] with Degulor-Lot 2 (745°C)
[Degussa AG] solder
2. Fluxsol flux [Bego, Germany] with Degulor-Lot 2 (745°C)
[Degussa AG] solder

Fluxes that do discolour porcelain

3. Minoxyd flux [Bego, Germany] with Degulor-Lot 2 (745°C)
[Degussa AG] solder

There was no wetting by the solder with the Resistal flux or with the Fluxsol flux. However there was good wetting by the solder with the Minoxyd flux (Figure 3.4). It was therefore decided to use this flux for subproblem two.

Subproblem three

Initially a wetting test was carried out on a cast sheet of base metal which had been gold plated for five minutes using Degulor-Lot 2 (745°C) [Degussa AG] solder with Resistal flux and T Flux [Degussa AG, Germany]. This was to establish if the gold plating would enhance the solderability of the base metal alloy to the extent that a flux that did not discolour the porcelain could be used. The result was only partial wetting of the surface with the Resistal flux and no wetting with the T Flux. A decision was then made to discard the T Flux.

A second wetting test was then carried out after increasing the length of time for the plating process and thereby the thickness of the gold plate, in order to establish if the wetting of the solder could be improved.

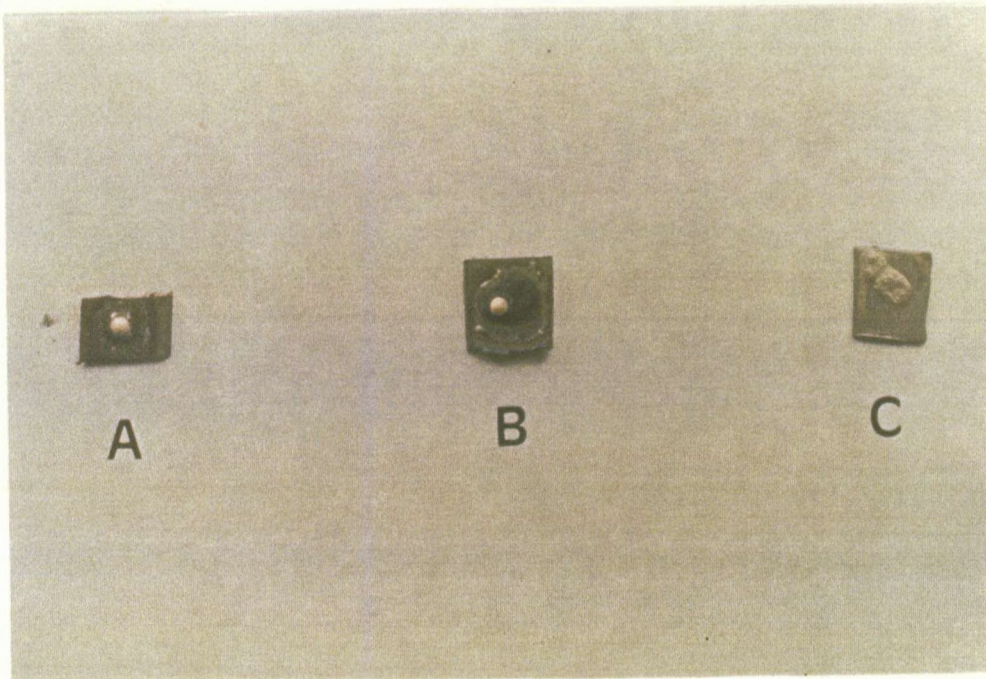


FIGURE 3.4: SOLDER WETTING TEST SHOWING:

- A - Degulor - Lot 2 solder with Resistal flux.
Note absence of wetting by the solder.
- B - Degulor - Lot 2 solder with Fluxsol flux.
Note absence of wetting by the solder.
- C - Degulor - Lot 2 solder with Minoxid flux.
Note good wetting by the solder.

Four cast sheets were prepared using a plating time of 10, 15, 20 and 25 minutes respectively. There was a slight improvement in the wetting by the solder up to the 15 minute time, but no improvement beyond this.

As a comparison, two cast base metal alloy sheets were prepared, one gold plated for 15 minutes and the other with no gold plating. These were tested using Minoxyd flux [Bego, Germany]. After soldering, it was observed that a greater amount of wetting occurred on the gold plated specimen compared to the non-gold plated specimen.

Overall, when the Minoxyd flux test was compared to the Resistal flux tests, a significant difference was observed between the Resistal flux, which exhibited only partial wetting despite the increased gold plating time, and the Minoxyd flux which exhibited good wetting, especially when gold plated (Figure 3.5).

Due to the fact that partial wetting had been observed with the Resistal flux, the first specimen from the group, base metal to base metal, was soldered using Resistal flux. After the soldering process it was observed that only partial wetting had occurred and that the solder had only flowed a third of the way into the gap.

At this stage it was decided to change to Minoxyd flux and the rest of the samples for subproblem three were soldered using Minoxyd flux.

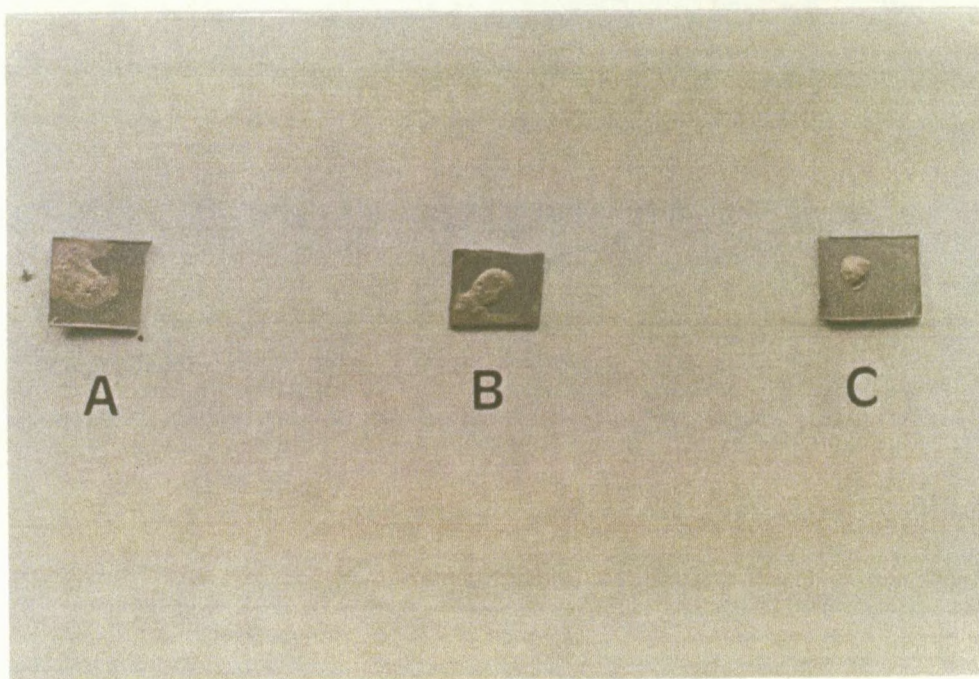


FIGURE 3.5: GOLD PLATED SOLDER WETTING TEST SHOWING:

- A - Degulor - Lot 2 solder with Minoxyd flux.
Note excellent wetting by the solder.
- B - Degulor - Lot 2 solder with Minoxyd flux.
Not gold plated. Note good wetting by the solder.
- C - Degulor - Lot 2 solder with Resistal flux.
Note minimal wetting by the solder.

The result of the above wetting tests meant that a flux that does discolour porcelain had to be used. In order to establish the differences between the fluxes letters were sent to the manufacturers requesting information about the composition and function of the elements in the fluxes. The manufacturer Degussa failed to reply after a letter had been sent directly to them and after a request sent via their local agent. Bego replied that their fluxes Minoxyd and Fluxsol were developed on an empirical basis and that they were a complex formula of potassium - sodium - fluoro - borate [Gundlach, 1992]. As this did not explain the differences between the fluxes and as no information was available for the Degussa fluxes the two Degussa fluxes Resistal flux and T flux plus the Bego flux Minoxyd were sent to the Department of Chemical Sciences - Analytical Chemistry of Technikon Natal for analysis (Figure 3.6).

Analysis by atomic absorption showed that all three fluxes consisted of basically the same elements, but the concentration of fluoro-borate in the Minoxyd flux, which does discolour porcelain, is higher than in the other two which don't discolour the porcelain [Sparks, 1992].

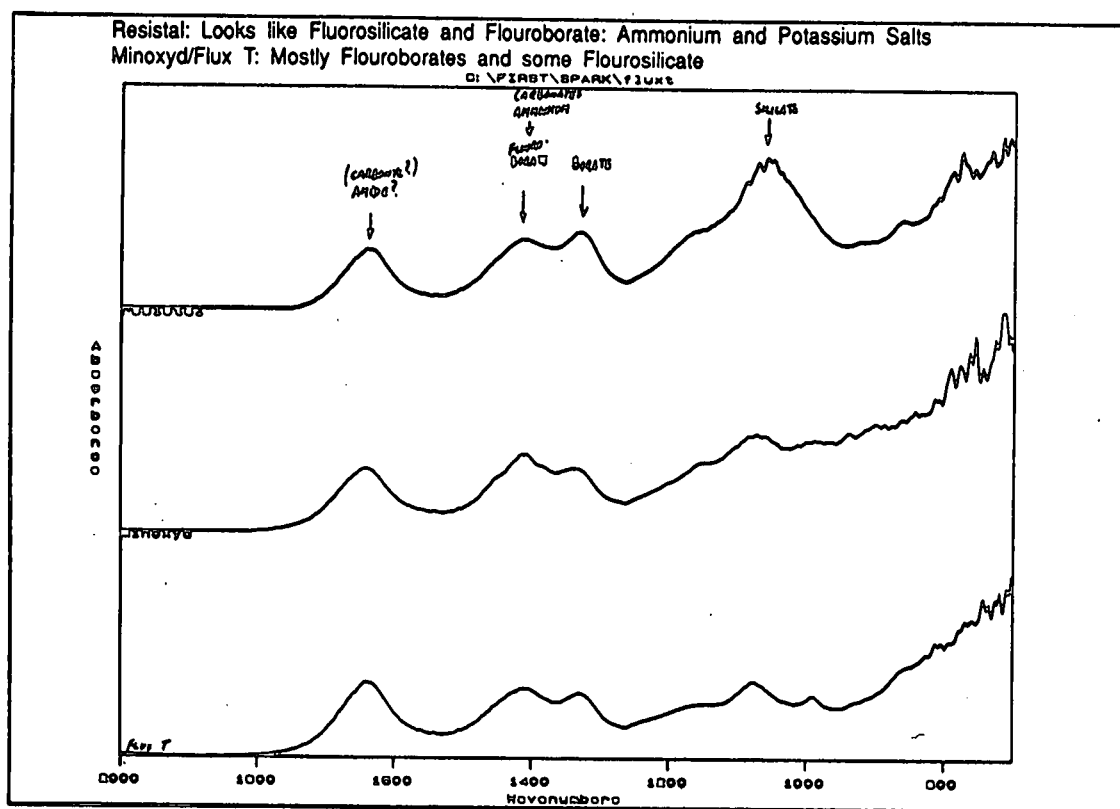


FIGURE 3.6: GRAPH SHOWING ANALYSIS OF FLUXES BY ATOMIC ABSORPTION
 (Fluxes analysed - T flux, Minoxyd and Resistal flux.
 Graph shows concentration of fluoro-borate in minoxyd flux
 is higher than that of Resistal flux and T flux.

3.3.11 PORCELAIN FURNACE SOLDERING FIRING CYCLE

The porcelain furnace used to solder the specimens was a Vita Vacumat 300 [Vita Zahnfabrik, Germany].

The following firing cycle was used to solder the specimens:

Preheating -	500°C for 8 minutes
Temperature climb -	500 - 800°C over 4 minutes
Hold temperature -	800°C for 3 minutes
Vacuum -	7 minutes

The length of the preheating time is to ensure that all moisture from the soldering investment block base has been eliminated. The time for the temperature climb is also dependent on the size of the bridge that is to be soldered and is based upon providing sufficient time for the soldering investment block and bridge to heat soak. A large case may require 6 minutes while a small cases could require as little as 3 minutes. In the same way the hold temperature time is based upon the length of time for the case to heat soak and sufficient time is allowed for the solder to be liquified and to be drawn into the gap by capillary action. This could range from 2-4 minutes depending on the size of the case. The hold temperature is usually set based on adding 50 - 55°C to the melting point of the solder being used. In this study the solder was Degulor-Lot 2 (745°C)[Degussa AG], and the melting point was 745°C.

It is important to note here that the precious and nonprecious alloys chosen had a melting temperature that would allow for a soldering temperature that was below 800°C., for the reason that postceramic soldering of porcelain restorations at a temperature between 800°C and 900°C can result in an increase in the coefficient of thermal expansion of the porcelain to the point that it surpasses that of the metal substructure with the result that the porcelain cracks on cooling [Yamamoto 1988:190]. Therefore it is important to choose an alloy that is going to act as an all-metal abutment (in this study Degulor M - 900°C and Realor - 860°C) that has a melting temperature at least 30° - 50° above 800°C so as to allow the selection of a solder that has a melting point of at least 50°C below 800°C [Description and Processing Instructions, Palladium-Silver Bonding Alloys, Degussa 1992:7].

The decision to use vacuum during the soldering cycle was based on the findings of Baran [1984:80], which showed that base metal alloys formed oxide layers approximately three times thicker when heated in air rather than in a furnace under vacuum. By applying vacuum it is assumed that the potential for oxide formation will be reduced, thus aiding the efficiency of the flux and the wetting by the solder. This decision is also supported by the conclusions of Kaylakie et. al., [1985:462].

3.4 METHOD - SUBPROBLEM ONE

The specimens for subproblem one were prepared in the same way as outlined in section 3.2, except that being the first specimens to be prepared, no small bevel was cut into the edge of the face of the surface prepared for soldering (section 3.2.9). All ten specimens were invested and fluxed as described. However, when the first specimen was placed into the porcelain furnace for the soldering cycle, the solder did not flow down the joint, but flowed back along the specimen towards the grip section. At this stage it was decided to cut a small V into the rest of the specimens using a diamond cutting disk, to channel the molten solder down into the joint. The joints were inspected visually and thought to be free of cutting swarf or contaminants. These specimens soldered at the first attempt, and after recovery and sandblasting appeared to be sound solder joints. The first specimen that did not solder was refluxed and solder was placed over the gap with a V cut into it. It was put through the soldering cycle again, resulting in an apparently sound solder joint.

Subsequent to this all other specimens for subproblem one, two and three were prepared with a slight bevel prior to assembling in the jig for investing so as to create the V to channel the molten solder down into the gap.

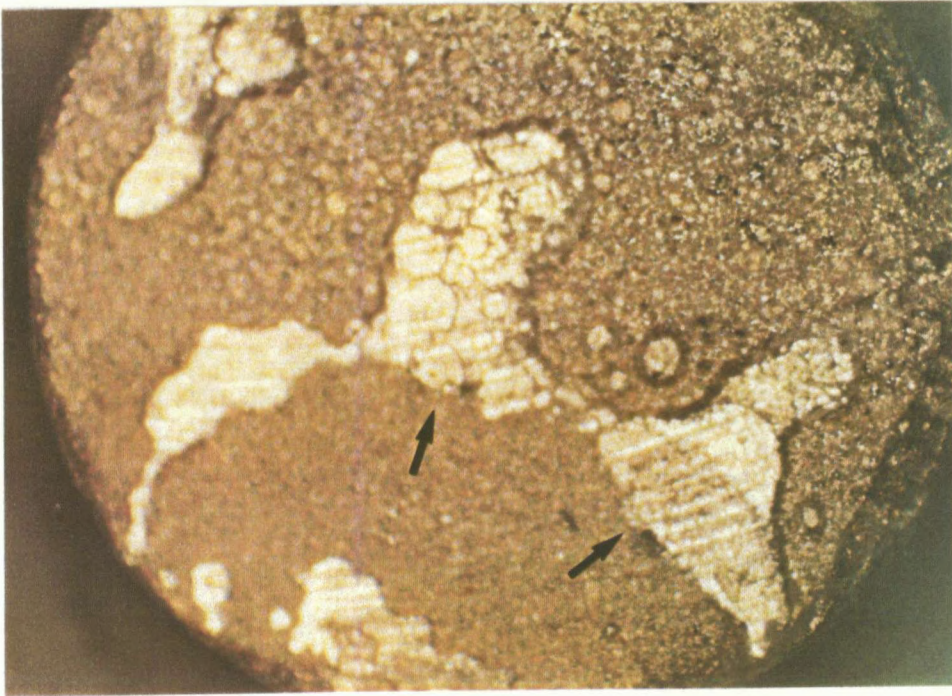


FIGURE 3.7: SHOWS SOLDER JOINT SURFACE CONTAMINATION PREVENTING WETTING OF THE SURFACE BY THE SOLDER (see arrows)

When the specimens were subjected to tensile testing, two of the specimens produced strengths that were below the minimum specification of 400 MPa. Microscope analysis of the specimens showed that some of the joints had been contaminated by cutting debris and swarf (Figure 3.7). This result did not comply with the requirement for a 100% predictable result (Table 4.1, Precious to precious 1st attempt).

On the basis of this evidence the sample was rejected and a new sample was prepared taking careful note of all the steps, especially the section on surface preparation, outlined in the general description of the method. All the tensile strengths for the second sample were above the minimum specification of 400 MPa (Table 4.1, Precious to precious 2nd attempt), and fulfilled the requirement for a 100% predictable result.

3.5 METHOD - SUBPROBLEM TWO

The specimens for subproblem two were prepared in the same way as outlined in section 3.2. using the flux, Minoxyd flux [Bego, Germany].

Prior to Minoxyd flux being chosen, the initial choice was Resistal flux [Degussa AG, Germany], which is recommended by the manufacturer, Degussa, for postceramic soldering the base metal alloy Resistal P [Degussa AG, Germany]. The first specimen to be

soldered from the group base metal to base metal was unsuccessful as the solder did not flow down into the solder gap but formed a ball in the V above the gap.

An exercise was then carried out to identify a suitable alternative flux. This consisted of a wetting test using the following brands of flux on cast square sheets of the base metal alloy Resistal P:

Fluxes that do not discolour porcelain

1. Resistal flux with Degulor-Lot 2 (745°C)

[Degussa AG] solder

2. Fluxsol flux [Bego, Germany] with Degulor-Lot 2 (745°C)

[Degussa AG] solder

Fluxes that do discolour porcelain

3. Minoxid flux [Bego, Germany] with Degulor-Lot 2 (745°C)

[Degussa AG] solder

There was no wetting by the solder with the Resistal flux, or with the Fluxsol flux. However there was good wetting by the solder with the Minoxid flux (Figure 3.4).

Based on the above findings the samples of the three groups in this subproblem, base metal to base metal, base metal to semi-precious and base metal to precious, were soldered using

Minoxyd flux so as to ensure adequate wetting of the base metal specimens.

3.6 METHOD - SUBPROBLEM THREE

The specimens for subproblem three were prepared in the same way as outlined in the section 3.2. using the flux Minoxyd flux [Bego, Germany].

Prior to deciding to use Minoxyd flux on the gold plated samples a wetting test was carried out on a cast sheet of base metal which had been gold plated for five minutes using Degulor-Lot 2 (745°C) [Degussa AG] solder with Resistal flux and T Flux [Degussa AG, Germany]. This was to establish if the gold plating would enhance the solderability of the base metal alloy to the extent that a flux that did not discolour the porcelain could be used. The result was only partial wetting of the surface with the Resistal flux and no wetting with the T Flux. A decision was then made to discard the T Flux.

A second wetting test was then carried out to increase the length of time for the plating process and thereby the thickness of the gold plate, in order to establish if the wetting of the solder could be improved.

Four cast sheets were prepared using a plating time of 10, 15, 20 and 25 minutes respectively. There was a slight improvement in the wetting by the solder up to the 15 minute time, but no improvement beyond this.

As a comparison, two cast base metal alloy sheets were prepared, one gold plated for 15 minutes and the other with no gold plating. These were tested using Minoxyd flux. After soldering, it was observed that a greater amount of wetting on the gold plated specimen occurred compared to the non-gold plated specimen.

Overall, when the Minoxyd flux test was compared to the Resistal flux test, a significant difference was observed between the Resistal flux, which exhibited only partial wetting despite the increased gold plating time, and the Minoxyd flux which exhibited good wetting, especially when gold plated (Figure 3.5).

Due to the fact that partial wetting had been observed with the Resistal flux, the first specimen from the group, base metal to base metal, was soldered using Resistal flux. After the soldering process it was observed that only partial wetting had occurred and that the solder had only flowed a third of the way into the gap.

At this stage it was decided to change to Minoxyd flux and the rest of the samples for subproblem three were soldered using Minoxyd flux.

CHAPTER FOUR

RESULTS

4.1 TENSILE TEST RESULTS

The results of the tensile tests on the samples for subproblem one, two and three, are summarised in Table 4.1 and Figure 4.1 showing the strengths in descending order.

TABLE 4.1 - SUMMARY OF TENSILE TEST RESULTS FOR
SUBPROBLEMS 1, 2 AND 3. TENSILE STRENGTHS IN MPa.

	CONTROL PRECIOUS TO PRECIOUS		SUBPROBLEM TWO NO GOLD PLATING			SUBPROBLEM THREE WITH GOLD PLATING		
	*1ST ATTEMPT	2ND ATTEMPT	BM TO BM	BM TO SP	BM TO P	BM TO BM	BM TO SP	BM TO P
1	656.60	645.60	501.50	624.20	551.90	529.90	511.40	619.30
2	650.20	613.20	485.90	506.00	526.20	507.90	459.50	593.80
3	630.00	583.10	435.60	477.00	506.00	506.80	406.60	558.70
4	605.60	575.80	418.80	475.30	503.40	499.90	385.60	538.80
5	599.40	575.80	412.50	463.30	500.40	483.00	341.20	477.00
6	495.40	569.10	407.40	449.40	496.40	458.50	334.80	426.60
7	447.20	566.70	194.50	426.00	457.40	456.00	260.00	353.60
8	442.20	542.30	193.30	421.50	454.40	450.60	246.30	
9	397.00	483.20		314.00	445.90	268.10		
10	395.40	459.60			339.70			
**	(n = 10)	(n = 10)	(n = 8)	(n = 9)	(n = 10)	(n = 9)	(n = 8)	(n = 7)
MEAN	531.90	561.44	381.19	461.86	478.17	462.30	368.18	509.69
SD	106.73	55.36	120.49	81.88	58.87	77.76	91.83	95.45
CV	.20	.09	.31	.17	.12	.16	.24	.18
MIN	400.00	400.00	360.00	350.00	400.00	360.00	350.00	400.00
SPEC								

BM = BASE METAL ALLOY

SP = SEMI-PRECIOUS ALLOY

P = PRECIOUS ALLOY

* This column shows the tensile strengths from the first attempt at establishing a control. This sample was rejected because two results were below specification.

** The calculations for the mean, standard deviation and coefficient of variation are based on the number of specimens that were tensile tested and not (n = 10).

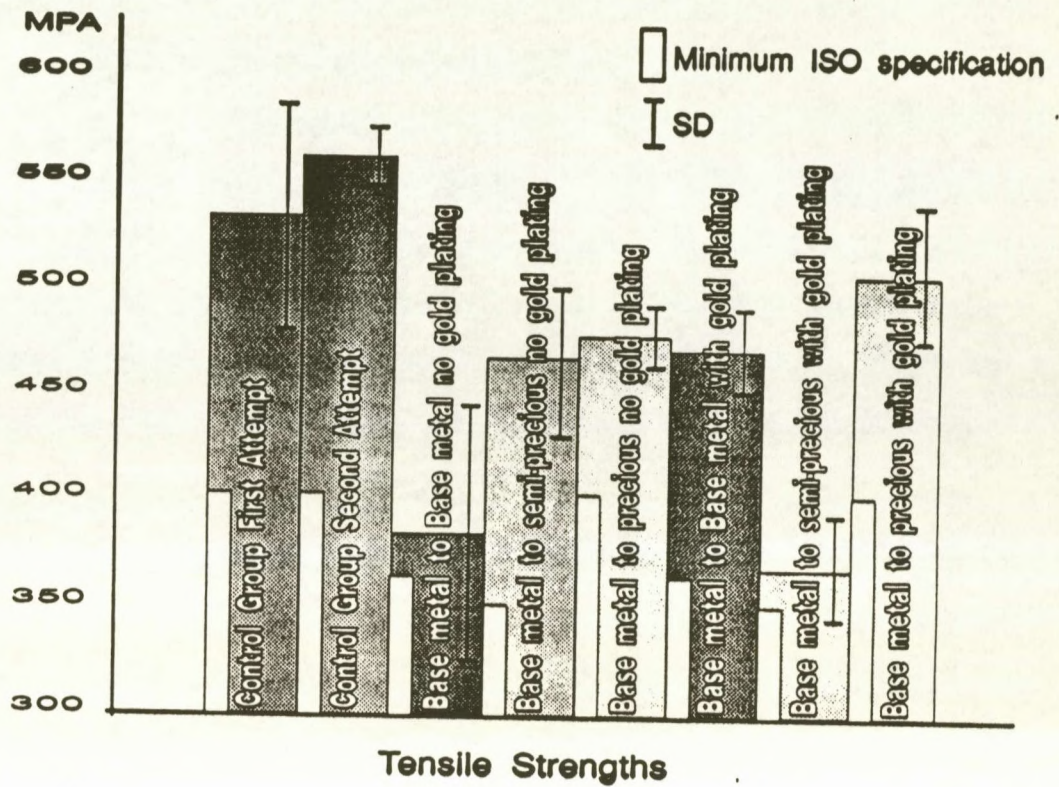


FIGURE 4.1: BAR GRAPH SHOWING MEAN TENSILE STRENGTHS FOR SUBPROBLEMS 1, 2 AND 3.

4.2 EXPLANATION OF TENSILE TEST RESULTS IN TABLE 4.1

The data in Table 4.1 have been arranged from the highest to the lowest so as to facilitate comparison between the various columns.

4.2.1 SUBPROBLEM ONE

Control

The second attempt sample produced a mean tensile strength of 561.44 MPa with a relatively small standard deviation (SD) of 55.36 MPa and a small coefficient of variation (CV) of 0.09. The mean strength was well above the minimum specification set for the study of 400 MPa and was also above the tensile strength of the parent metal of 520 MPa(soft), [Technical Data, Degussa AG, Germany, 1987]. None of the specimens in the sample was below minimum specification (Table 4.1). This is in contrast to the first attempt sample where two of the specimens produced breaks that were below specification, hence the reason for the second attempt. In addition, the CV of the first attempt was relatively high at 0.20 when compared to the CV of 0.09 in the second attempt.

The results of the second attempt sample are better than those of previous studies:

Mean (MPa)	SD	CV	Reference
561.44 ± 55.36	0.09		Control - 2nd attempt
508.23 ± 56.94	0.11		[Stade et. al., 1975:527]
394.15 ± 95.21	0.24		[Stade et. al., 1975:527]
460.58 ± 182.71	0.41		[Marshall et. al., 1984:668]
324.06 ± 34.47	0.10		[Sloan et. al., 1982:688]
303.38 ± 97.08	0.32		[Staffanou et. al., 1980:33]

Comment

The method that produced the results is taken as valid and reliable because the results compare well with the previous studies cited.

4.2.2 SUBPROBLEM TWO

Subproblem two - base metal to base metal, no gold plate

The sample produced a mean tensile strength of 381.19 MPa with a relatively large SD of 120.49 MPa, and a relatively large CV of 0.31. The mean strength was just above the minimum specification set for the study of 360 MPa and was well below the tensile strength of the parent metal of 660 MPa (as cast), [Technical Data, Degussa AG, Germany, 1987]. There were only eight

specimens in the sample because one specimen broke while being machined on the lathe and one tensile readout could not be accepted due to an error in setting the testing machine. Of the eight specimens tested two were below the minimum specification of 360 MPa (Table 4.1).

The following list details the results of other studies which evaluated base metal to base metal gold solder joints:

Mean (MPa)	SD	CV	Reference
634.90 ± 91.6	0.14		Sobieralski [1987:40]
550.00 ± 46.1	0.08		Kaylakie et. al., [1985:459]
524.02 ± 62.88	0.12		Staffanou et. al., [1980:33]
381.19 ± 120.49	0.31		Base metal to base metal no G.P.
365.43 ± 55.16	0.15		Sloan et. al., [1982:688]
282.00 ± 102.04	0.36		Marshall et. al., [1984:668]
275.00 ± 70.6	0.25		Kaylakie et. al., [1985:459]

The CVs reported in other studies range from 0.36 to 0.08, with only Kaylakie et. al., achieving the CV under 0.10.

Comment

The results obtained in this study are considered valid and reliable for the following reasons:

- these results are similar to the results reported in the list above
- the mean of the results obtained in this study is above the minimum specification.

Subproblem two - base metal to semi-precious, no gold plate

The sample produced a mean tensile strength of 461.86 MPa, a SD of 81.88 MPa and a CV of 0.17. The mean strength was just above the minimum specification set for the study of 350 MPa and was above the tensile strength of the parent metal of 240 MPa(soft), [Technical Data, Degussa AG, Germany, 1987]. There were only nine specimens in the sample because one specimen broke while being machined on the lathe. Of the nine specimens only one tested below the minimum specification of 350 MPa (Table 4.1).

Below is a comparison with previous studies which shows that these results are similar. Once again, none of the CVs were below 0.10 which indicates the wide range of results produced and the difficulty in producing consistent results when base metal alloys are involved in the solder joint.

Mean	SD	CV	Reference
(MPa)			
461.86 ± 81.88	0.17		Base metal to Semi-Precious, no G.P.
448.17 ± 224.08	0.50		Staffanou et. al., [1980:33]
365.43 ± 68.95	0.18		Sloan et. al., [1982:688]

Comment

Based on the similarity of the SD and CV of this study to those of Sloan et. al., and being substantially better than those of Staffanou et. al., the results can be considered as reliable and valid.

Subproblem two - Base metal to precious, no gold plate

The sample produced a mean tensile strength of 478.17 MPa, a relatively small SD of 58.87 MPa and a CV of 0.12. The mean strength was just above the minimum specification set for the study of 400 MPa but was below the tensile strength of the weakest of the parent metals of 520 MPa (soft), [Technical Data, Degussa AG, Germany, 1987]. Only one specimen tested below the minimum specification of 400 MPa (Table 4.1).

The results listed below show a comparison with previous studies:

Mean	SD	CV	Reference
(MPa)			
478.17 ± 58.87	0.12		Base metal to Precious no G.P.
317.17 ± 68.95	0.21		Sloan et. al., [1982:688]
310.27 ± 235.80	0.76		Staffanou et. al., [1980:33]

Comment

These results are substantially better than those of Staffanou et. al., and Sloan et. al., especially when the SD and CV results are considered. Therefore the result can be considered reliable and valid.

4.2.3 SUBPROBLEM THREE

For purposes of comparison in subproblem four the object in subproblem three was to establish what the effect of the gold plate would have on the solder joints in terms of the strengths they produced. Therefore comment will be confined to highlighting the main points that the table of results revealed.

Subproblem three - base metal to base metal, with gold plate

The sample produced a mean tensile strength of 462.3 MPa with a SD of 77.76 MPa and a CV of 0.16. The mean strength was above the minimum specification set for the study of 360 MPa and was well below the tensile strength of the parent metal of 660 MPa(as cast), [Technical Data, Degussa AG, Germany, 1987]. Only nine specimens were successfully soldered. The first specimen which was soldered with a flux that does not discolour porcelain only flowed a third of the way into the gap, thus requiring a change to a stronger flux. Of the nine successful solder joints one was below the minimum specification of 360 MPa (Table 4.1).

Subproblem three - base metal to semi-precious, with gold plate

The sample produced a mean tensile strength of 368.18 MPa with a SD of 91.83 MPa and a relatively high CV of 0.24. The mean strength was just above the minimum specification set for the study of 350 MPa and was above the tensile strength of the parent metal of 240 MPa (soft), [Technical Data, Degussa AG, Germany, 1987]. There were only eight specimens in the sample

because two specimens broke while being machined on the lathe. Of the eight specimens four tested below the minimum specification of 350 MPa (Table 4.1).

Comment

The high CV of 0.24 combined with the fact that 50% of the sample tested below the minimum specification would question the reliability of this result. The implications of this will be discussed in section 5.3.

Subproblem three - base metal to precious, with gold plate

The sample produced a mean tensile strength of 509.69 MPa with a SD of 95.45 MPa and a CV of 0.18. The mean strength was above the minimum specification set for the study of 400 MPa but was below the tensile strength of the weakest of the parent metals of 520 MPa (soft), [Technical Data, Degussa AG, Germany, 1987]. There were only seven specimens in the sample because in three of the specimens the solder only flowed $\pm 20\%$ into the gap. Of the seven specimens one specimen tested below the minimum specification of 400 MPa (Table 4.1).

4.2.4 SUMMARY OF TENSILE TEST RESULTS

The major trends that are evidenced by Table 4.1 are:

1. The control group (second attempt) produced the best results. This is based on the fact that it produced the highest mean of all the samples and the lowest SD and CV of all the samples. In addition none of the specimens tested below minimum specification.
2. Pertaining to subproblem two, the base metal to precious with no gold plating sample produced the best results because it produced the lowest SD and CV. The worst sample was the base metal to base metal no gold plating which produced the highest SD and CV, as well as two specimens that tested below minimum specifications.
3. Pertaining to subproblem three, the base metal to base metal with gold plating sample produced the lowest SD and CV. The least successful sample was the base metal to semi-precious with gold plating which produced the highest SD and CV, and breaks occurred below the minimum specification in 50% of the sample.
4. With regard to the reliability and validity of the results, the control and subproblem two samples compared well to the previous studies cited and they all produced mean strengths that were above the minimum specification. Although the

samples pertaining to subproblem three could not be compared to previous studies, they also produced mean strengths that were above the minimum specification.

4.3 LIGHT MICROSCOPE RESULTS

4.3.1 Subproblem One

A description of the end-on and side-on view of the solder joint breaks is given in Table 4.2. The data are to be read in conjunction with the photographs in Annexure B.

4.3.2 Subproblems Two and Three

A description of the end-on and side-on view of the solder joint breaks is given in the following Tables, which are to be read in conjunction with the photographs in the relevant Annexures:

Table Annexure

Subproblem two

base metal to base metal	4.3	C
base metal to semi-precious	4.4	D
base metal to precious	4.5	E

Subproblem three

base metal to base metal	4.6	F
base metal to semi-precious	4.7	G
base metal to precious	4.8	H

IT SHOULD BE NOTED that the colour of the photostat prints in the annexures will be slightly dissimilar to the original colour photograph prints which, as a result of processing, are slightly dissimilar to specimens that were originally photographed. However, they are similar enough to illustrate the reality of the physical specimen and therefore are taken as valid.

4.4 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.2 - CONTROL

The microscopic evidence showed that all the breaks occurred through the solder. These findings differ from Staffanou et. al., [1980:33] and Sloan et. al., [1982:688] who reported that half the breaks had occurred through the solder and half through the parent metal. In this test, only one specimen, 2A&B, showed one small porosity hole and only one specimen, 4A&B, showed two small points of flux inclusion. Otherwise the rest of the sample was porosity free.

The two side-on mounted specimens showed good surface apposition and contact at the solder to parent metal interface. These findings are similar to the results reported by Marshall et. al., [1984:668], Staffanou et. al., [1980:33] and Sloan et. al., [1982:688]. There was no sign of alloying between the solder and the parent metal as the interface between the solder and the parent metal is clearly visible. Specimen 3A&B showed a few small porosity holes in the solder and specimen 3A showed a few porosity holes in the parent metal.

**TABLE 4.2 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF
SOLDER JOINT BREAKS - SUBPROBLEM ONE - CONTROL**

All magnification X50 unless stated otherwise.
Strengths in MPa.

* = observation highlighted in annexure by arrow.

SPECIMEN	OBSERVATIONS - END-ON VIEW	STRENGTH
1 A&B	Break through the solder. No porosity.	566.70
2 A&B	Break through the solder. One small porosity hole.*	645.60
3 A&B	Break through the solder. No porosity.	459.60
4 A&B	Break through the solder. Two small porosity holes, from flux inclusion.*	542.30
5 A&B	Break through the solder. No porosity.	583.10
6 A&B	Break through the solder. No porosity.	569.10
7 A&B	Break through the solder. No porosity.	613.20
8 A&B	Break through the solder. No porosity.	483.20
9 A&B	Break through the solder. No porosity.	575.80
10 A&B	Break through the solder. No porosity.	575.80

SPECIMEN	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
2 A&B	This was the strongest specimen. The interface between the solder and Degulor M alloy can be seen especially on 2A *. There is no sign of porosity and the break occurs through the solder.	645.60
3 A&B	This was the weakest specimen. The interface between the solder and Degulor M alloy can be seen on both specimens 3A and 3B. There are porosity holes in the solder which probably accounts for the lower strength of the joint.*	459.60

4.5 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.3 - BASE METAL TO BASE METAL, NO GOLD PLATE

The microscopic evidence showed that the majority of the breaks were a combination of cohesive (solder to solder) and adhesive (solder to parent metal). The adhesive break appeared to be an oxide to oxide break as evidenced by the grey substance which appeared on both surfaces. There appeared to be no relationship between the type of break and the strength of the joint. These findings are similar to the results reported by Marshall et. al., [1984:670], Sobieralski [1987:39] and Sloan et. al., [1982:688]. However there does appear to be a direct

relationship between the strength and the 30% lack of wetting by the solder in specimen AB1&2. No explanation can be offered as to the poor strength performance of specimen AE1&2 which had a 95% cohesive break that appeared porosity free. There was evidence of small amounts of flux inclusion in half of the joints.

Of interest, is specimen AJ1&2 which hooked the cutting tool and broke on the lathe. This specimen showed a combination of cohesive within the parent metal and an adhesive break. This is similar to the findings reported by Kriebel, Moore, Goodacre and Dykema, [1984:62]. Kriebel did not use tensile testing to break the solder joints but instead used a bending method. Specimen AJ1&2 was probably subjected to a similar stress when it hooked the lathe cutting tool.

Although specimen AF1&2 produced a strength of 501.50 MPa, the side-on view showed the presence of porosity in the solder joint. The side-on views of specimen AJ2 showed good wetting by the solder to the parent metal as well as a porosity free solder joint.

TABLE 4.3 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF SOLDER JOINT BREAKS - SUBPROBLEM TWO - BASE METAL TO BASE METAL, NO GOLD PLATE

All magnification X50 unless stated otherwise.

Strengths in MPa

SAMPLE column - 1 = base metal specimen and

2 = semi-precious specimen.

* = observation highlighted in annexure by arrow.

SAMPLE	OBSERVATIONS - END-ON VIEW	STRENGTH
AA1 & AA2	60% solder to solder break. 10% oxide to oxide on AA2 interface. 30% oxide to oxide on AA1 interface. One small spot of flux inclusion.	407.40
AB1 & AB2	Break occurred on the AB2 interface. 30% oxide to oxide *, with 70% very good wetting as the break is a solder to solder fracture. Several small spots of flux inclusion.	193.30
AC1 & AC2	45% of break solder to solder. 5% interface break on AC1 50% interface break on AC2. The interface break appears to be oxide to oxide.	error in read-out
AD1 & AD2	Break on AD2 interface, 10% solder to solder, and 90% oxide to oxide. Small number of spots of flux inclusion. *	435.60
AE1 & AE2	95% solder to solder break. 5% oxide to oxide on AE2 interface. Appears porosity free.	194.50
AF1 & AF2	70% solder to solder break. 30% oxide to oxide on AF1 interface.	501.50
AG1 & AG2	70% oxide to oxide break on AG2 interface. 30% solder to solder break.	485.90
AH1 & AH2	65% solder to solder break. 35% oxide to oxide on AH1 interface.	412.50
AI1 & AI2	70% solder to solder break. 10% oxide to oxide on AI1 interface. 20% oxide to oxide on AI2 interface. One small spot of flux inclusion.	418.80
AJ1 & AJ2	Hooked and broke on lathe. Appears to be 100% oxide to oxide break on AJ1.	

SAMPLE	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
AB1 & AB2	X400 magnification. Dark line at interface shows possibility of oxide layer. *	193.30
AF1 & AF2	Note porosity* near AF2 interface. The break at the AF1 solder to solder appears to be through a line of porosity in a similar position to the porosity line near the AF2 interface.	501.50
AF2	X400 magnification. Shows close-up of porosity.*	
AJ1 & AJ2	X50 magnification shows break to be on AJ1 interface. However X200* and X400 magnification show that part of the break occurred through the parent metal. Of interest is that there is no sign of the dark line of possible oxide as seen on the X400 magnification on AB1.*	

4.6 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.4 - BASE METAL TO SEMI-PRECIOUS, NO GOLD PLATE

The microscopic evidence showed that the majority of the breaks were a combination of 30% cohesive (solder to solder) and 70% adhesive (solder to base metal). These adhesive breaks appeared to be oxide to oxide as evidenced by the grey substance that was present to differing degrees on the surfaces of the breaks. None of the breaks occurred at the semi-precious to solder interface. There appeared to be some relationship between the type of break and the strength of the joint, the stronger joints being a combination of half cohesive and adhesive. This is similar to the findings reported by Sloan et. al., [1982:688], whereas Staffanou et. al., [1980:33] showed that 50% of the specimens failed in the semi-precious parent metal and 50% failed in the solder. In this test there was evidence of small amounts of flux inclusion in half of the specimens. However, this was insufficient to significantly affect the strength of the joints as the specimen with the most flux inclusion, i.e. 5%, produced the second highest break strength.

The side-on views showed that there was no sign of an interface between the solder and the semi-precious alloy. Instead there was evidence of alloying between the two. However there is a fair amount of evidence of porosity in the solder. Of particular interest was the alloying by diffusion between the gold solder and the base metal as was seen in specimen BJ1.

TABLE 4.4 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF SOLDER JOINT BREAKS - SUBPROBLEM TWO - BASE METAL TO SEMI-PRECIOUS, NO GOLD PLATE

All magnification X50 unless stated otherwise.

Strengths in MPa

SAMPLE column - 1 = base metal specimen and

2 = semi-precious specimen.

* = Observation highlighted in annexure by arrow.

SAMPLE	OBSERVATIONS - END-ON VIEW	STRENGTH
BA1 & BA2	Caught and broke on lathe. 55% oxide to oxide break on BA1 and 45% solder to solder at BA1 interface.	
BB1 & BB2	50% oxide to oxide break at BB1 interface, 50% solder to solder break. Three spots of flux inclusion.*	506.00
BC1 & BC2	40% oxide to oxide break at BC1 interface, 60% solder to solder break.	449.40
BD1 & BD2	60% oxide to oxide break at BD1 interface, 40% solder to solder break.	624.20
BE1 & BE2	80% oxide to oxide break at BE1 interface, 20% solder to solder break.	421.50
BF1 & BF2	70% oxide to oxide break at BF1 interface, 30% solder to solder break.	463.30
BG1 & BG2	80% oxide to oxide break at BG1 interface, 20% solder to solder break.	426.40
BH1 & BH2	60% oxide to oxide break at BH1 interface, 40% solder to solder break.	475.30
BI1 & BI2	90% oxide to oxide break at BI1 interface, 10% solder to solder break.	314.00
BJ1 & BJ2	75% oxide to oxide break at BJ1 interface, 25% solder to solder break.	477.00

SAMPLE	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
BC1 & BC2	Note porosity in solder*, dark line at BC1 solder interface* - possibly oxide layer and alloying between solder and semi-precious alloy.	449.40
BD1 & BD2	Note increase in alloying moving from solder to semi-precious alloy.	
BG1 & BG2	Same as in BC1&2.	624.20
BH1 & BH2	Note porosity in solder, dark line at B1 solder interface - possibly oxide layer and alloying between solder and semi-precious alloy.	426.40
BJ1	X400 magnification. Shows diffusion of solder into the base metal*.	475.30
BJ2	X400 magnification. Shows diffusion of base metal alloy into the alloyed semi-precious solder alloy.*	477.00

4.7 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.5 - BASE METAL TO PRECIOUS, NO GOLD PLATE

The microscopic evidence showed that the majority of the breaks were a combination of 15% cohesive (solder to solder) and 85% adhesive (solder to base metal). This was once again characterised by the appearance of a grey substance which was probably an oxide to oxide break. None of the breaks occurred at the precious to solder interface. The only joint that did not meet minimum specification showed a 70% cohesive break. This finding is different to that of Sloan et. al., [1982:688], who found that 50% of the breaks were at the base metal interface and 50% in the precious parent metal. Of interest here is that they reported no cohesive breaks. On the other hand, Staffanou et. al., [1980:33] showed that 50% of the specimens failed in the precious parent metal and 50% failed in the solder. In this test there was evidence of a small amount of flux inclusion on specimens CA1&2, CG1&2 and CI1&2. Also one large spot of flux inclusion appeared on specimen CJ2. Apart from this the specimens appear to be flux inclusion free.

The X200 magnification of the interface break on specimen CF2 revealed what appeared to be a large number of small particles of base metal which had broken from the base metal parent metal. The side-on mounted specimens showed good surface apposition and contact at the solder to parent metal interface. There was virtually no sign of porosity. No explanation can be offered for the supposed impurity spot in specimen CG2 except the possibility of base metal diffusion into the solder as was

supposedly evidenced in specimen BJ2, X400 magnification
(Annexure E).

TABLE 4.5 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF SOLDER JOINT BREAKS - SUBPROBLEM TWO - BASE METAL TO PRECIOUS, NO GOLD PLATE

All magnification X50 unless stated otherwise.

Strengths in MPa

SAMPLE column - 1 = base metal specimen and

2 = semi-precious specimen.

* = Observation highlighted in annexure by arrow.

SAMPLE	OBSERVATIONS - END-ON VIEW	STRENGTH
CA1 & CA2	80% oxide to oxide break at CA1 interface, 20% solder to solder break. *	457.40
CB1 & CB2	45% oxide to oxide break at CB1 interface, 55% solder to solder break.	500.40
CC1 & CC2	97% oxide to oxide break at CC1 interface, 3% solder to solder break.	445.90
CD1 & CD2	30% oxide to oxide break at CD1 interface, 70% solder to solder break.	339.70
CE1 & CE2	97% oxide to oxide break at CE1 interface, 3% solder to solder break.	551.90
CF1 & CF2	97% oxide to oxide break at CF1 interface, 3% solder to solder break.	496.40
CF2	X200 magnification. Appears that small amounts of base metal have broken from CF1 and adhered to solder.* X400 magnification. Appears that small amounts of base metal have broken from CF1 and adhered to solder.*	
CG1 & CG2	97% oxide to oxide break at CG1 interface, 3% solder to solder break. A few spots of flux inclusion.	503.40
CH1 & CH2	97% oxide to oxide break at CH1 interface, 3% solder to solder break.	526.20
CI1 & CI2	97% oxide to oxide break at CI1 interface, 3% solder to solder break.	506.00
CJ1 & CJ2	97% oxide to oxide break at CJ1 interface, 3% solder to solder break. A large spot of flux inclusion.*	454.40

SAMPLE	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
CB1 & CB2	Shows interface between solder and CB1 clearly.*	500.40
CF1 & CF2	Solder appears porosity free. Break at CF1 interface. Solder CF2 interface difficult to pick up. *	496.40
CG1 & CG2	Interface between solder and CG2 clearly seen. Break clearly across CG1 interface. Unknown crystallisation or impurity in solder.*	503.40
CG2	X100 & 200* magnification of impurity. Could be diffusion of base metal into solder.	

4.8 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.6 - BASE METAL TO BASE METAL, WITH GOLD PLATE

The microscopic evidence showed that the majority of the breaks were a combination of cohesive (solder to solder) and adhesive (solder to parent metal) on opposite interfaces. In other words both base metal interfaces failed half on one side and half on the other side with a tear across the solder. This was once again characterised by the appearance of a grey substance which was probably an oxide to oxide break. There appeared to be no relationship between the type of break and the strength of the joint. However there did appear to be a direct relationship between the strength and the amount of flux inclusion.

There was evidence of a significant increase in the amount of flux inclusion and porosity as compared to the base metal to base metal with no gold plating in the sample pertaining to subproblem two (Table 4.2). The cause of this could be the gold plating. However, further study would need to be done to address this question.

Of interest is the evidence provided by the side-on view on specimen DH2 at X400 magnification, showing good surface apposition and contact at the solder to gold plate interface and gold plate to base metal interface. This specimen produced the strongest tensile strength for the sample of 529.9 MPa. There was no sign of alloying between the gold plate and the solder.

TABLE 4.6 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF SOLDER JOINT BREAKS - SUBPROBLEM THREE - BASE METAL TO BASE METAL, WITH GOLD PLATE

All magnification X50 unless stated otherwise.

Strengths in MPa

SAMPLE column - 1 = base metal specimen and

2 = semi-precious specimen.

* = Observation highlighted in annexure by arrow.

SAMPLE	OBSERVATIONS - END-ON VIEW	STRENGTH
DA1 & DA2	95% oxide to oxide break on DA1 interface. 5% solder to solder break. There appears to be gold plating showing beneath the oxide of DA1 and also on the surface of DA2.	506.80
DB1 & DB2	10% solder to solder break, 60% oxide to oxide break on DB1 interface and 30% oxide to oxide break on DB2 interface.	499.90
DC1 & DC2	95% oxide to oxide break on DC1 interface. 5% solder to solder break. Several spots of flux inclusion.*	456.00
DD1 & DD2	10% parent metal to parent metal break on DD1, 25% solder to solder break, 10% flux inclusion, 20% oxide to oxide break on DD1 interface and 35% oxide to oxide break on DD2 interface.	450.60
DE1 & DE2	25% solder to solder break, and 75% oxide to oxide break on DE2 interface. There appears to be gold plating showing beneath the oxide of DE2 and also on the surface of DE1. A few spots of flux inclusion.	458.50
DF1 & DF2	Large number of small spots of flux inclusion. * This does not appear to have significantly affected the strength of the joint. There appears to be gold plating showing beneath the oxide of DF1. 95% oxide to oxide break at DF1 interface, 5% solder to solder break.	507.90
DG1 & DG2	Interface break at DG2. This appears to be an oxide to parent metal/gold plate break. DG1 shows a smooth clean surface whereas the surface of the solder of DG1 has the colour of oxide.	483.00
DH1 & DH2	20% solder to solder break, 40% oxide to oxide break on DH1 interface and 40% oxide to oxide break on DH2 interface.	529.90
DI1 & DI2	5% parent metal to parent metal break on DH2, 25% solder to solder break, 15% flux inclusion,* 5% oxide to oxide break on DI1 interface and 50% oxide to oxide break on DI2 interface. Low strength probably due to large amount of flux inclusion.	268.10

SAMPLE	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
DB1 & DB2	Interface between solder and base metal clearly seen. Example of combined cohesive and adhesive.	499.90
DD1 & DD2	Note parent metal break on DD2.*	450.60
DG1 & DG2	Example of 95% base metal interface break.	
DH2	X400 magnification. Shows interface between solder and base metal alloy with gold plating and oxide between them.*	529.90

4.9 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.7 - BASE METAL TO SEMI-PRECIOUS, WITH GOLD PLATE

The microscopic evidence showed that the majority of the breaks were a combination of cohesive (solder to solder) and adhesive (solder to gold plated base metal). None of the breaks occurred in the semi-precious alloy. There appeared to be no relationship between the type of break and the strength of the joint. However, the evidence showed large amounts of flux inclusion, especially in those specimens that produced low strength.

The side-on view photomicrographs showed that there was evidence of alloying between the solder and the semi-precious alloy. This was revealed in specimens ED2 and EG2. This phenomenon also occurred in base metal to semi-precious, no gold plate sample. The interface between the base metal and solder was clearly seen in ED1 and EG1. The layer of gold plate between the solder and base metal parent metal alloy was seen on the X400 magnification of ED1. Porosity was clearly seen in the area of the solder in EB2 which was one of the weakest joints.

TABLE 4.7 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF SOLDER JOINT BREAKS - SUBPROBLEM THREE - BASE METAL TO SEMI-PRECIOUS, WITH GOLD PLATE

All magnification X50 unless stated otherwise.

Strengths in MPa

SAMPLE column - 1 = base metal specimen and

2 = semi-precious specimen.

* = Observation highlighted in annexure by arrow.

SAMPLE	OBSERVATIONS - END-ON VIEW	STRENGTH
EA1 & EA2	50% solder to solder break, 50% Gold plate to gold plate* break at EA1 interface.	459.50
EB1 & EB2	50% gold plate to gold plate break at EB1 interface, 25% flux inclusion and 25% solder to solder break. Low strength probably due to excessive flux inclusion.	260.00
EC1 & EC2	45% solder to solder break. 55% oxide to oxide break at EC1 interface.	341.20
ED1 & ED2	15% solder to solder break, 25% flux inclusion * and 60% gold plate to gold plate break at ED1 interface.	334.80
EE1 & EE2	50% solder to solder break, 45% gold plate to gold plate break at EE1 interface. 5% flux inclusion.	385.60
EF1 & EF2	10% non wetting by solder.* 45% solder to solder break, 45% gold plate to gold plate break at EF1 interface.	406.60
EG1 & EG2	45% solder to solder break, 50% gold plate to gold plate break at EG1 interface. 5% flux inclusion.	511.40
EH1 & EH2	50% solder to solder break, 35% gold plate to gold plate break at EH1 interface. 15% flux	246.30

SAMPLE	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
ED1 & ED2	Note lack of interface between solder and semi-precious alloy on ED2 and the alloying of the solder with the parent metal. On ED1 there is a distinct interface between the solder and the base metal alloy. Also signs of porosity in the solder.	334.80
ED1	X400 magnification. Interface between the solder and the base metal alloy showing the layer of gold plating.*	
ED2	X400 magnification. Shows alloying between solder and semi-precious alloy. Also porosity holes.*	
EG1 & EG2	Note lack of interface between solder and semi-precious alloy on EG2 and the alloying of the solder with the parent metal. On EG1 there is a distinct interface between the solder and the base metal alloy. Also signs of porosity in the solder.*	511.40
EB2	Note excessive porosity or flux inclusion in the solder.*	260.00

4.10 EXPLANATION OF LIGHT MICROSCOPE RESULTS IN TABLE 4.8 - BASE METAL TO PRECIOUS, WITH GOLD PLATE

The microscopic evidence showed that the breaks were either cohesive (solder to solder) or adhesive (solder to gold plated base metal). Two specimens, FC1&2 and FD1&2, showed evidence of the grey coloured substance, probably oxide, which had been present on the base metal surfaces of the other samples. Four of the specimens namely FA1&2, FB1&2, FC1 and FF1&2 showed significant amounts of flux inclusion. There appeared to be a direct relationship between the stronger breaks being of an adhesive nature and the weaker breaks being of a cohesive nature.

The side-on views of FA1 and FB1 showed the interface between the solder and the base metal. This illustrates the fact that in these areas the bond strength between the solder to gold plated base metal alloy was stronger than the cohesive strength of the solder. In contrast, the side-on view of FD1&2 showed a clean break at the base metal interface. Of interest is the fact that the interface between the solder and the precious metal was not seen, whereas it could be distinguished in the specimens in the samples of the control group and the base metal to precious, no gold plate sample.

TABLE 4.8 - DESCRIPTION OF END-ON AND SIDE-ON VIEW OF SOLDER JOINT BREAKS - SUBPROBLEM THREE - BASE METAL TO PRECIOUS, WITH GOLD PLATE

All magnification X50 unless stated otherwise.

Strengths in MPa

SAMPLE column - 1 = base metal specimen and

2 = semi-precious specimen.

* = Observation highlighted in annexure by arrow.

SAMPLE	OBSERVATIONS - END-ON VIEW	STRENGTH
FA1 & FA2	70% solder to solder break, 15% flux inclusion.* 5% oxide to oxide break and 10% gold plate to gold plate break at FA1 interface.	426.60
FB1 & FB2	85% solder to solder break, 5% flux inclusion, 10% gold plate to gold plate break at FB1 interface	353.60
FC1 & FC2	5% flux inclusion, 5% oxide to oxide break at FC1 interface and 90% gold plate to gold plate break. Note presence of grey substance on surface.*	558.70
FD1 & FD2	10% solder to solder break, 25% oxide to oxide break at FD1 interface, 65% gold plate to gold plate break at FD1 interface.	538.80
FE1 & FE2	95% gold plate to gold plate break at FB1 interface, 5% solder to solder break.	619.30
FF1 & FF2	55% solder to solder break, 5% flux inclusion, 20% gold plate to gold plate break at FB1 interface and 20% oxide to oxide break.	477.00
FG1 & FG2	95% gold plate to gold plate break at FB1 interface, 5% solder to solder break.	593.80
	Specimens FJ, FH and FI, the solder only flowed +- 20% into the gap. Due to being unsuccessful they were not subjected to tensile testing.	

SAMPLE	OBSERVATIONS - SIDE-ON VIEW	STRENGTH
FA1 & FA2	Interface between solder and FA1 clearly seen. Interface between solder and FA2 not seen.	426.60
FB1 & FB2	Interface between solder and FB1 clearly seen. Note that break occurred through the solder. Note porosity in solder in FB2 which could have caused the solder to solder break.*	353.60
FD1 & FD2	Note porosity or flux inclusion in solder in FD2.* Break at FD1 interface.	538.80
FE1 & FE2	Break at FE1 interface. Interface between solder and FE2 just visible. *	

4.11 SUMMARY OF LIGHT MICROSCOPE RESULTS

The following trends are evident from the light microscope results (Table 4.9):

1. Location of breaks. All the breaks in the control sample were cohesive through the solder. In the samples pertaining to subproblem two (no gold plating) the majority of the breaks were adhesive at the base metal interface. In contrast, the samples pertaining to subproblem three (with gold plating) showed that the majority of the breaks were half cohesive and half adhesive. The gold plating would appear to have had an influence on the nature and location of the breaks.
2. Alloying took place between the gold solder and the semi-precious parent metal alloy in both the non-gold plated and gold plated samples.
3. The gold plating appeared to have had a direct influence on the increased incidence of porosity formation in the samples pertaining to subproblem three.

4. The gold plating appeared to have had a direct influence on the increased incidence of excessive flux inclusion in the samples pertaining to subproblem three.
5. There appeared to be an indirect relationship between the amount of flux inclusion and the strength of the joints: The more flux inclusion the lower the joint strength.
6. In determining the relationship between break strength and location of the break, no distinctive pattern emerged. However, the stronger breaks in the base metal to base metal, non-gold plated and gold plated, were a mixture of adhesive through the interface and combined adhesive and cohesive. The stronger breaks in the base metal to precious, non-gold plated and gold plated, were adhesive through the base metal interface. The stronger breaks in the base metal to semi-precious, non-gold plated and gold plated, were a combination adhesive and cohesive through the solder on the base metal specimen side. This was probably due to the alloying between the semi-precious alloy and the solder.

TABLE 4.9 TABULATED SUMMARY OF LIGHT MICROSCOPE RESULTS

The table shows the relationship between the samples and the nature of the breaks, evidence seen on the break surfaces and potential effects on the break strengths.

	CONTROL	SUBPROBLEM TWO NO GOLD PLATING			SUBPROBLEM THREE WITH GOLD PLATING		
	P TO P	BM TO BM	BM TO SP	BM TO P	BM TO BM	BM TO SP	BM TO P
Cohesive break through solder	100%	3 of 10	4 of 10	Nil	Nil	Nil	2 of 7
Adhesive break at interface	Nil	3 of 10	BM 6 of 10	BM 8 of 10	3 of 9	Nil	4 of 7
Cohesive break through parent metal	Nil	1 of 10	Nil	Nil	$\frac{1}{2}$	Nil	Nil
Half cohesive and half adhesive	Nil	3 of 10	Nil	2 of 10	6 of 9	8 of 8	1 of 7
Alloying between parent metal & solder	No	No	Yes - both SP to sol BM to sol	No	No	SP Yes	P Possibly
Porosity	Minimal	Nil	Moderate	Minimal	Moderate	Moderate	Moderate
Flux inclusion	Minimal	Minimal	Minimal	Minimal	Excessive	Excessive	Excessive
Flux inclusion affected strength	No	No	No	No	Yes	Yes	Yes
Cohesive break through solder stronger	Yes	No	N/a	N/a	N/a	N/a	No
Adhesive break through inter-face stronger	N/a	Similar	No	BM Yes	Similar	N/a	BM Yes
Combined Adhesive & cohesive stronger	N/a	Similar	BM Yes	No	Similar	Yes	No

P = Precious

BM = Base metal to base metal

SP = Semi-precious

4.12 SCANNING ELECTRON MICROSCOPE RESULTS

Although the study was not aimed at analysing the metallurgy of the evidence that the light microscopy revealed, the presence of the grey substance which was reported in most of the samples on the base metal to solder interface, appeared to be an oxide layer. This has been referred to as an oxide to oxide break in the microscopy results - Tables 4.4 to 4.9. This phenomenon was also observed in previous studies [Kaylakie, et. al., 1985:460; Staffanou, et. al., 1980:33, and Marshall et. al., 1984:671], and the authors hypothesised that it was an oxide layer. However no SEM evaluation was carried out to confirm this.

In order to establish whether this was an oxide, a typical specimen exhibiting this grey oxide-like substance (Figures 4.2 & 4.3) was analysed using the method outlined in section 3.2.18. The results showed that there was no oxygen present and therefore this substance was not an oxide.

QUALITATIVE ELEMENT IDENTIFICATION

SAMPLE ID:CC 1-SUBSTANCE ON SURFACE

POSSIBLE IDENTIFICATION

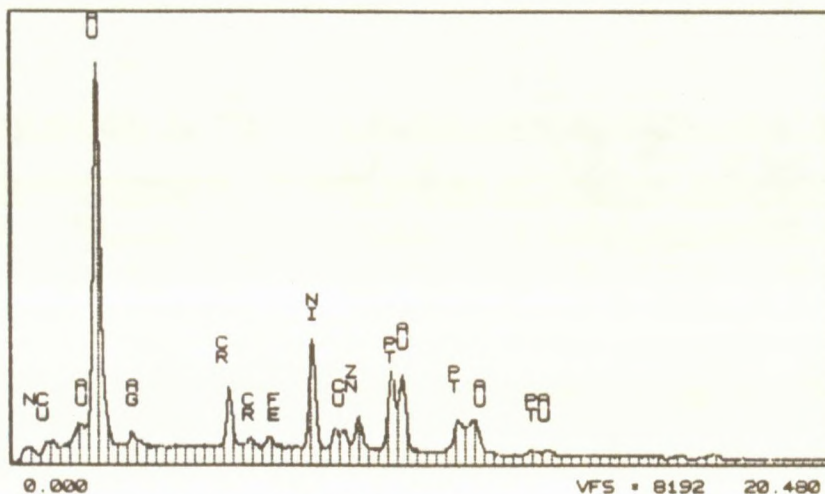
AU LA LB MA MZ1
 NI KA KB
 CR KA KB OR PM LA LB
 ZN KA OR PT LA LB LB
 ZN KA OR RE LA
 CU KA LA
 AR KA OR AG LA OR TH LA MA
 FE KA
 N KA

SERIES II

THU 17-DEC-92 15:16

Cursor: 0.000KeV = 0

ROI (0) 0.000: 0.000



PEAK LISTING

	ENERGY	AREA	EL. AND LINE
1	0.365	1527	N KA
2	0.942	1539	CU LA
3	1.662	1458	AU MZ1
4	2.122	48244	AU MA
5	3.005	1669	AG LA
6	5.428	8636	CR KA
7	5.952	1202	CR KB
8	6.419	1577	FE KA
9	7.486	16282	NI KA
10	8.038	1991	CU KA
11	8.283	1504	NI KB
12	8.638	4633	ZN KA
13	9.439	8191	PT LA
14	9.731	7873	AU LA
15	11.092	2723	PT LB
16	11.516	2709	UNIDENTIFIED
17	12.940	778	PT LB
18	13.379	939	AU LB



FIGURE 4.2: SEM ANALYSIS OF GREY SUBSTANCE ON BREAK SURFACE OF SPECIMEN CC1 (see arrow).

QUALITATIVE ELEMENT IDENTIFICATION

SAMPLE ID: CC 2-SUBSTANCE ON SURFACE

POSSIBLE IDENTIFICATION

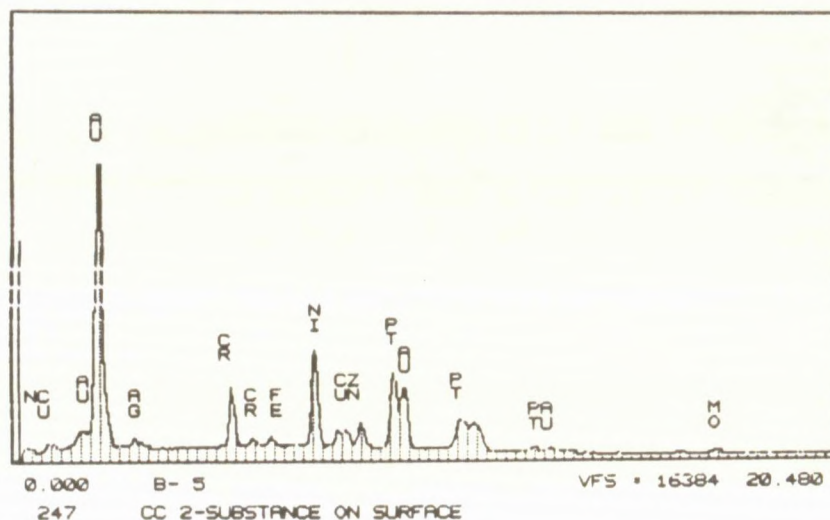
AU LA LB MA MZ1
 NI KA KB
 ZN KA OR PT LA LB LB
 CR KA KB OR PM LA LB
 ZN KA OR RE LA
 FE KA
 CU KA LA
 N KA
 AR KA OR AG LA OR TH LA MA
 MO KA

SERIES II

THU 17-DEC-92 15:32

Cursor: 0.000keV = 0

ROI (0) 0.000: 0.000



PEAK LISTING

	ENERGY	AREA	EL. AND LINE
1	0.365	2601	N KA
2	0.925	2044	CU LA
3	1.663	2193	AU MZ1
4	2.118	74058	AU MA
5	2.998	2174	AG LA
6	5.424	16258	CR KA
7	5.947	2392	CR KB
8	6.402	3053	FE KA
9	7.484	29232	NI KA
10	8.046	2676	CU KA
11	8.286	2741	NI KB
12	8.639	7341	ZN KA
13	9.440	16359	PT LA
14	9.794	11676	AU LA
15	11.090	5364	PT LB
16	11.512	4035	UNIDENTIFIED (Au)
17	12.945	1453	PT LB
18	13.376	1977	AU LB
19	17.453	1152	MO KA

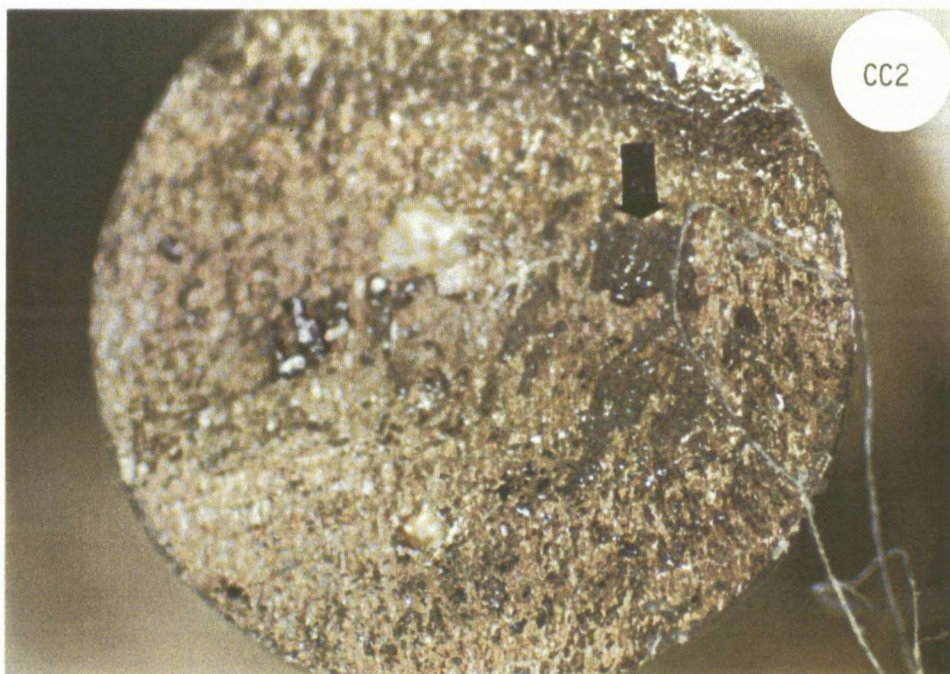


FIGURE 4.3: SEM ANALYSIS OF GREY SUBSTANCE ON BREAK SURFACE OF SPECIMEN CC2 (see arrow).

4.13 STATISTICAL RESULTS

4.13.1 SUBPROBLEM ONE - CONTROL

Tensile strength:

One sample sign test.

The null hypothesis of the sample mean for the sample, precious to precious - control, equalling the specification of 400 MPa, was rejected at $P = 0,005$, and the alternative hypothesis of the sample mean being greater than 400 MPa was accepted. The result - the sample produced a mean tensile strength that was higher than the minimum specification of 400 MPa at a 99.5% confidence level.

Predictability:

For the control, 10 successful solder joints were produced out of a sample size $n = 10$. The binomial test showed that the specified proportion of 100% was being satisfied at a 99,9% confidence level (i.e. $P = 0,001$).

4.13.2 SUBPROBLEM FOUR

Tensile strength:

Note: All the sample means are based on $n =$ the number of specimens tested and not on $n = 10$.

significant difference between sample means for gold plating versus no gold plating - Mann-Whitney U test.

This statistical test is to show if there is *statistically* a significant difference between the sample mean of the non-gold plated sample compared to the gold plated sample. If there is a significant difference, this will *statistically* imply that the gold plating has had a *statistically* significant improvement or worsening effect on the sample mean. If there is an insignificant *statistical* result this will show that the gold plating has had no effect.

The test showed the following:

1. Base metal to base metal.

In this case the sample mean of base metal to base metal, with gold plate, was significantly larger than the sample mean, base metal to base metal, no gold plate, for $P = 0,05$, that is at a 95% confidence level.

This *statistically* implies that the gold plating significantly improved the solderability.

2. Base metal to semi-precious.

In this case the sample mean of base metal to semi-precious, with gold plate was significantly lower than the sample mean, base metal to semi-precious, no gold plate for $P = 0,05$, that is at a 95% confidence level.

This statistically implies that the gold plating significantly lowered the solderability.

3. Base metal to precious.

In this case the sample mean of base metal to precious, with gold plate, was significantly larger than the sample mean base metal to precious, no gold plate, for $P = 0,05$, that is at a 95% confidence level.

This statistically implies that the gold plating significantly improved the solderability.

4. Overall view of gold plating versus no gold plating.

In this case the null hypothesis of all the gold plated sample means equals all the non-gold plated sample means could not be rejected at $P = 0,05$ (5%) or even $P = 0,1$ (10%).

This implies that when an overall view is taken, gold plating the joints prior to soldering did not improve or lower the results.

One sample sign test

This test shows statistically whether or not the sample mean is above the minimum ISO specification.

1. Base metal to base metal no gold plating

The null hypothesis of the sample mean equalling the specification of 360 MPa, was not rejected at $P = 0,1$. The result - the specification was not being met at a 90% confidence level.

2. Base metal to base metal with gold plating

The null hypothesis of the sample mean equalling the specification of 360 MPa, was rejected at $P = 0,05$, and the alternative hypothesis of the sample mean greater than 360 MPa was accepted. The result - the specification was being met at a 95% confidence level.

3. Base metal to semi-precious no gold plating

The null hypothesis of the sample mean equalling the specification of 350 MPa, was rejected at $P = 0,05$, and the alternative hypothesis of the sample mean greater than 350 MPa was accepted. The result - the specification was being met at a 95% confidence level.

4. Base metal to semi-precious with gold plating

The null hypothesis of the sample mean equalling the specification of 350 MPa, was not rejected at $P = 0,1$. The result - the specification was not being met at a 90% confidence level.

5. Base metal to precious no gold plating

The null hypothesis of the sample mean equalling the specification of 400 MPa, was rejected at $P = 0,05$, and the alternative hypothesis of the sample mean greater than 400 MPa was accepted. The result - the specification was being met at a 95% confidence level.

6. Base metal to precious with gold plating

The null hypothesis of the sample mean equalling the specification of 400 MPa, was rejected at $P = 0,1$, and the alternative hypothesis of the sample mean greater than 400 MPa was accepted. The result - the specification was being met at a 90% confidence level.

Predictability:

Note: All the sample means for the following statistics are based on $n = 10$.

Relationship analysis using χ^2 statistic - gold plating
versus no gold plating (Table 4.10)

The X^2 analysis reflected that at a 95% level of confidence the success of the solder joint did not depend on whether or not gold plating was used.

Relationship analysis using X^2 statistic - control group versus all base metal solder joints (Table 4.10)

The X^2 analysis reflected that at a 95% level of confidence the success of the solder joint did depend on whether the solder joint was precious to precious and not where base metal alloys joints were involved.

Percentage of successful solder joints that met the minimum specification for each sample, n = 10

control group -	100%
base metal to base metal no gold plating -	60%
base metal to base metal with gold plating -	80%
base metal to semi-precious no gold plating -	80%
base metal to semi-precious with gold plating -	40%
base metal to precious no gold plating -	90%
base metal to precious with gold plating -	60%

Of all the above statistical tests the one that has the most significance to the commercial dental technician is the percentage of successful solder joints. The ceramist wants to know that if he uses a soldering method or is persuaded to try a new soldering method or product, it will have a 99.9% success

rate. He cannot afford costly remakes. Therefore, caution must be exercised when reporting results to convey them in a manner that has meaning for the every-day commercial dental technician.

TABLE 4.10 RELATIONSHIP ANALYSIS USING χ^2 STATISTIC

PREDICTABILITY COMPARISON BETWEEN GOLD PLATING AND NO GOLD PLATING

	SUBPROB. TWO NO GOLD PLATE SUCCESSFUL SOLDER JOINT	SUBPROB. THREE WITH GOLD PLATE SUCCESSFUL SOLDER JOINT	TOTAL
YES	23	18	41
NO	7	12	19
TOTAL	30	30	60

PREDICTABILITY COMPARISON BETWEEN CONTROL GROUP AND ALL BASE METAL SAMPLES

	CONTROL GROUP SUCCESSFUL SOLDER JOINT	BASE METAL SAMPLES SUCCESSFUL SOLDER JOINT	TOTAL
YES	10	41	51
NO	0	19	19
TOTAL	10	60	70

CHAPTER FIVE

DISCUSSION

5.1 DISCUSSION - SUBPROBLEM ONE

The hypothesis for subproblem one, the soldering technique will produce predictable, strong solder joints when soldering precious to precious alloys, thus validating the soldering method, can be accepted on the following bases:

1. The criteria for tensile strength were met.
2. The light microscope evidence confirmed the quality of the solder joints and thereby the soldering method.
3. The criteria set for the statistical analysis were met.

A discussion on each of these results follows:

1. Tensile strengths

The method used to produce the sample produced good results that met the requirements for reliability and validity (section 4.3.1), and the results obtained compared favourably with those of the previous studies (Table 5.1).

TABLE 5.1 LIST OF STRENGTHS REPORTED IN PREVIOUS STUDIES

Mean (MPa)	SD	CV	Reference	Joint gap
561.44 ± 55.36	0.09		Control - 2nd attempt	0.2 mm
508.23 ± 56.94	0.11		[Stade et. al., 1975:527]	0.7 mm
394.15 ± 95.21	0.24		[Stade et. al., 1975:527]	0.3 mm
460.58 ± 182.71	0.41		[Marshall et. al., 1984:668]	0.4 mm
324.06 ± 34.47	0.10		[Sloan et. al., 1982:688]	0.5 mm
303.38 ± 97.08	0.32		[Staffanou et. al., 1980:33]	0.3 mm

Although the strengths reported in this study appear to be better than in the previous studies, it would be incorrect to assume that the results of this study are better. The studies cited did not include the technical specifications of the materials used and as they reported that the majority of the breaks had occurred in the solder, it must be assumed that the strength of the solder was the determining factor. Indeed Sobieralski [1987:40], who studied base metal to base metal with gold solder, attributed the difference between his results and the previous studies he cited to the increased strength of the solder he used. Sloan et. al., [1982:688] and Staffanou et. al., [1980:33] reported that some breaks occurred in the parent metal. This would indicate that the tensile strength of the parent metal used in their studies was similar to the mean tensile strengths they reported. Based on this evidence it would be more correct to compare the coefficient of variation's (CV)

of the various studies, as this is a better indicator of the reliability of the method they used, and not to make comparisons based on the mean tensile strengths reported.

When one compares the CV of the previous studies to that of the control sample which produced a CV 0.09, it is argued that only Sloan et. al., [1982:688] who reported a CV of 0.10 and Stade et. al., [1975:527] who reported a CV of 0.11 used a soldering technique that is comparable to the control sample.

A factor that could have affected the range of strengths obtained in other studies is the various gap distances used by the different authors. Table 5.1 illustrates this. Indeed, the study by Stade et. al., [1975:527] was designed to measure the effect of various gap distances on the solderability and strength of gold solder joints. Although they found that the very wide gap of 0.7 mm produced the strongest joints, i.e. 508.23 MPa and a CV of 0.11, it was not recommended for application in practice as the excessive solder shrinkage would cause too much distortion in the bridge and a resultant lack of fit in the mouth. This is the reason why most manufacturers recommend a gap distance of 0.3 mm to 0.2 mm depending on the type of solder being used. Conversely this study produced a mean strength of 561.4 MPa and a CV of 0.09 with a gap distance of 0.2 mm. Therefore it is the opinion of this researcher that gap distance is not a major factor, but that major factors are the soldering method and the choice of solder and flux. Evidence of this can be

seen in the results of previous studies in the discussion on base metal to base metal (section 5.2) where high strengths were achieved with small gap distances.

The reliability of the sample can be accepted, as the first attempt at soldering the sample produced four breaks that were below the lowest strength of the specimens tested in the second sample. The microscopic evaluation showed that these four specimens had been contaminated by cutting swarf during joint surface preparation. This was in contrast to the other specimens in the sample which produced strong break strengths that were not contaminated. When this factor was addressed (section 3.4) and the sample redone, consistently strong break strengths were achieved.

2. Microscopic evaluation

The microscopic evidence showed that all the breaks occurred through the solder. This differed from findings by Staffanou et. al., [1980:33] and Sloan et. al., [1982:688] who reported that half the breaks had occurred through the solder and half through the parent metal. Because they did not report the technical data of the parent metal in their studies, it must be assumed that the tensile strength of the parent metals was similar to the solder and mean strengths they achieved. In this study the two side-on mounted specimens showed good surface apposition and contact at the solder to parent metal interface. This was similar to the findings reported by Marshall et. al., [1984:668], Staffanou et. al., [1980:33] and Sloan et. al., [1982:688].

In addition, the fact that there was no evidence of alloying between the solder and the parent metal, can be regarded as an indication of the correct matching of solder to parent metal alloy combined with a suitable soldering firing cycle for the porcelain furnace. This evidence confirms the predictability and reliability of soldering precious to precious alloys with gold solders.

3. Statistical analysis

The statistical analysis showed that the sample met the requirements of minimum specification and predictability (section 4.23).

Of note here is that at the first attempt to solder the sample the criteria of predictability were not met. It was only when the contamination had been eliminated (section 3.4), that the second sample met the criteria set.

4. General comments

By building in the requirement of a minimum specification to be met by the specimens in the sample and at the same time measuring the predictability of the result, the results and therefore the soldering method can be taken as valid and reliable.

The fact that the mean tensile strength of the first attempt was above the minimum specification but did not meet the requirements of predictability, thus requiring the sample to be redone, shows that the element of personal bias was minimised.

On this basis, the soldering method was considered sound and therefore suitable for addressing subproblems two and three. In addition, the assumption that a 2 mm gap distance is suitable, was validated.

5.2 DISCUSSION - SUBPROBLEM TWO

The hypothesis for subproblem two, the selected alloy types that are soldered without gold plating the joints will not produce the same level of predictability and strength as the control, and will thereby set parameters for comparison, can be accepted on the following bases:

1. The mean tensile strengths produced by the samples were below the mean of the control group, but comparable to the means reported in previous studies.
2. The criteria set for the statistical analysis were met.
3. The critical assumptions were validated.

A discussion on each of these results follows:

1. Tensile strengths

The discussion will look at the results of this study compared to the results obtained in previous studies, and will centre mainly on the reasons why the results are different or similar.

Base metal to base metal, no gold plate

The soldering method produced a mean tensile strength of 381.19 MPa, a SD of 120.49 MPa, and a CV of 0.31 (Table 4.1). Of the eight specimens two tested below the minimum specification of 360 MPa.

The following list details the results of other studies which evaluated base metal to base metal gold solder joints:

Mean (MPa)	SD	CV	Reference
634.90 ± 91.6	0.14		Sobieralski [1987:40]
550.00 ± 46.1	0.08		Kaylakie et. al., [1985:459]
524.02 ± 62.88	0.12		Staffanou et. al., [1980:33]
381.19 ± 120.49	0.31		base metal to base metal no G.P.
365.43 ± 55.16	0.15		Sloan et. al., [1982:688]
282.00 ± 102.04	0.36		Marshall et. al., [1984:668]
275.00 ± 70.6	0.25		Kaylakie et. al., [1985:459]

Because the authors did not report the technical data on the alloys and solders used, a comparison of the CVs in the previous studies is the best form of comparison to determine the reliability and the predictability of the soldering method used. The fact that the CVs reported in the other studies ranged from 0.36 to 0.08, with only Kaylakie et. al., achieving a CV under 0.10, is indicative of the difficulty in achieving consistent results when soldering base metal alloys. Indeed, most of the studies cited report this problem. If the CV values are used to rank all the studies, then this study ranks second to last. The main reason for this is the two specimens that produced tensile strengths below the minimum specification (Table 4.1). No obvious reason can be offered as to their low strengths, as both showed cohesive breaks through the solder with no signs of excessive porosity or flux inclusion.

All the authors stated that they had found the soldering process unpredictable. In order to achieve their results and to obtain sufficient specimens for their sample, they simply soldered more specimens, whenever a sample did not solder or broke while machining on the lathe. In fact, Kaylakie et. al., [1985:457] and Sobieralski [1987:37] x-rayed the specimens in order to detect any defects. It can therefore be argued that, if the two specimens of this study that did not meet minimum specification had been omitted from the sample (Table 4.1), a mean of 443.61 MPa would have been achieved. As it is, the achieved mean of 381.19 MPa is still

above the results obtained by Sloan et. al., [1982:688] and Marshall et. al., [1984:668], and the second result obtained by Kaylakie et. al., [1985:459].

In order to confirm the conclusion reached that soldering gap is not a major factor (section 5.1), the following studies were compared: Sobieralski [1987:40] achieved strengths of 634.9 MPa with a gap distance of 0.33 mm while Sloan et. al., [1982:688] only achieved strengths of 365.43 MPa \pm 55.16 with an increased gap of 0.5 mm. This study produced a mean strength of 381.19 MPa \pm 120.49 with a gap of 2 mm. Therefore, solder joint gap distance does not appear to be a significant factor.

Base metal to semi-precious, no gold plate

The sample produced a mean tensile strength of 461.86 MPa, a SD of 81.88 MPa and a CV of 0.17 (Table 4.1). Of the nine specimens one tested below the minimum specification of 350 MPa. No explanation can be offered for this as the nature of the break was similar to others that produced stronger tensile strengths.

The list below shows the results in relation to other studies:

Mean (MPa)	SD	CV	Reference
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461.86 ± 81.88	0.17		base metal to semi-precious, no G.P.
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448.17 ± 224.08	0.50		Staffanou et. al., [1980:33]
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365.43 ± 68.95	0.18		Sloan et. al., [1982:688]
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These results compare well with those of Staffanou et. al., [1980:33] who produced results of 448.17 MPa, and are better than the results of Sloan et. al., [1982:688] who achieved a mean of 365.43 MPa ± 68.95. However, when one compares the CVs in the previous studies to each other and to the present study, a different conclusion is reached. In this study, a CV of 0.17 compares well with the results of Sloan (CV of 0.18) and both these results are significantly better than the results of Staffanou (CV of 0.50). The same can be said for a comparison of the SDs of the three studies, reporting 81.88 MPa, 68.95 MPa and 224.08 MPa respectively. This large discrepancy of the SDs and CVs between Staffanou's and Sloan's studies and the present study leads one to question the validity and reliability of particularly Staffanou's results. Suffice it to say that these relatively large CV results, when compared to the CV of 0.09 in the control sample, illustrate the unpredictable nature of achieving consistent results when base metal alloys are involved.

Base metal to precious, no gold plate

The sample produced a mean tensile strength of 478.17 MPa, a SD of 58.87 MPa and a CV of 0.12 (Table 4.1). One specimen tested below the minimum specification of 400 MPa. No definite explanation can be offered for this low strength as the nature of the break was similar to that of the breaks that were above the minimum specification. However, the break was predominantly cohesive within the solder and this type of break produced the weaker results in the sample (Table 4.9).

The list below shows a comparison with previous studies:

Mean (MPa)	SD	CV	Reference
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478.17 ± 58.87	0.12		base metal to precious no G.P.
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317.17 ± 68.95	0.21		Sloan et. al., [1982:688]
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310.27 ± 235.80	0.76		Staffanou et. al., [1980:33]
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The results of this study are better than those of Sloan et. al., [1982:688] and Staffanou et. al., [1980:33] who produced results of 317.17 ± 68.95 MPa and 310.27 ± 235.80 MPa respectively. Once again a comparison of the CVs of this study (0.12) and Sloan's (0.21) relative to the CV of Staffanou's study (0.76) leads to the conclusion that the reliability and validity of Staffanou's results are

questionable. Although these authors provided no technical data on the solders they used, it is surmised that, in the case of Sloan et. al., the large difference in strength results between their study and this study is probably due to a difference in strength between the solders used. However, the report by Staffanou et. al., implied that the same solder was used for all the parent metal combinations that were soldered. Since a mean strength of 448.17 MPa was achieved in their base metal to semi-precious combination, one can only conclude that the fault did not lie with the strength of the solder, but with either the choice of flux or the soldering method.

2. Statistical analysis

Incorrect interpretation of statistical results can lead to wrong conclusions. This is especially the case where there is a difference between a pure academic approach and the practical commercial implications of results. An example of this is when the one sample sign test for minimum specification being met by each sample mean, (n = number of specimens tensile tested, Table 4.1), showed the following:

Base metal to base metal, no gold plate. The minimum specification was not met.

Base metal to semi-precious, no gold plate. The minimum specification was met.

Base metal to precious, no gold plate. The minimum specification was met.

In contrast, if one considers at the percentage of successful solder joints that met the minimum specification for each sample based on $n = 10$, (the requirement in terms of commercial application and predictability), the following success rate is evident:

	% Success
control group -	100%
base metal to base metal no gold plating -	60%
base metal to semi-precious no gold plating -	80%
base metal to precious no gold plating -	90%

Although two of the samples met the requirements of minimum specifications, none of the samples produced a success rate of 100%, and therefore statistically the results of subproblem two did not match those of subproblem one, thus validating the hypothesis for subproblem two.

3. Assumptions

The assumption that the manufacturer's recommended gap width of 2 mm between the two surfaces to be soldered is suitable [Degussa AG, 1980: Working Instructions, Ceramic Alloys], was validated. However the assumption that the flux recommended by the alloy manufacturer for base metal alloys will not discolour the porcelain and will facilitate

adequate wetting by the recommended solder was invalidated by the fact that a flux that does discolour porcelain had to be used in order to facilitate adequate wetting of the base metal surfaces. This assumption was not critical to the acceptance of hypothesis four (section 1.2.3.4) at this stage of the study. It only serves to illustrate once again the difficulty in postceramic soldering of base metal alloys.

4. Summary

1. The mean tensile strengths produced by the three alloy groups were lower than that of the control.
2. By comparing the specimens in the sample to the minimum specification and at the same time establishing their similarity to previous studies, the results can be considered valid and reliable.
3. With regard to predictability, it would be incorrect to statistically compare these results with previous studies where only successful solder joints were included in the sample. An example of this is the x-raying of specimens before selecting them for the sample, which implies that the effects that could have influenced the statistics had been eliminated. (Kaylakie et. al., [1985:459] and Sobieralski et. al., [1987:37]). Aside from this fact all the authors cited in this section commented on the difficulty of attaining predictable successful solder joints when base metal alloys were involved.

4. The critical assumptions were validated.

5. The element of personal bias was minimised for this study by the fact that the mean tensile strength of specimens that tested below the minimum specification, as well as unsuccessful solder joints that could not be tensile tested, were included in the sample.

On the above bases, the selected alloy types that were soldered without gold plating the joints did not produce the same level of predictability and strength as the control, and therefore they set parameters for comparison with the results pertaining to subproblem three.

5.3 DISCUSSION - SUBPROBLEM THREE

The hypothesis, the selected alloy types that are soldered after gold plating the joints will produce a level of predictability and strength that are suitable for comparison with the identified parameters, cannot be accepted on the following bases:

1. The critical assumptions were invalidated.
2. The tensile strengths showed no significant improvement.
3. Statistically the results were suitable for comparison which confirmed that there was no significant improvement in the solderability of the gold plated joints.

A discussion on each of these results follows:

1. Assumptions

In the formulation of the study the assumption was made that the flux recommended by the alloy manufacturer for base metal alloys would not discolour the porcelain and would facilitate adequate wetting by the recommended solder.

However, despite gold plating of the base metal specimens prior to soldering, this assumption was invalidated by the fact that a flux that does discolour porcelain had to be used in order to facilitate adequate wetting of the base metal surfaces.

The following are the surmised reasons requiring further investigation as to why the assumption was not valid:

- Gold plating solution

The type of gold plating solution was not suitable for high temperature soldering as it was primarily designed and formulated for jewellery purposes. An analysis of the solution showed that it contained 6% silver [Sparks, 1992].

ASTM B - 488, [1992:237] states that a Type 1 gold, (purity of 99.90%) should be used for high temperature application. Unfortunately this information was only obtained at the conclusion of the study.

◦ Flux

A review of the fluxes used in the electronics industry showed that their composition is similar to the dental flux that ultimately had to be used to facilitate wetting of the gold plated parts. This could indicate that even if a type 1 gold plate is used, it may still require a flux that will discolour the porcelain. Only further study will answer this question.

2. Tensile strengths

The discussion on tensile strengths will mainly centre on comparing the gold plated results to the non-gold plated results. Although there were increases in the mean tensile strengths for two of the samples, namely base metal to base metal with gold plate and base metal to precious with gold plate, these increases in strength were insignificant when compared to the control group strengths. In addition, the means were calculated based on n = number of specimens tested and not on $n = 10$. For this reason the coefficient of variation (CV) is a better indicator for comparison.

Note should be taken that where comparison of strengths to previous studies is made, those studies did not involve gold plated joints. The studies are simply cited for comparative reasons.

Base metal to base metal, with gold plate

The sample produced a mean tensile strength of 462.3 MPa, a SD of 77.76 MPa and a CV of 0.16. Of the nine successful solder joints one tested below the minimum specification. A possible reason for this is the excessive amount of flux inclusion revealed by the light microscope results (Table 4.6, specimen DI1&2).

The following list ranks the results of other studies which evaluated base metal to base metal gold solder joints:

Mean (MPa)	SD	CV	Reference
634.90 ± 91.6	0.14		Sobieralski [1987:40]
550.00 ± 46.1	0.08		Kaylakie et. al., [1985:459]
524.02 ± 62.88	0.12		Staffanou et. al., [1980:33]
462.32 ± 77.76	0.16		base metal to base metal with G.P.
381.19 ± 120.49	0.31		base metal to base metal no G.P.
365.43 ± 55.16	0.15		Sloan et. al., [1982:688]
282.00 ± 102.04	0.36		Marshall et. al., [1984:668]
275.00 ± 70.6	0.25		Kaylakie et. al., [1985:459]

It can be seen that there was no significant increase in strength as a result of gold plating the joints prior to soldering. The mean tensile strength of the gold plated joints was only 81.11 MPa above the mean of the base metal to

base metal, no gold plate result. What is of interest is the 50% improvement of the CV of the gold plated joints from 0.31 to 0.16. This would indicate that the gold plate exercised a positive influence on the solderability of the specimens. However, from a practical viewpoint the improvement is not significant.

Base metal to semi-precious, with gold plate

The sample produced a mean tensile strength of 368.18 MPa, a SD of 91.83 MPa and a CV of 0.24 (Table 4.1). Of the eight specimens four tested below the minimum specification of 350 MPa. This can be directly attributed to the excessive flux inclusion revealed by the light microscope results (Table 4.7).

The following list ranks the results of other studies which evaluated base metal to semi-precious alloy gold solder joints:

Mean (MPa)	SD	CV	Reference
461.86 ± 81.88	0.17		base metal to semi-precious, no G.P.
448.17 ± 224.08	0.50		Staffanou et. al., [1980:33]
368.18 ± 91.83	0.24		base metal to semi-precious, with G.P.
365.43 ± 68.95	0.18		Sloan et. al., [1982:688]

It can be seen that there was a significant decrease in strength of 93.68 MPa as a result of gold plating the joints prior to soldering. In addition, there was a negative increase in the CV from 0.17 to 0.24. This would indicate that overall the gold plate exercised an inhibiting effect on the solderability of the specimens. Despite this the potential is there for strong joints to be produced as the strongest specimen in this sample produced a tensile strength of 511.40 MPa (Table 4.1).

Base metal to precious, with gold plate

The sample produced a mean tensile strength of 509.69 MPa, a SD of 95.45 MPa, and a CV of 0.18 (Table 4.1). There were only seven specimens in the sample because in three of the specimens the solder only flowed $\pm 20\%$ into the gap. No explanation can be offered for this except to say that the gold plating must have been a major factor, especially as all the specimens soldered in the sample, base metal to precious, no gold plate. Of the seven specimens one specimen tested below the minimum specification of 400 MPa. No explanation can be offered as to the cause of this sub-minimum strength.

The following list ranks the results of other studies which evaluated base metal to precious alloy gold solder joints:

Mean (MPa)	SD	CV	Reference
509.69 ± 95.45	0.18		base metal to precious with G.P.
478.17 ± 58.87	0.12		base metal to precious no G.P.
317.17 ± 68.95	0.21		Sloan et. al., [1982:688]
310.27 ± 235.80	0.76		Staffanou et. al., [1980:33]

It can be seen that there was no significant increase in strength, only 31.51 MPa, as a result of gold plating the joints prior to soldering. In contrast, there was a slight negative increase in the CV from 0.12 to 0.18. This would indicate a lowering of the efficiency and predictability of the soldering method.

3. Statistical analysis

As was discussed in section 5.3, Statistical Analysis, one must be careful not to arrive at a wrong conclusion with regard to the practical application of statistical results. In the test for minimum specification being met by each sample mean, the one sample sign test based on n = number of specimens tensile tested, (section 4.23), showed the following:

Base metal to base metal, with gold plate. The minimum specification was met at a 95% confidence level.

Base metal to semi-precious, with gold plate. The minimum specification was not met at a 90% confidence level.

Base metal to precious, with gold plate. The minimum specification was met at a 90% confidence level.

The two positive results could imply that the gold plating had *statistically* significantly improved the solderability.

In contrast, if one looks at the percentage of successful solder joints that met the minimum specification for each sample based on $n = 10$, (the requirement in terms of commercial application and predictability), compared to the success rate shown in subproblem two where the joints were not gold plated, the following success rate is evident:

% Success			
	No G.P.	With G.P.	\pm
control group -	100%		
base metal to base metal	60%	80%	+ 20%
base metal to semi-precious	80%	40%	- 40%
base metal to precious	90%	60%	- 30%

Despite two of the samples meeting the requirements of minimum specifications, none of the samples could produce a success rate of 100%, and two of the samples showed a significant decrease in success rate when compared to their non-gold plated equivalent.

Based on the evidence of the percentage success rate, the results can be accepted as valid and reliable. Had this only been based on the one sample sign test, this would have led to an incorrect conclusion as to the validity of the results.

Statistically the percentage success rate results of subproblem three provided results suitable for comparison in subproblem four thus validating the hypothesis for subproblem three.

4. Summary

1. The mean tensile strengths produced by the three alloy groups were lower than that of the control. While the mean strengths of the gold plated base metal to base metal and base metal to precious showed an increase over those of their non-gold plated counterparts, this increase was not significant.
2. With regard to predictability the problem of excessive flux inclusion and, consequently, the low percentage success rate show that the gold plating did not enhance the predictability of the soldering technique.

3. It would appear that the gold plating had an inhibiting effect on the free flow of the molten solder into the gap. Evidence of this is the 3 joints that failed to flow in the base metal to precious sample, and the excessive amounts of flux inclusions seen in all the samples, a phenomenon which was not present in the samples pertaining to subproblem two. Although the wetting tests showed good wetting by the solder on the gold plated specimens, a slightly larger gap of ± 3 mm or more could be tried. Thus the assumption that the manufacturer's recommended gap width of 2 mm between the two surfaces to be soldered is suitable [Degussa AG, 1980: Working Instructions, Ceramic Alloys], is questionable and will require further study.
4. The assumption that the flux recommended by the alloy manufacturer for base metal alloys will not discolour the porcelain and will facilitate adequate wetting by the recommended solder was invalidated by the fact that a flux that did discolour porcelain had to be used in order to facilitate adequate wetting of the base metal surfaces.
5. The fact that the mean tensile strength of specimens that tested below the minimum specification as well as results pertaining to unsuccessful solder joints were included in the sample, shows that the element of personal bias was minimised.

Statistically, the hypothesis, the selected alloy types that are soldered after gold plating the joints will produce levels of predictability and strength that are suitable for comparison with the identified parameters, can be accepted.

However, from a practical perspective, both the assumptions and the predictability of the success of the solder joints, were questioned. Based on this, the hypothesis, the selected alloy types that are soldered after gold plating the joints will produce levels of predictability and strength that are suitable for comparison with the identified parameters, cannot be accepted.

5.4 DISCUSSION OF LIGHT MICROSCOPE AND SEM RESULTS

5.4.1 LIGHT MICROSCOPE RESULTS

The main purpose of this study was to establish whether gold plating would enhance the predictability and strength of the solder joints. Just tensile testing the joints would provide an answer to this, but would not reveal an understanding of the reasons for the nature of the breaks. Such knowledge would enable one to make observations and deductions as to why the joints performed as they did. This study was conducted through the eyes of a commercial dental technician and not from the point of view of a metallurgist. With this in mind, the following observations are made and possible answers to the phenomena encountered in the study are offered:

1. Alloying between the gold solder and the semi-precious alloy

The microscopic examination revealed that there was no sign of an interface between the solder and the semi-precious alloy in the samples base metal to semi-precious, no gold plate and base metal to semi-precious, with gold plate. Instead there was evidence of alloying between the solder and semi-precious alloy (Table 4.9). According to Phillips [1991:532], the cause of alloying could be twofold: either the flow point of the solder could be too close to or above the solidus of the parent metal, or the temperature could have remained high for too long. The cause in this study was probably a combination of both these factors as the melting range for the semi-precious alloy Realor was 860-1035°C, and the soldering temperature was 800°C (a difference of only 60°C) which was held for 3 minutes. In this case a lower melting point solder should have been used.

Of particular interest was the alloying by diffusion of the base metal alloy into the gold solder and solder into the base metal observed in specimen BJ1 (Table 4.4). Although Phillips [1991:533] states that the cause for a similar phenomenon in his study was probably a combination of a too high temperature being held for too long, the example that he refers to was for a high temperature pre-ceramic solder being used to solder base metal alloys. In this study, the solder was for postceramic soldering and the difference in temperature between the solder and the base metal alloy melting point was 545°C. In the case that Phillips refers to, the difference in temperature between the high temperature

solder and the base metal alloy would probably have been 230°C. The point of interest here is that one would not expect to encounter this phenomenon with such a large temperature difference.

2. Relationship between joint strength, increased flux inclusion and gold plating

All the gold plated samples were characterised by significant amounts of excess flux inclusion, which was not evidenced in the non-gold plated samples (Table 4.9). Therefore, for this study it can be concluded that the gold plating was a significant factor in this phenomenon. Despite this, many gold plated specimens achieved stronger breaks than their non-gold plated counterparts. Where there was no flux inclusion, the strengths produced in the gold plated specimens were higher than those in their non-gold plated counterparts, but, with the exception of one base metal to precious specimen, not as high as the mean tensile strength of the control. Flux inclusion appeared to be the main cause for the lowering of strength. If there had been no flux inclusion then maybe breaks would have been stronger still. If one poses the question, "Why did some joints exhibit flux inclusion and others not?" - especially as all the specimens were subjected to the same soldering firing cycle - then a possible answer could be that the problem is directly related to the amount of flux that was placed into the gap. This problem will require further study.

3. Relationship between joint strength, increased porosity and gold plating

The microscopic examination revealed that there was an increase in the incidence of porosity in the gold plated specimens which contributed, along with the increased flux inclusions, to a weakening of the joints (Table 4.9). The reason for this is unexplained as some of the gold plated specimens were porosity free, despite having been subjected to the same soldering method. This problem will require further study.

4. Relationship between nature of break and joint strength

The stronger joints showed fewer flux inclusions or porosities and fractured adhesively at the base metal - solder interface, leaving both the solder and parent metal surfaces intact. The weaker joints showed combined adhesive and cohesive fractures and often had defects, such as porosity or flux inclusions, within the cohesive fractures.

5.4.2 DISCUSSION OF SEM RESULTS

There was a high incidence of a grey substance appearing on the surfaces of the joints at the base metal interface. This grey substance was initially thought to be an oxide. This deduction was based on its colour and the fact that it was reported as an oxide layer in previous studies [Kaylakie et. al., 1985:460;

Staffanou et. al., 1980:33, and Marshall et. al., 1984:671] However, logic suggested that if it had been an oxide, then the solder would not have wet the surface [Phillips, 1991:530].

The SEM results in section 4.22 confirmed that the grey substance was in fact not an oxide. However, no explanation can be offered as to what it was. This phenomenon will require further study in order to establish what it is, how it is formed, and how it influences joint strengths.

5.5 DISCUSSION - SUBPROBLEM FOUR

The hypothesis, It will be possible to integrate the results and show that gold plating the joint surfaces prior to investment for soldering will produce predictable strong solder joints, cannot be accepted on the following bases:

1. Assumptions

The assumption that the flux recommended by the alloy manufacturer for base metal alloys will not discolour the porcelain and will facilitate adequate wetting by the recommended solder was invalidated by the fact that a flux that does discolour porcelain had to be used in order to facilitate adequate wetting of the gold plated base metal surfaces.

2. Statistical analysis

◦ Predictability

With regard to predicability, the relationship analysis using χ^2 statistic for gold plating versus no gold plating showed that at a 95% level of confidence the success of the solder joint did not depend on whether or not gold plating was used. It can therefore be concluded that the gold plating did not enhance the predictability of the soldering method.

In addition, the relationship analysis using χ^2 statistic for the control group versus all base metal solder joints showed that at a 95% level of confidence the success of the solder joint did depend on whether or not the solder joint was precious to precious and not a solder joint where base metal alloys were involved. The conclusion here is that precious to precious solder joints are strong and predictable, whereas joints involving base metal, whether or not they are gold plated, are not predictable.

The percentage of successful solder joints that met the minimum specification for each sample showed that none of the samples could produce the 100% success rate achieved by the control group. The commercial significance of this is that when bridges that will require postceramic soldering are to be made, it would be unwise to choose a base metal alloy.

◦ Tensile strengths

When the coefficient of variation is used as a method of comparison, none of the samples, whether gold plated or not, could produce consistent strength results to match the control group. Once again, the commercial significance of this is that when bridges that will require postceramic soldering are to be made, it would be unwise to choose a base metal alloy.

General comments

The fact that the hypothesis has been rejected shows that the methodology in the study has been sound. Had the deductions been purely based on increased tensile test results by the two gold plated samples, as is predominantly the case in previous studies where extra specimens were fabricated to achieve sample size, an incorrect acceptance of the hypothesis could have resulted. By including the measurement of predictability this study has avoided bias and therefore the results can be accepted as valid and reliable.

CHAPTER SIX

CONCLUSIONS, RECOMMENDATIONS AND PRACTICAL SIGNIFICANCE

6.1 CONCLUSIONS

1. Within the budget constraints of this project, and when using the type of gold plating solution and method described (section 3.2.6), the gold plating of metal ceramic base metal alloys prior to investing for postceramic soldering, did not enhance the strength, solderability and predictability of the solder joints to an extent that a flux that does not discolour the porcelain can be used.
2. SEM analysis showed that a grey substance commonly found on all the base metal specimens was not oxide.
3. The predictability of solder joints involving base metal alloys is questionable. This conclusion supports the findings of previous studies.
4. The role of a suitable flux is critical to the successful wetting of the base metal alloy by the solder.
5. Achieving contamination free surfaces in the preparation of the solder joint prior to the commencement of the soldering process is critical to the successful attainment of maximum strength in the solder joint.

6. Until further studies have been made on the effects of plating dental solder joints prior to postceramic soldering, conventional methods for soldering these joints should be used.

6.2 RECOMMENDATIONS

1. On hind sight, insufficient attention was given to the importance of the type of gold plating solution used prior to the study commencing. This study should be redone in the light of this information. As this was a study combining the technologies used by the production electronics industry, jewellery manufacturing industry and the dental industry, further study is needed in order to combine these technologies and caution will have to be exercised in the way they are utilised for a different application.
2. Extensive SEM analysis should be carried out on future base metal solder joints to establish the exact nature of the substances found on the break surfaces.
3. The relationship between the amount and thickness of flux placed in the gap to the amount of flux inclusion produced in the joint should be investigated. This should be related to the soldering firing cycle.

4. The effects that different abrasives used to prepare solder joint surfaces have on the strengths of the joints should be investigated.

6.3 PRACTICAL SIGNIFICANCE

For the commercial dental technician this study has highlighted two important points:

1. The results obtained by postceramic soldering of base metal alloys are not predictable. This is contrary to a perception that could be gained from incorrect interpretation of previously published reports - i.e. that such results are predictable.
2. It is essential for the technician to carry out a wetting test prior to attempting to solder any joints, in order to establish or to confirm the right choice of flux and solder for the brand of parent metal alloy he is using.

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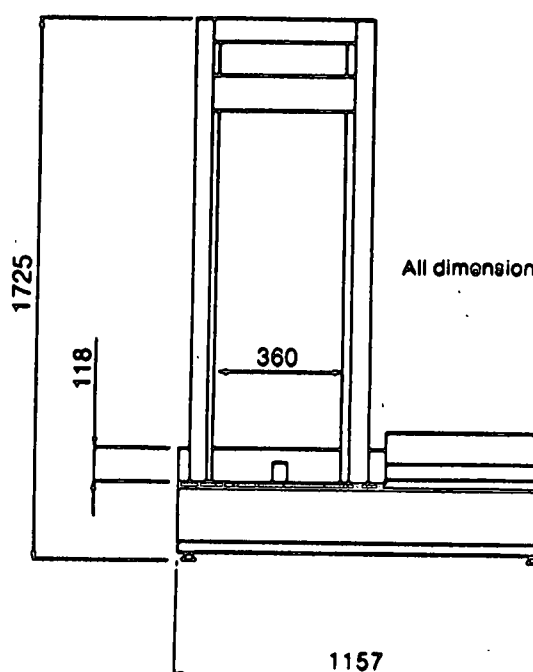
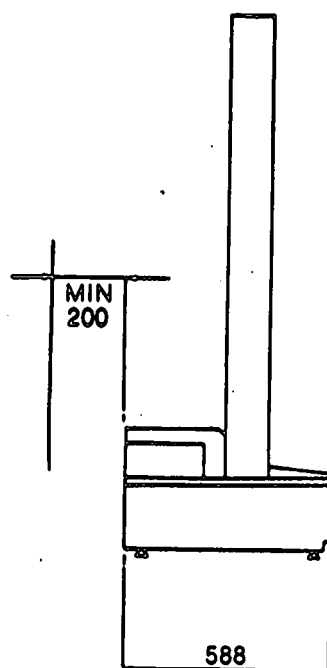
SPECIFICATIONS FOR INSTRON TESTING MACHINE MODEL 4302

SPECIFICATION

MODEL 4301

MODEL 4302

Load capacity	5kN Tension or Compression	10kN Tension or Compression
Load weighing accuracy	$\pm 1\%$ of reading down to $\frac{1}{10}$ of load cell capacity. Meets or exceeds the following international standards: BS.1610 Grade A DIN 51221 Class 1 AFNOR NF A03-501 Class 1 ASTM E4	$\pm 1\%$ of reading down to $\frac{1}{10}$ of load cell capacity. Meets or exceeds the following international standards: BS. 1610 Grade A DIN 51221 Class 1 AFNOR NF A03-501 Class 1 ASTM E4
Position measuring accuracy	± 0.1 mm	± 0.1 mm
Strain measuring accuracy (depending on the extensometer gauge length and strain range)	BS.3846 Grades C and D ASTM E83, B-2, C and D	BS.3846 Grades C and D ASTM E83, B-2, C and D
Crosshead speed range	0.5 - 500 mm/min	0.5 - 500 mm/min
Max load/speed capability	5kN at 500 mm/min	10kN at 250 mm/min 5kN at 500 mm/min
Return speed	1000 mm/min	500 mm/min
Crosshead travel (excluding grips and fixtures)	970 mm	970 mm
Maximum vertical daylight	1070 mm	1070 mm
Frame weight	145kg	145kg
Power requirements		
Voltage	100/120/220/240V $\pm 10\%$ single phase	100/120/220/240V $\pm 10\%$ single phase
Frequency	50/60Hz	50/60Hz
Power	1440VA max.	1440VA max.

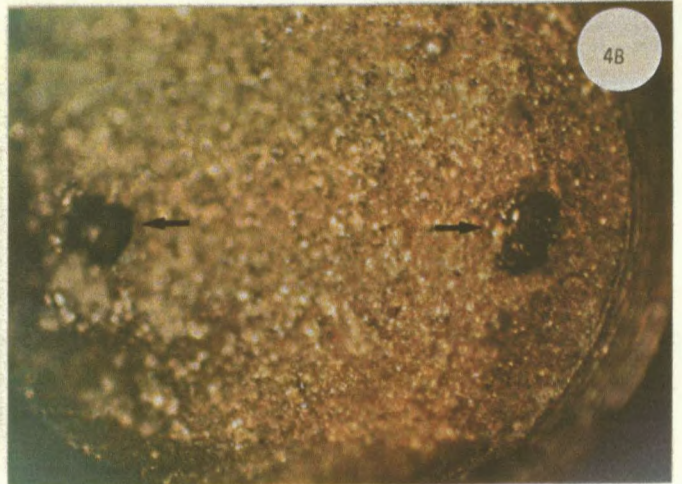
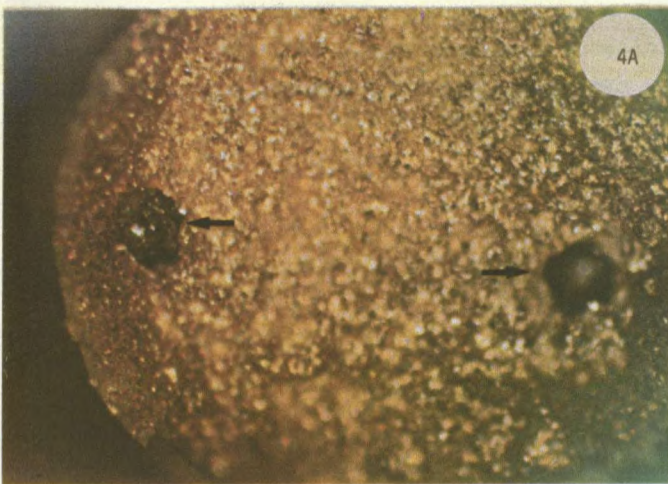
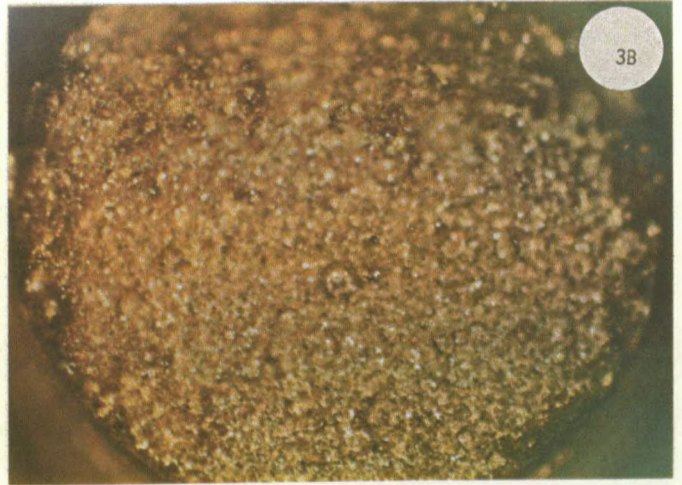
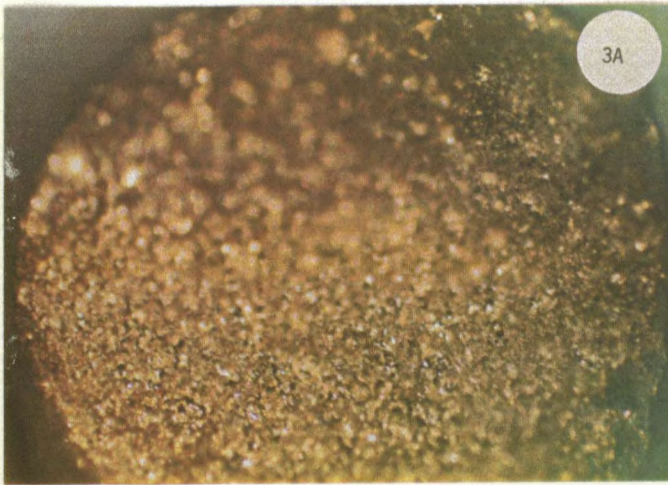
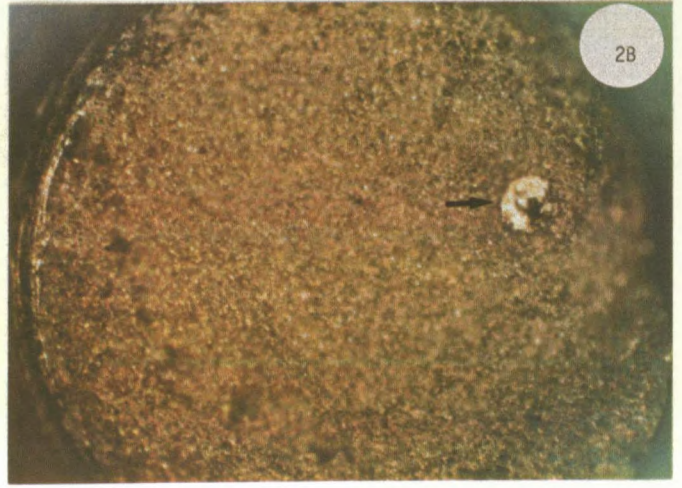
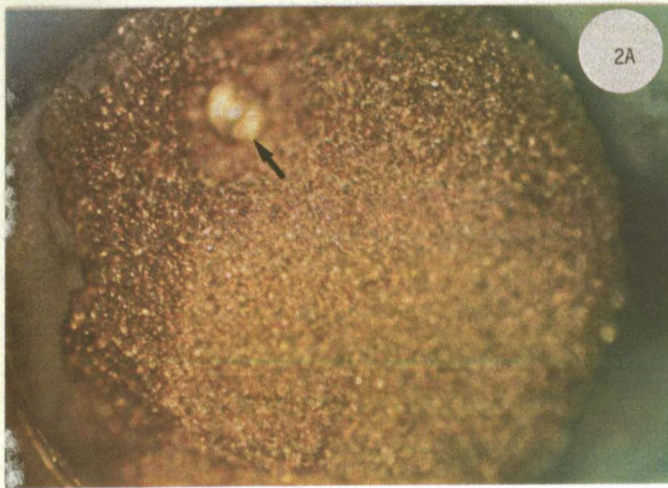
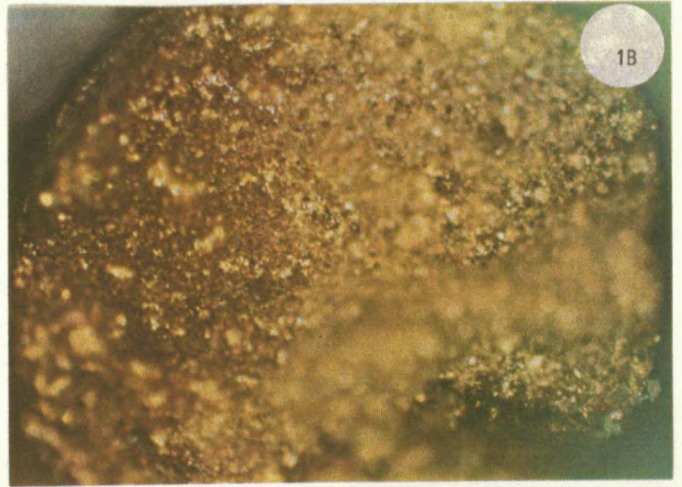
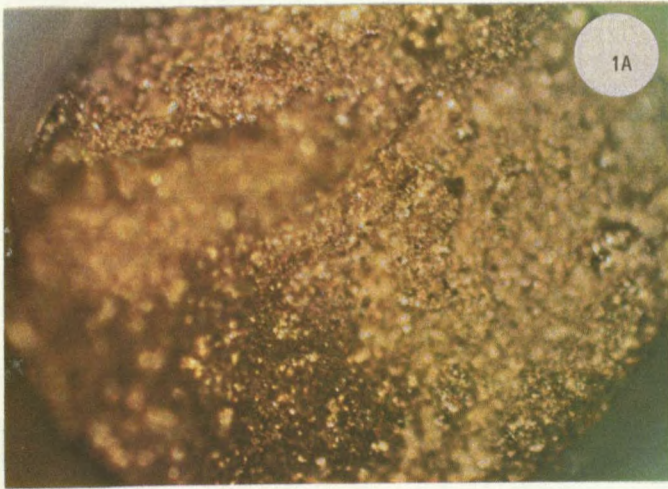


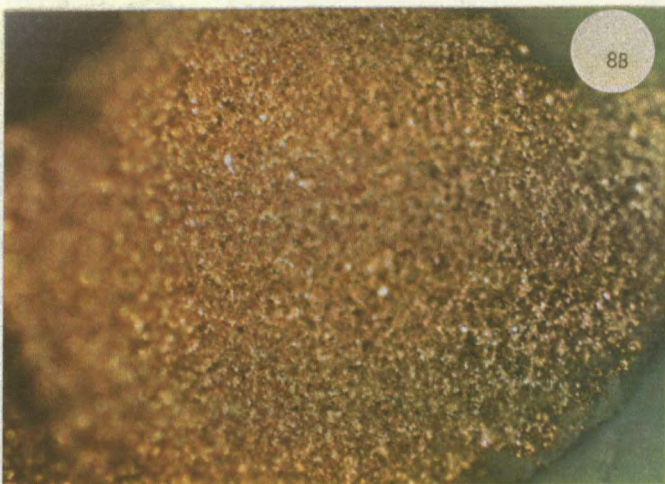
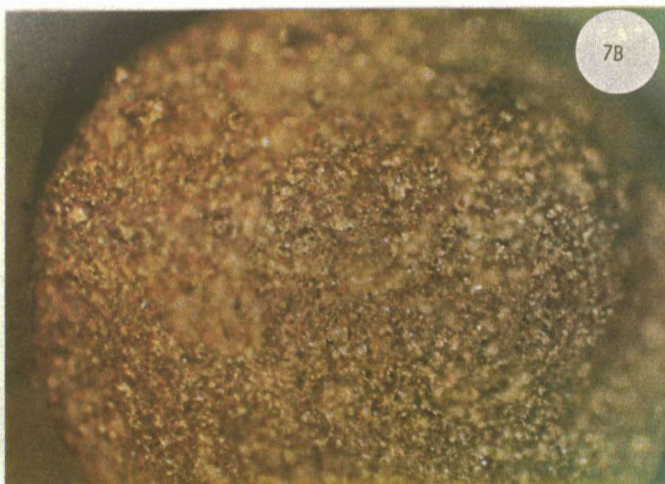
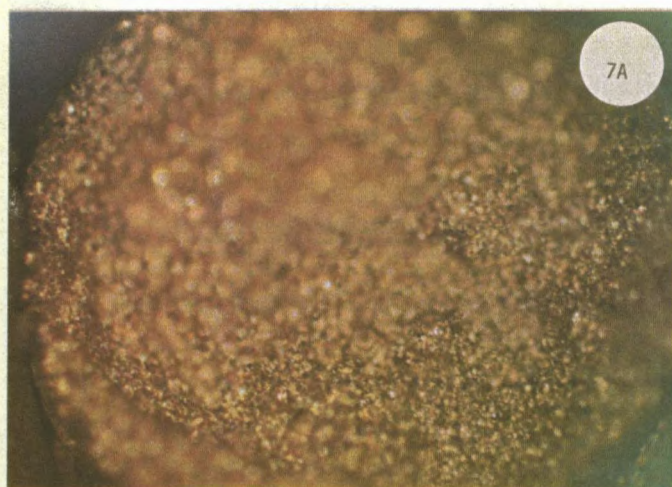
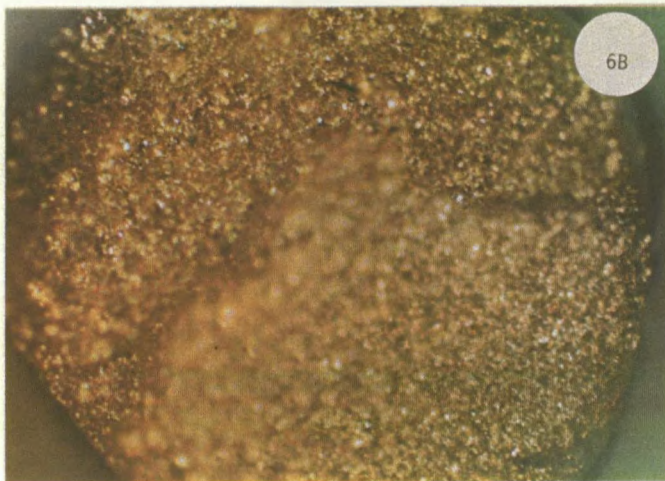
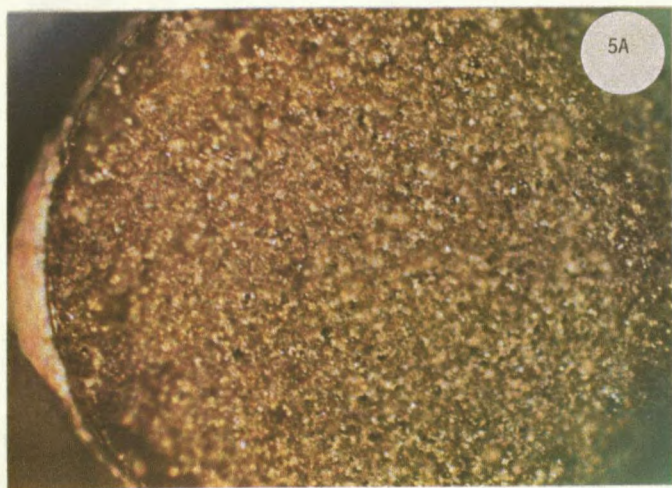
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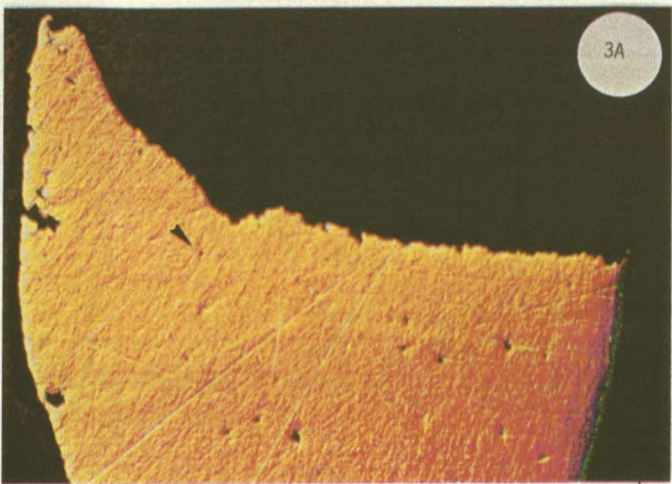
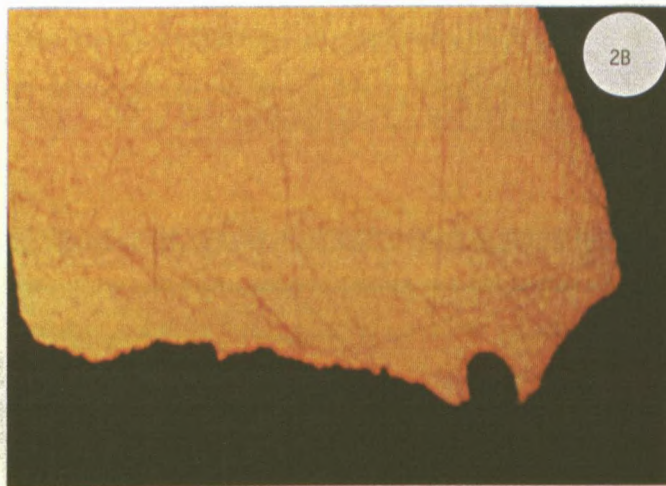
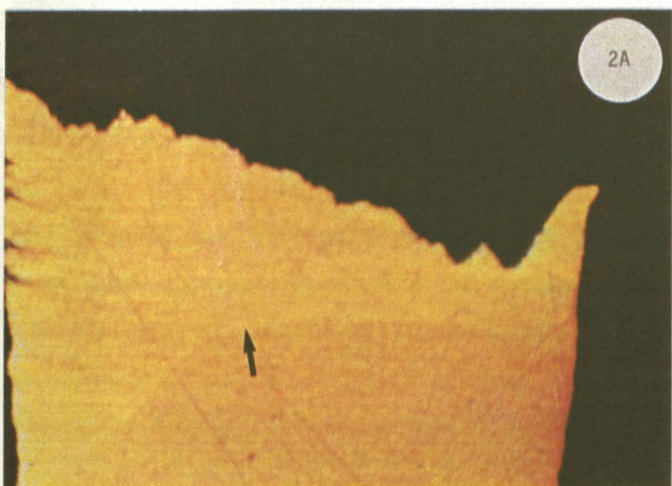
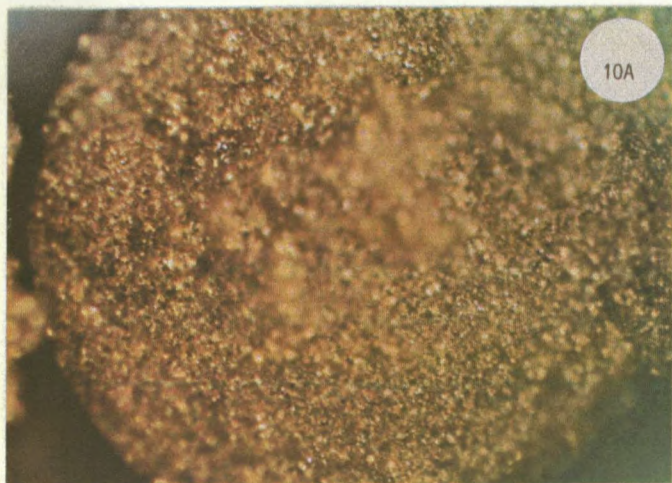
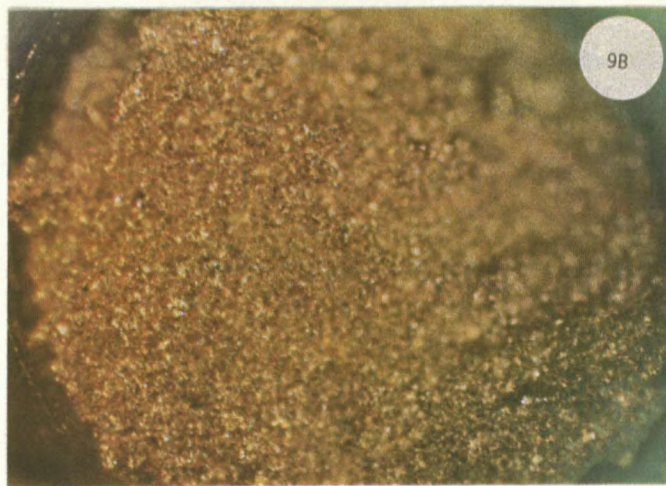
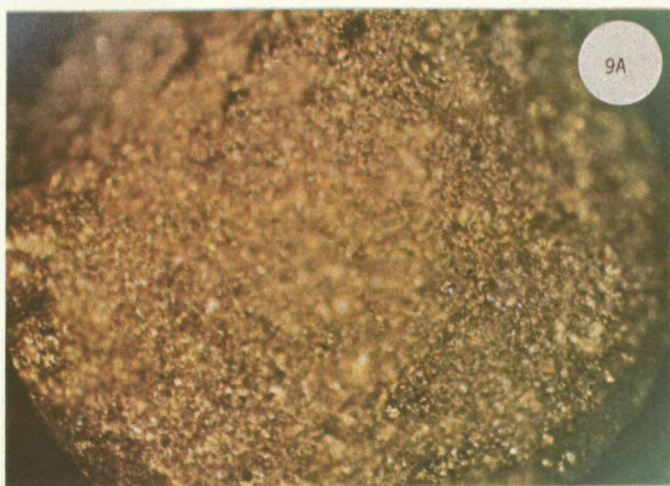
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ANNEXURE B

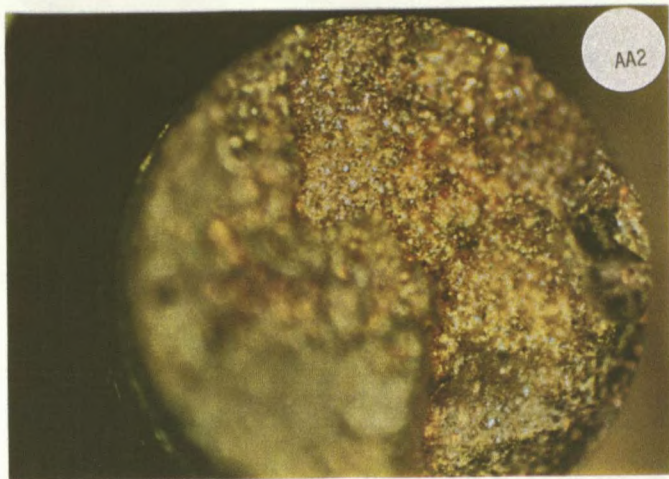
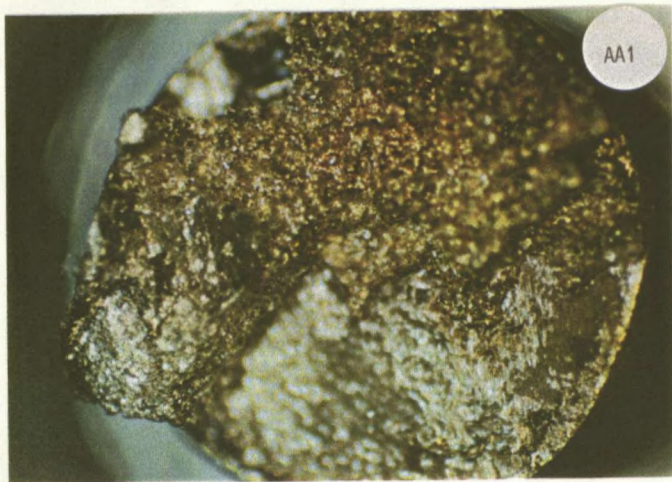
PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM ONE - CONTROL

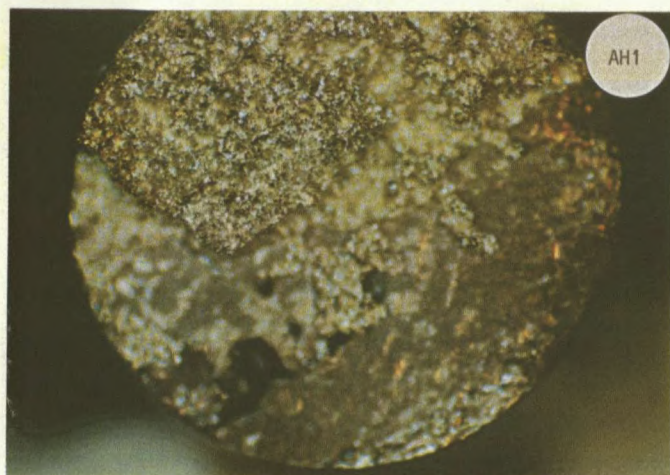
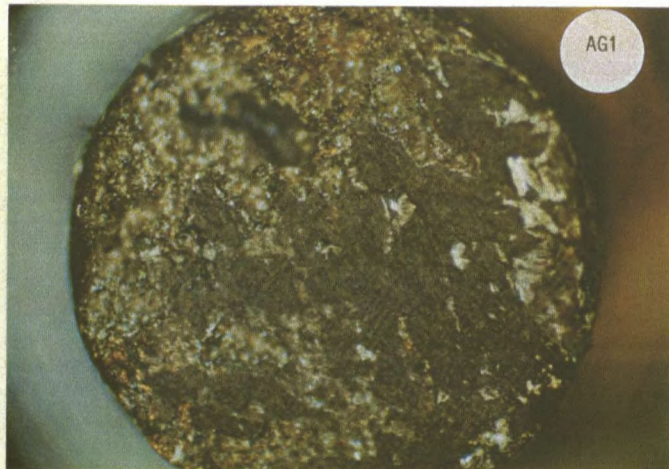
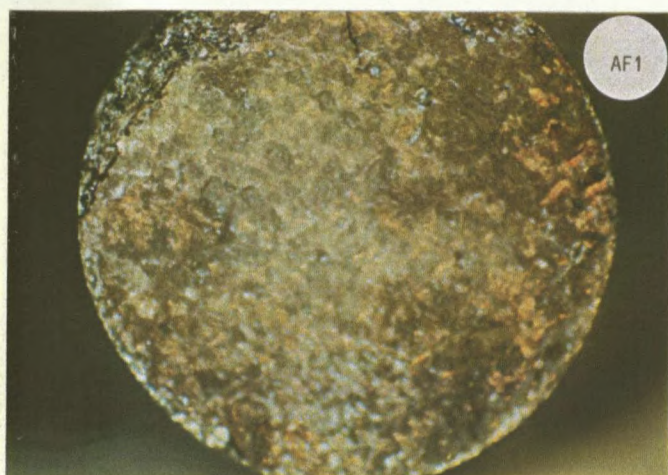
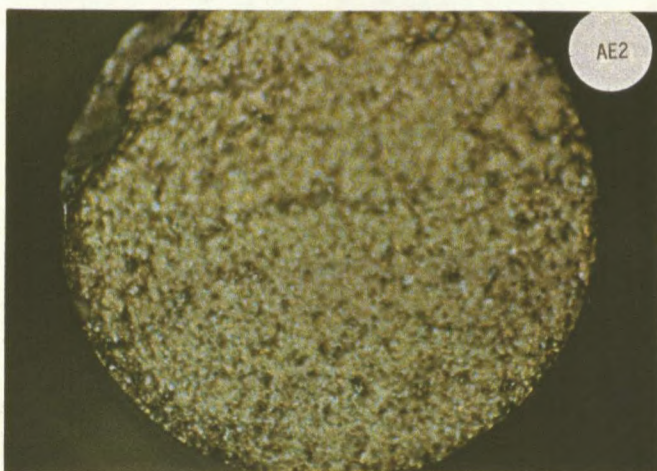
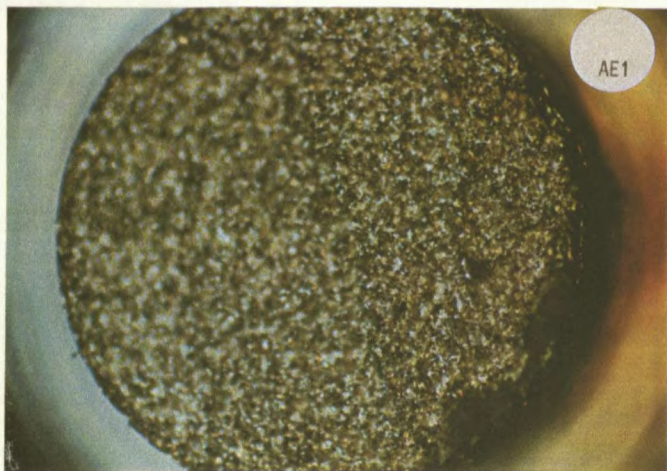


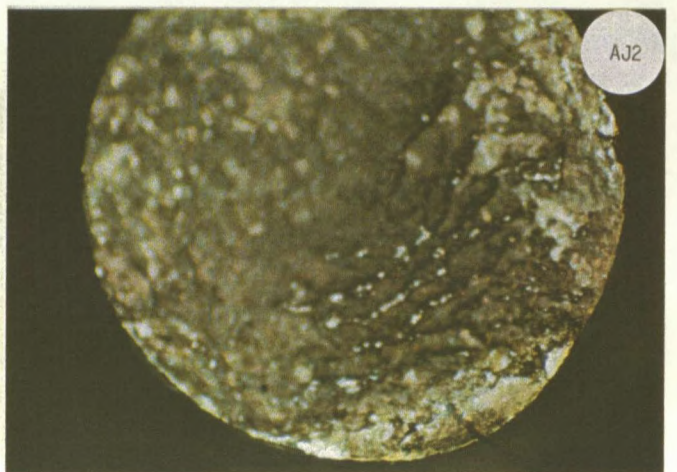
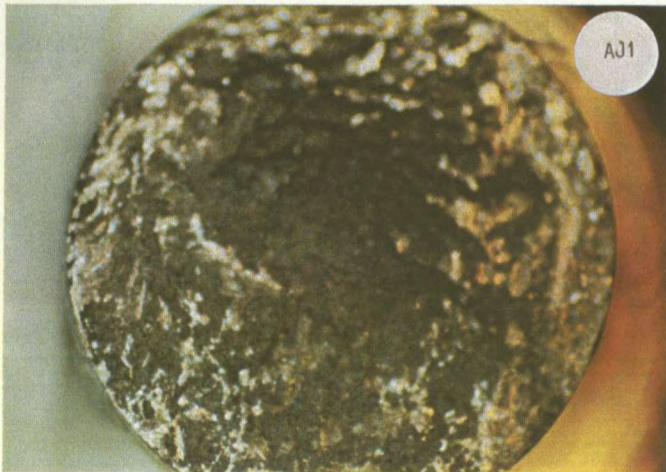
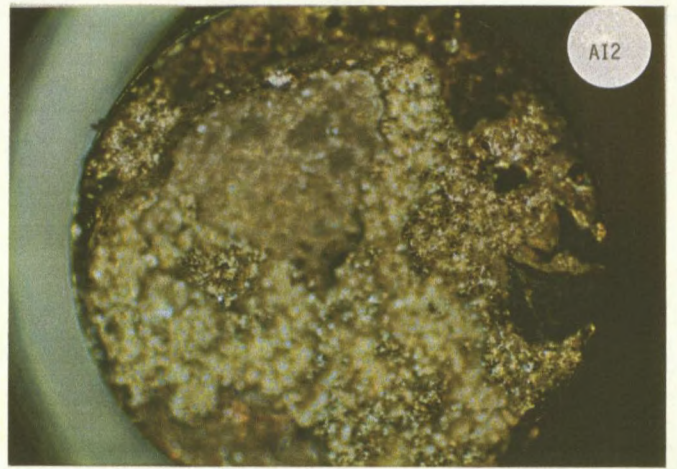




PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM TWO - BASE METAL TO BASE METAL,
NO GOLD PLATE

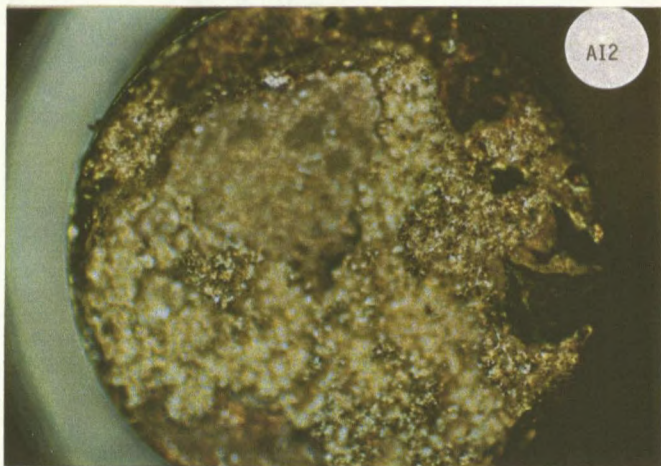








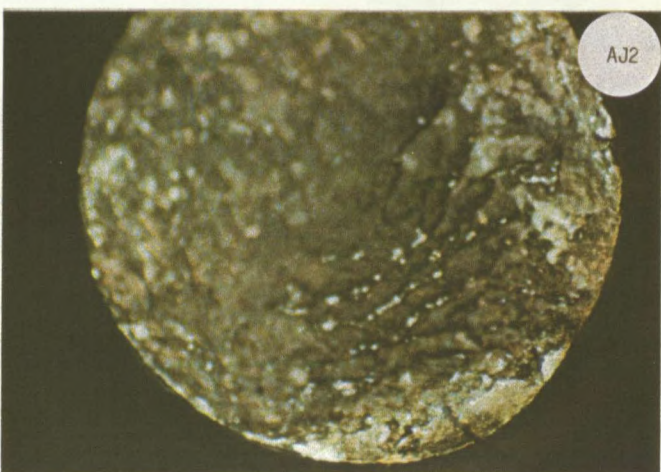
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AI2



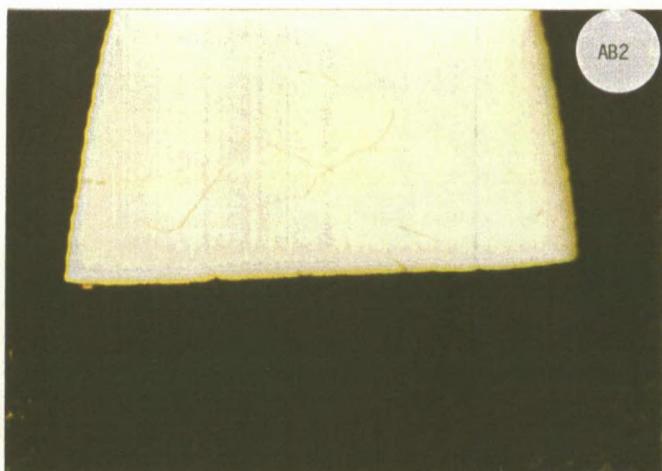
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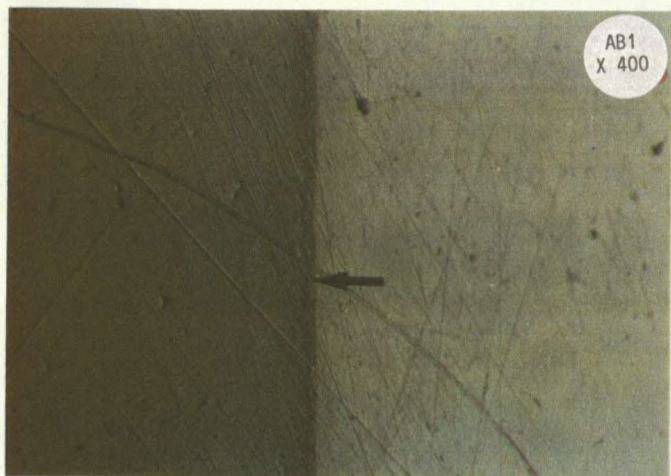
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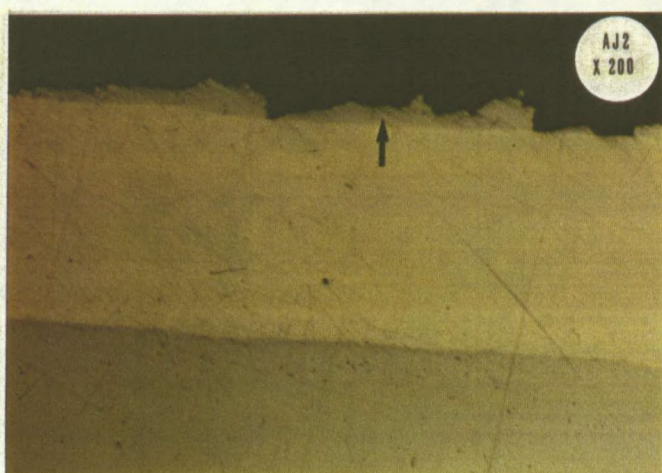
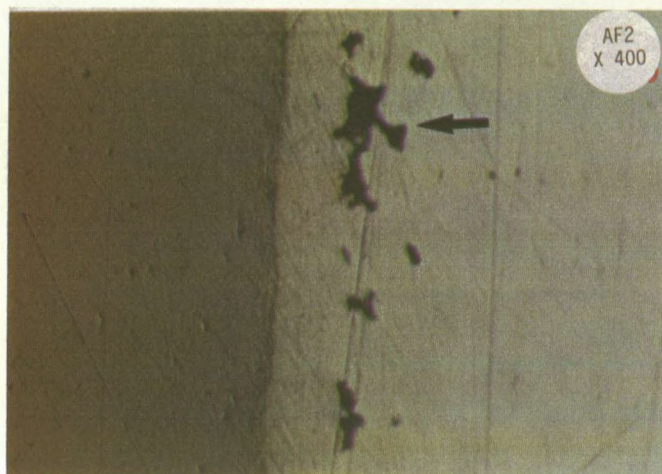
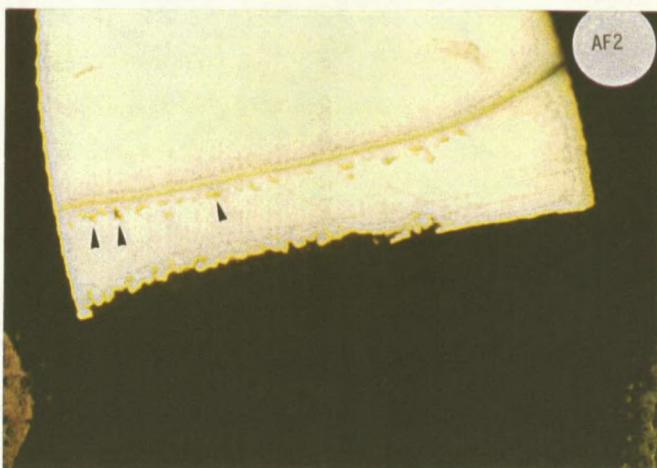
AB1



AB2

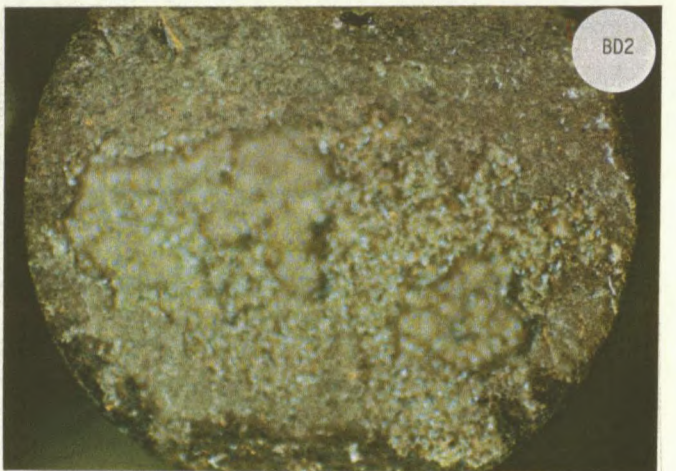
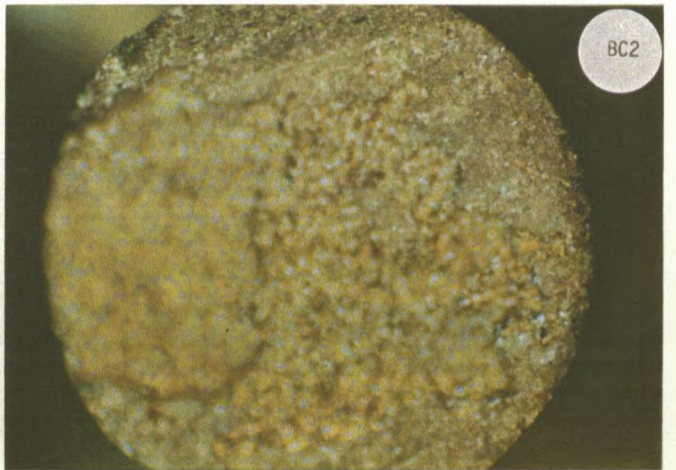
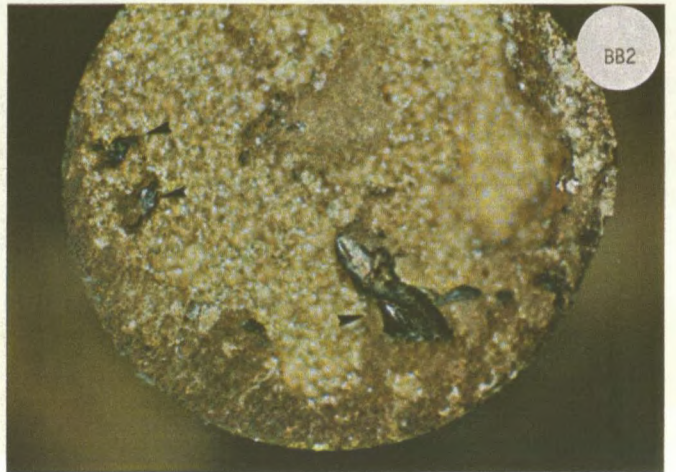
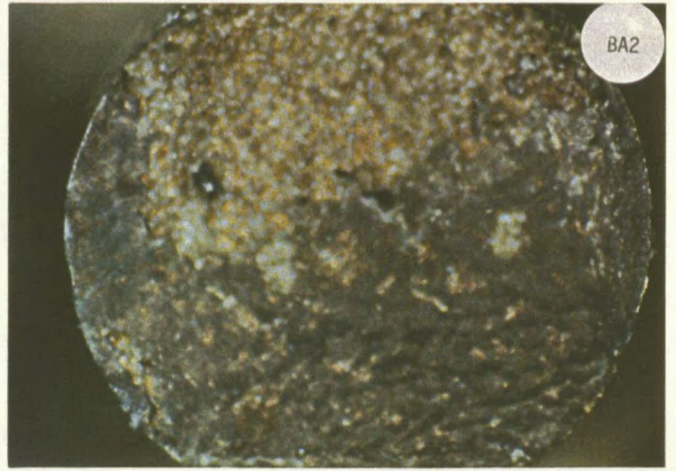
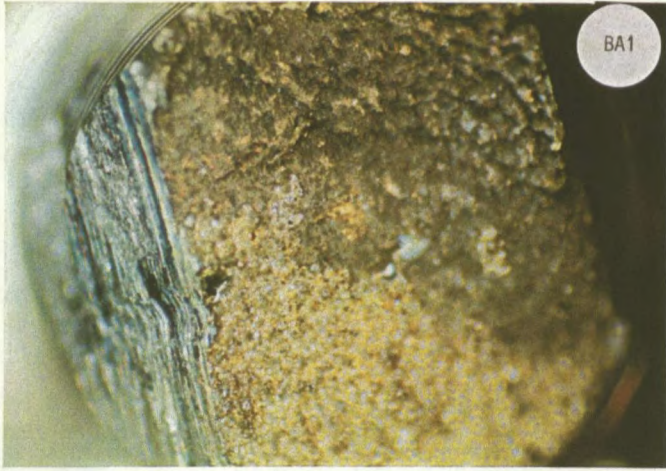


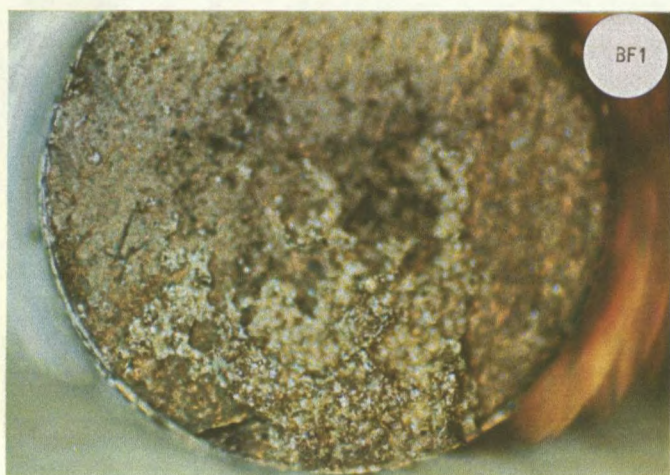
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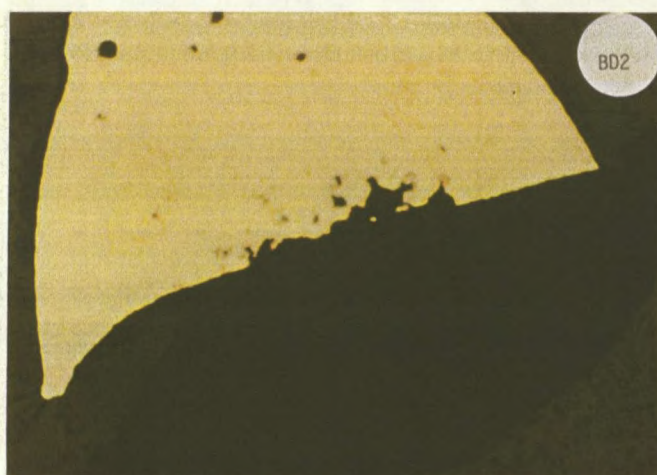
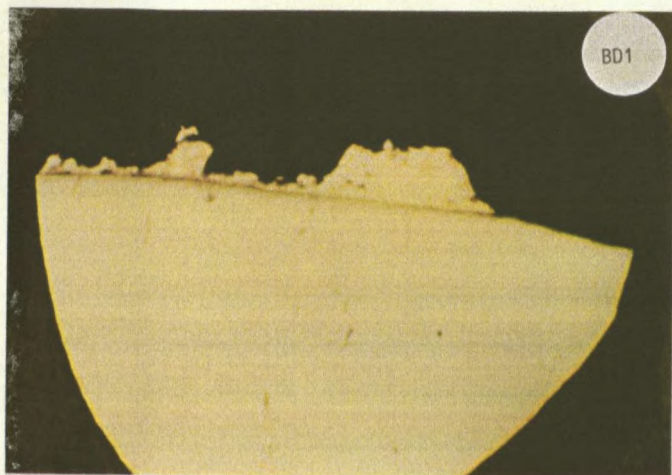
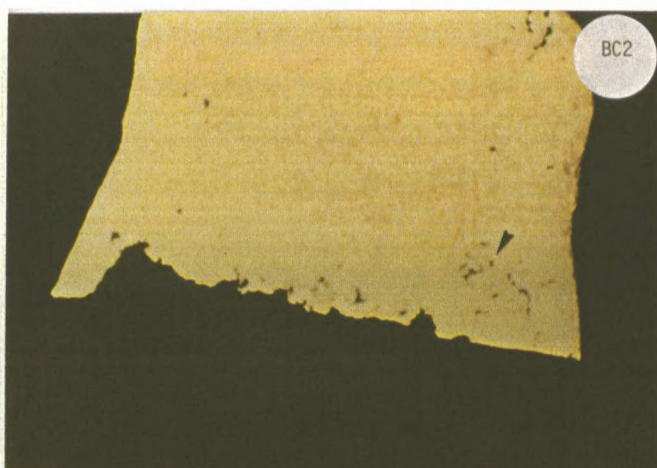
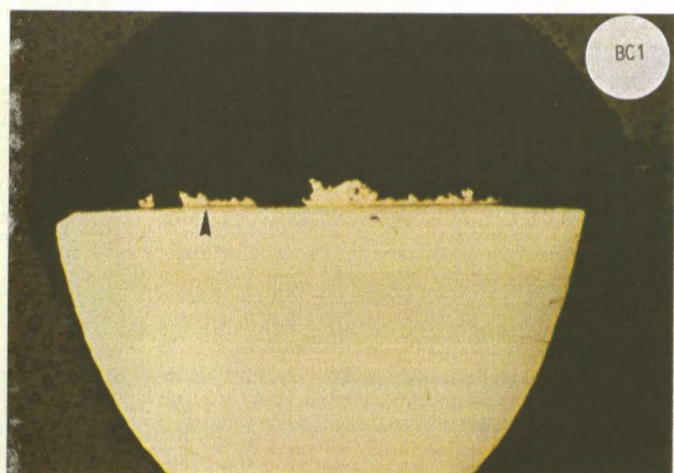
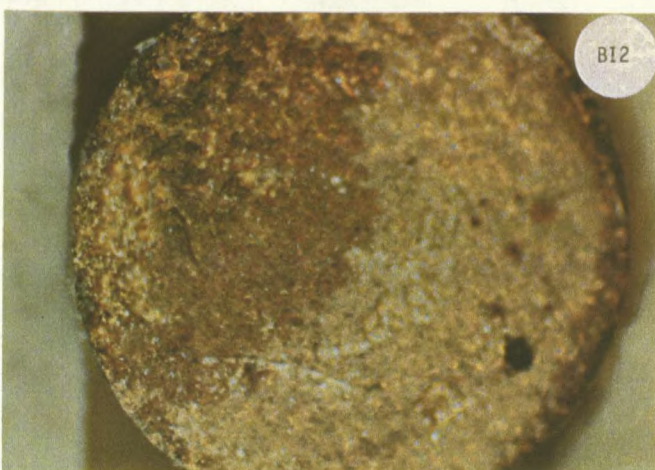


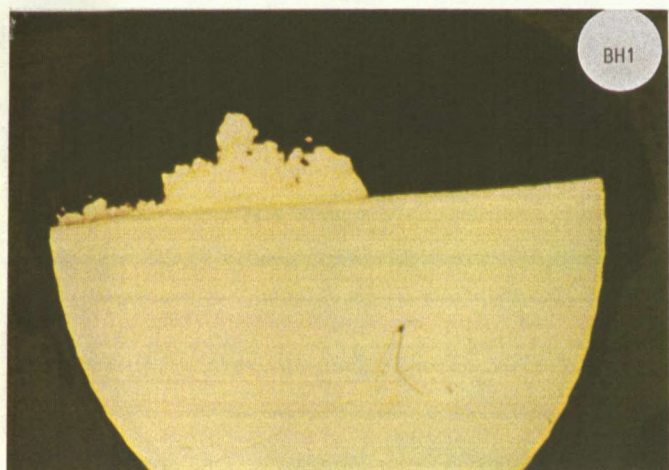
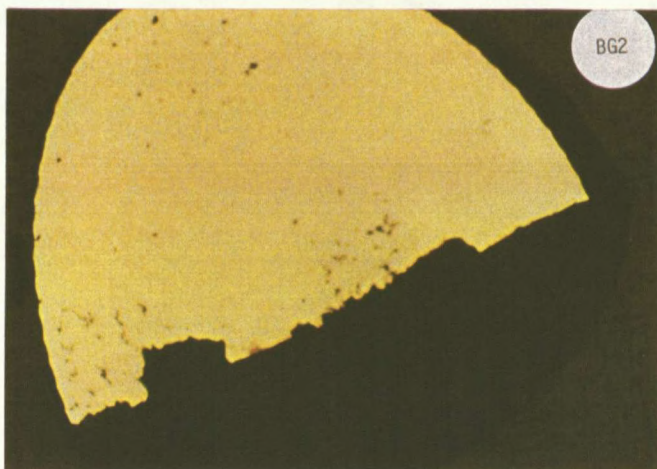
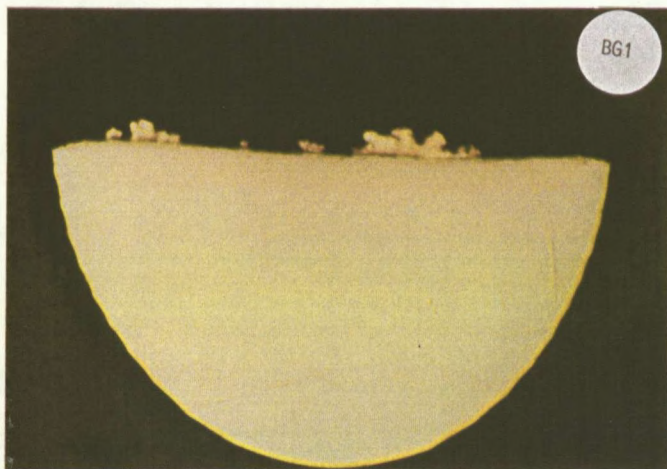
ANNEXURE D

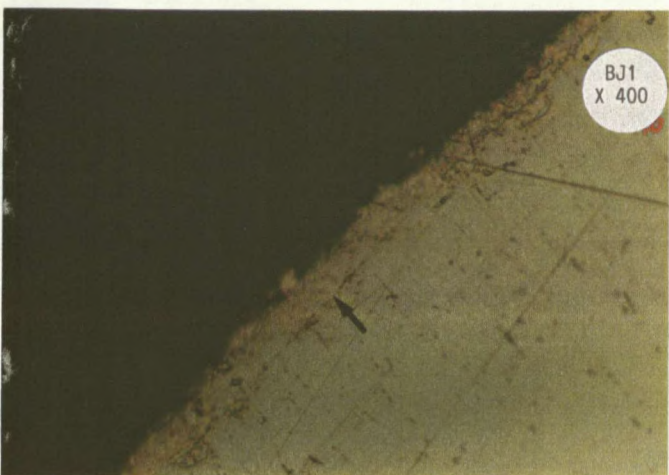
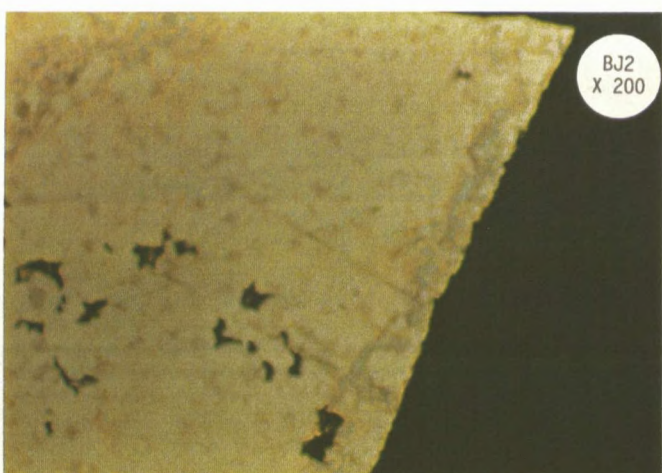
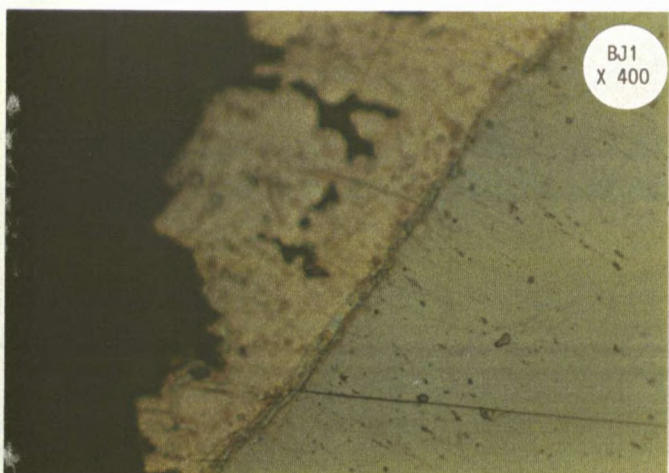
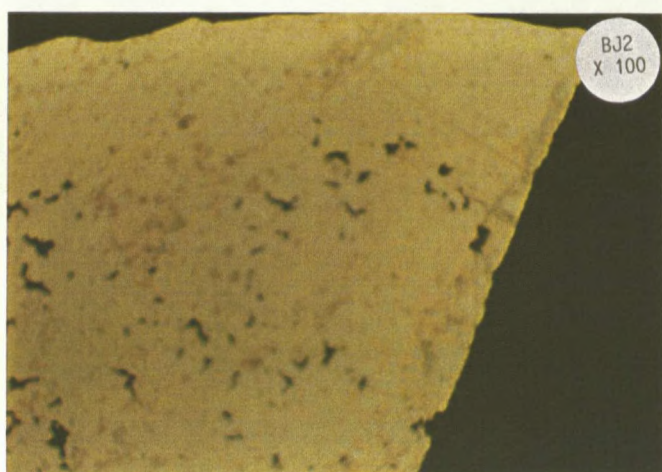
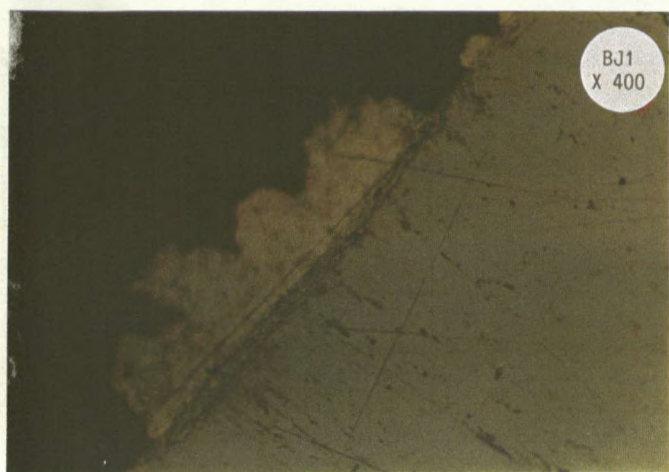
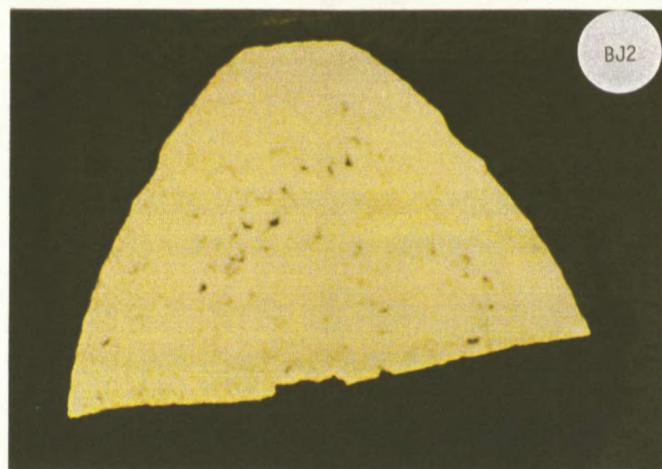
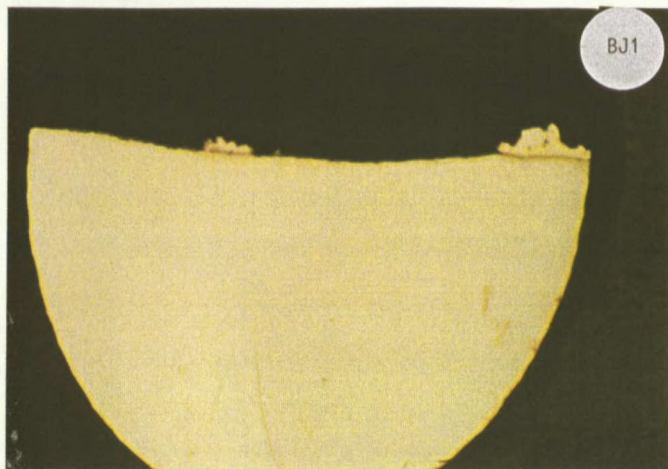
PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM TWO - BASE METAL TO SEMI-PRECIOUS,
NO GOLD PLATE





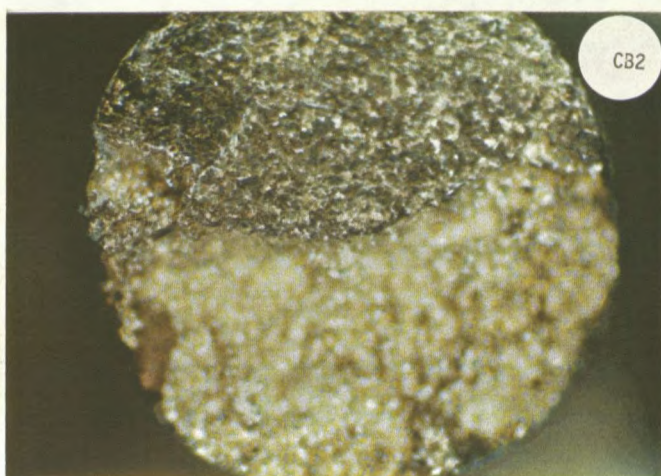
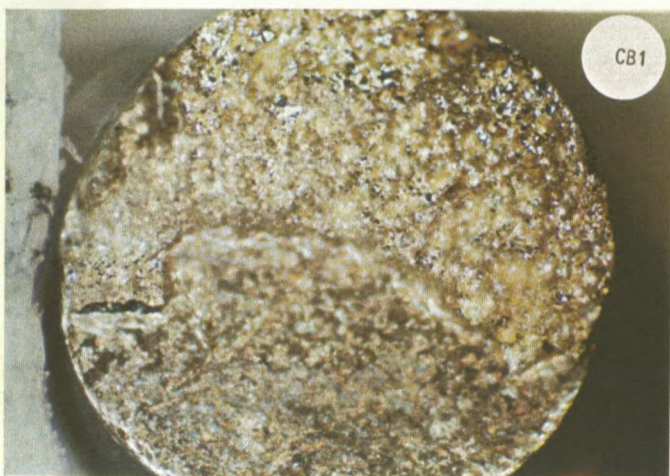


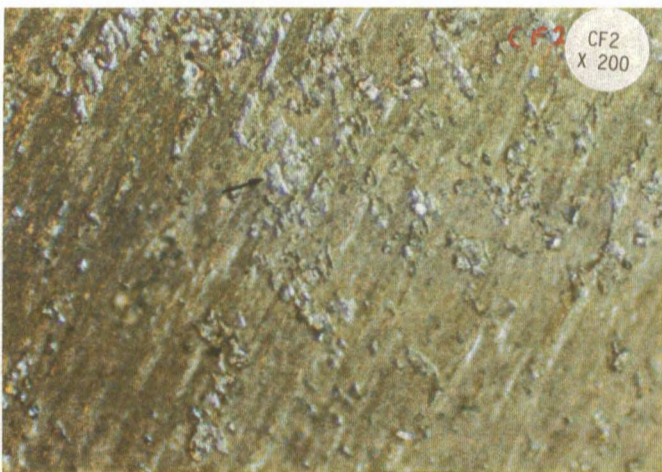
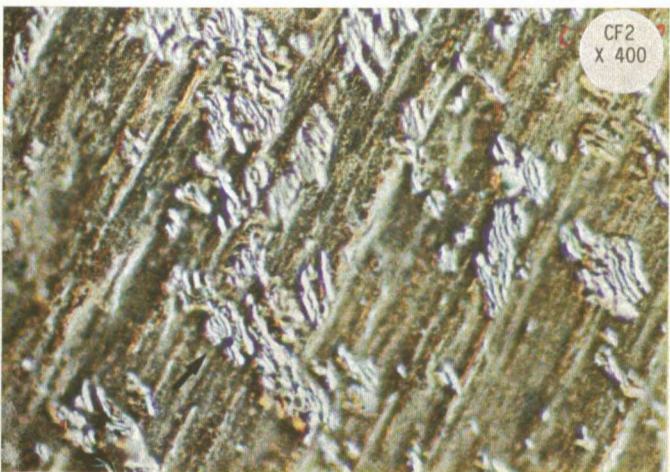
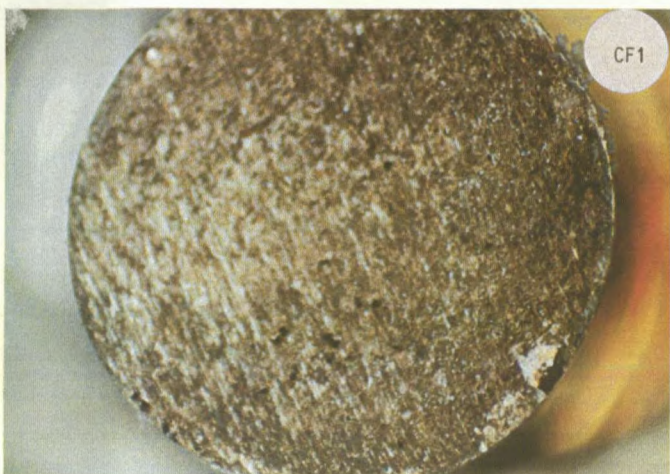
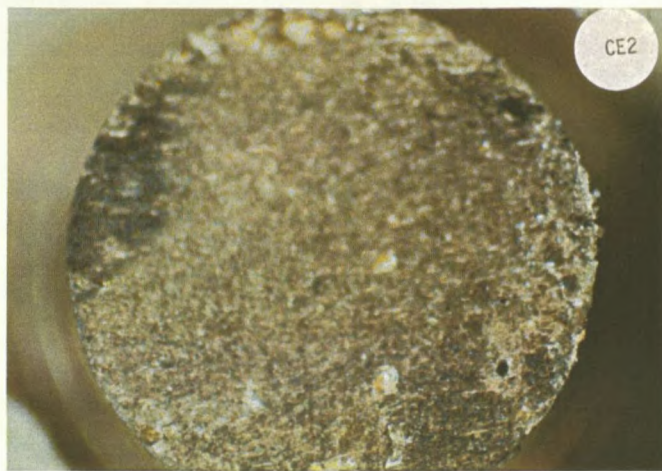


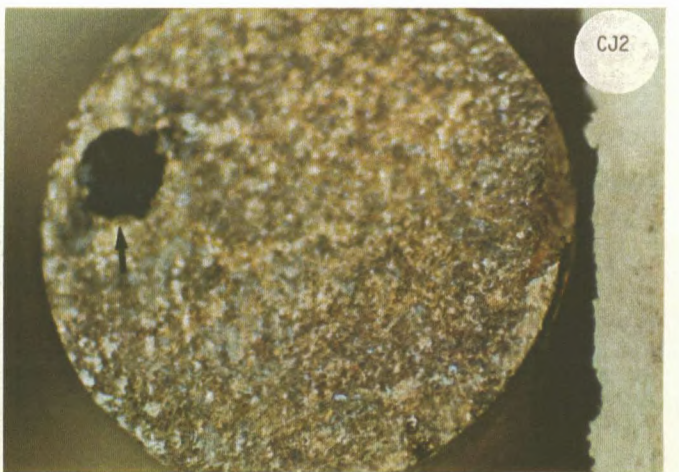
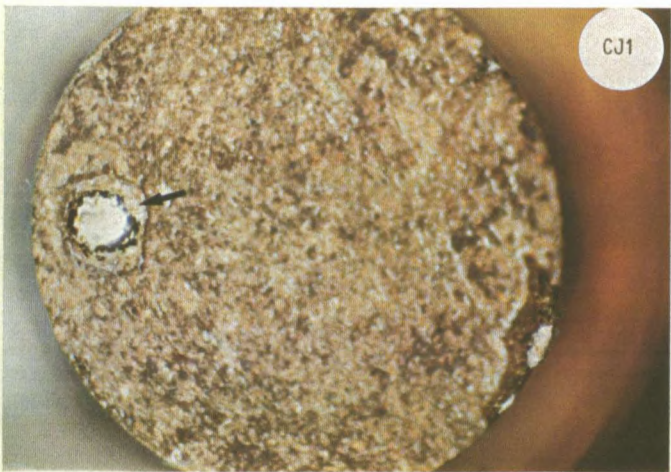
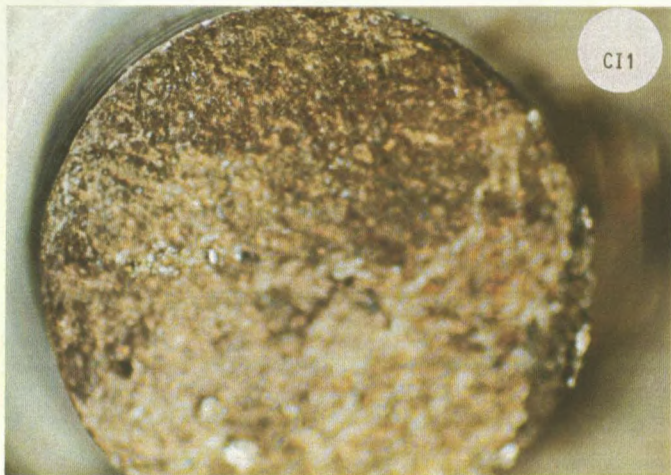
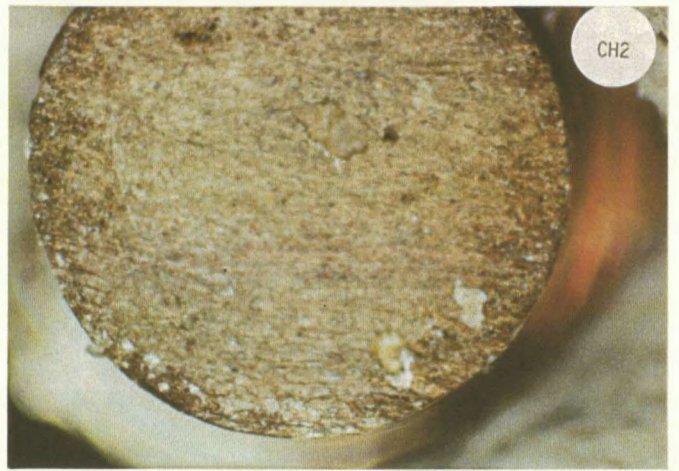


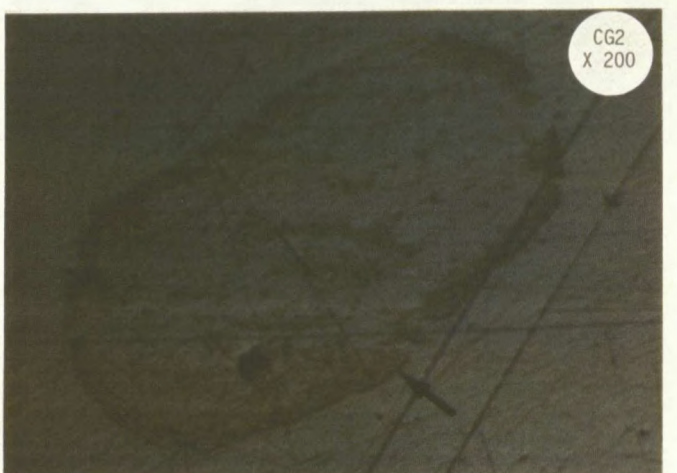
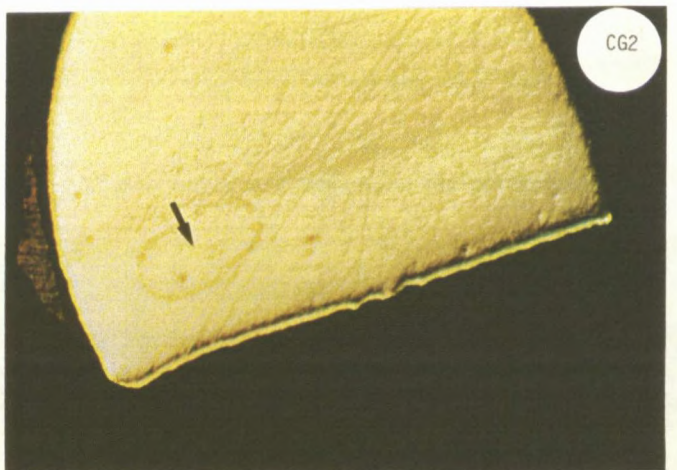
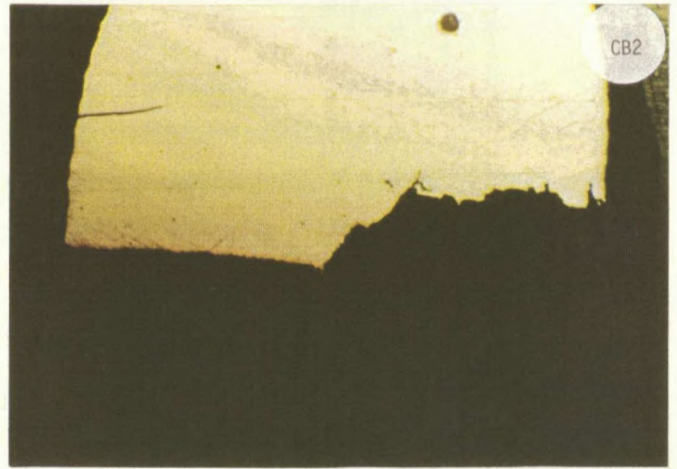
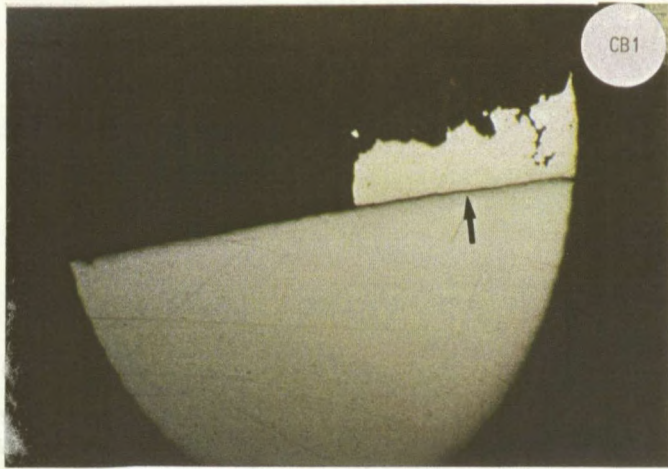
ANNEXURE E

PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM TWO - BASE METAL TO PRECIOUS,
NO GOLD PLATE

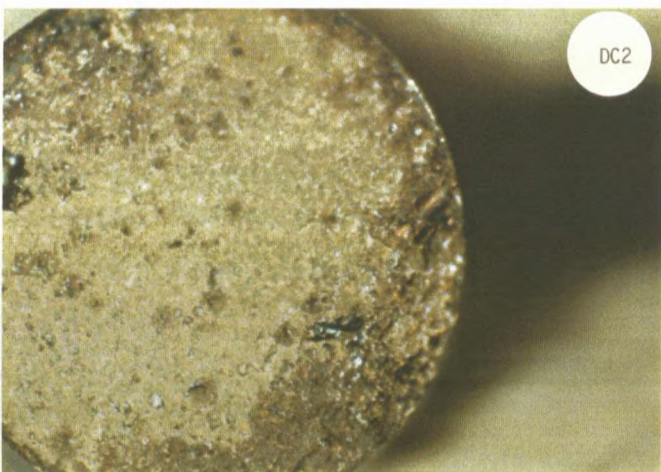
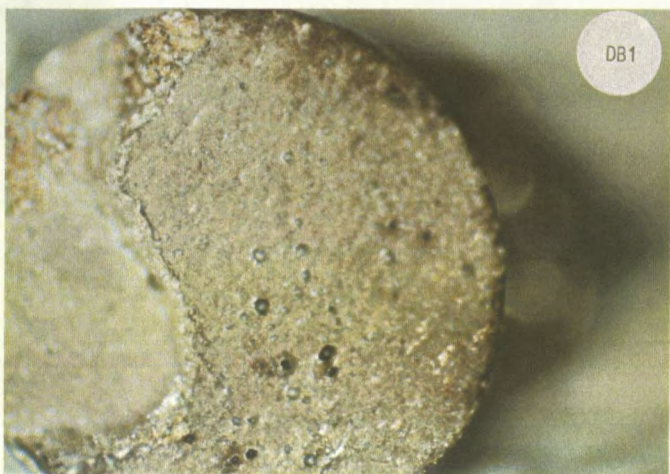
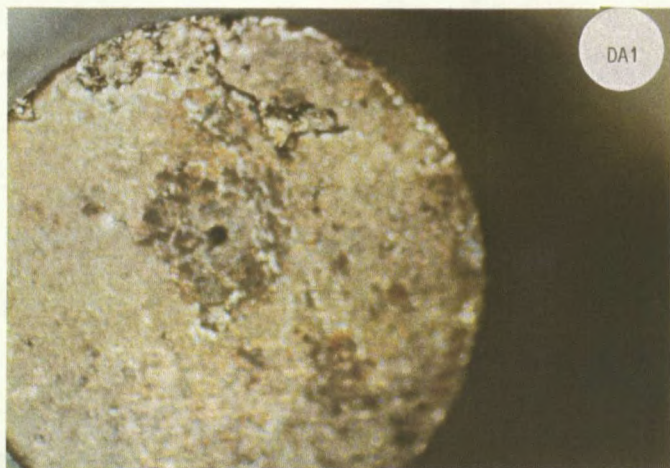


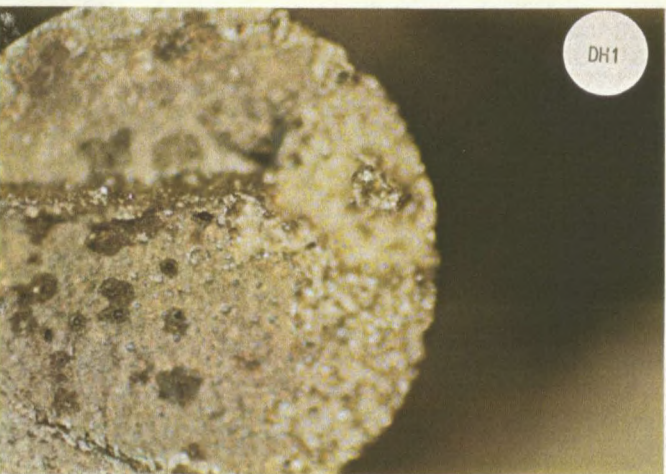
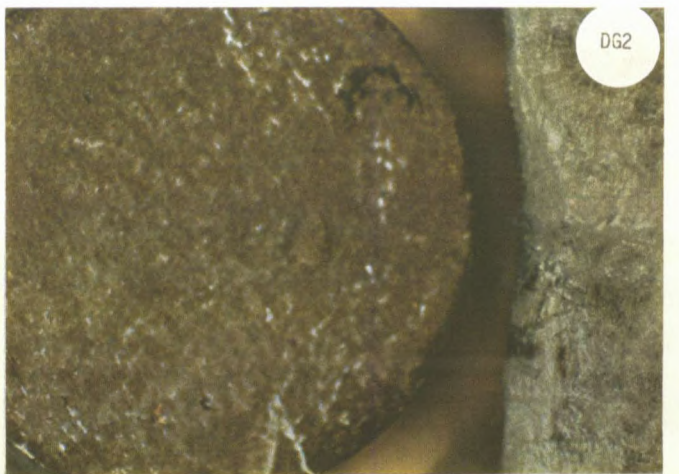
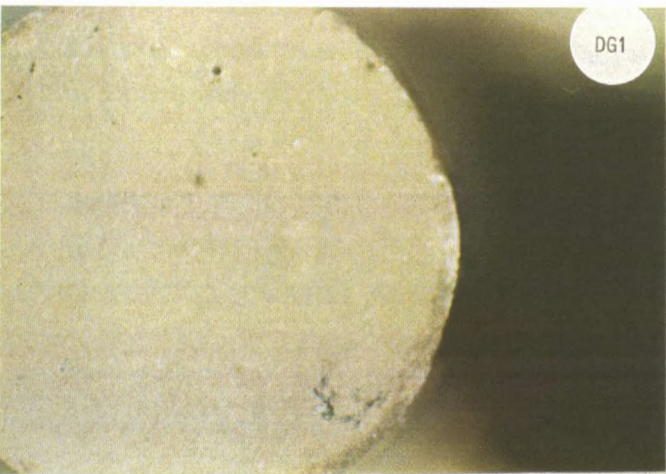
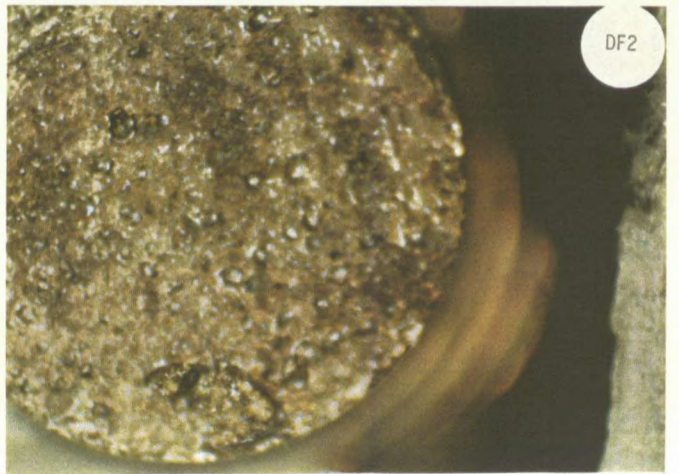
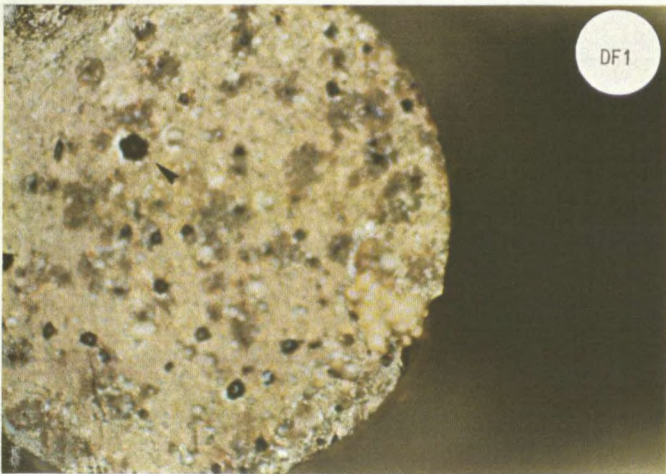
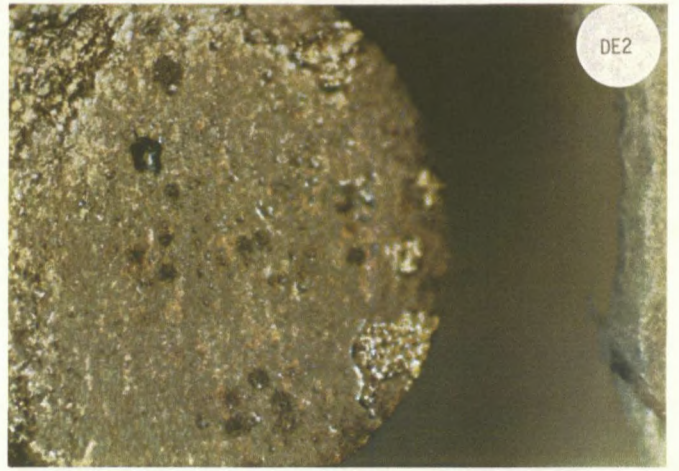
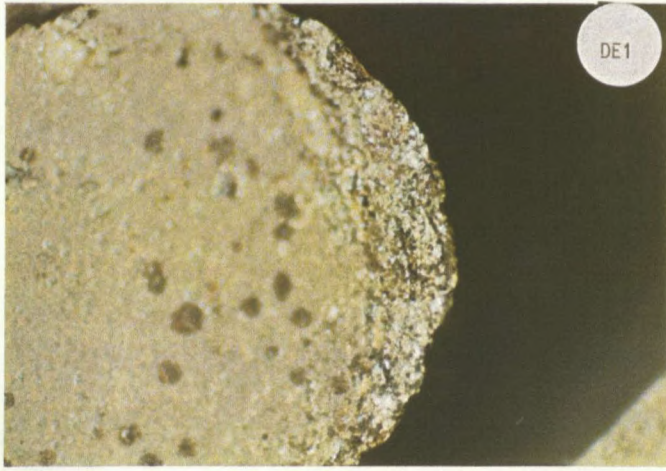


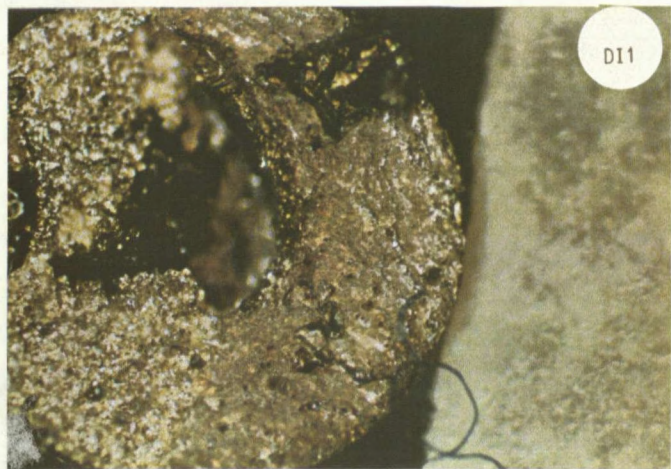


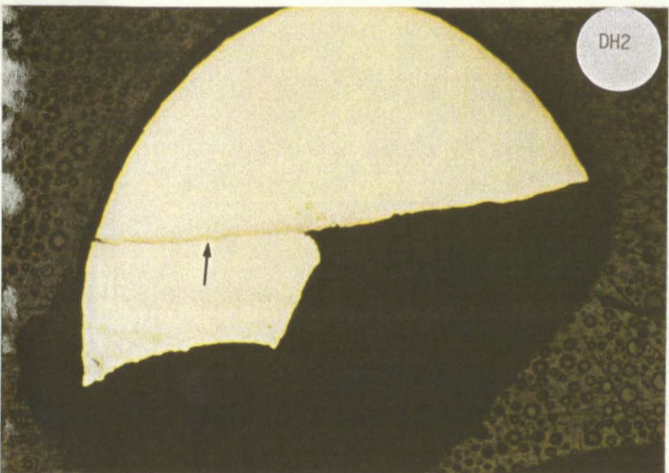
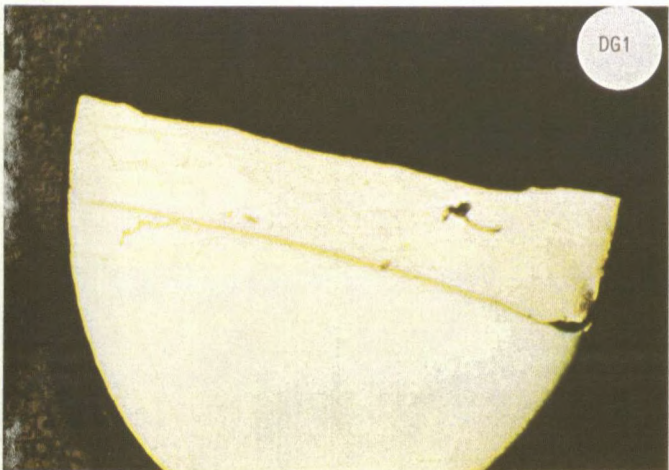
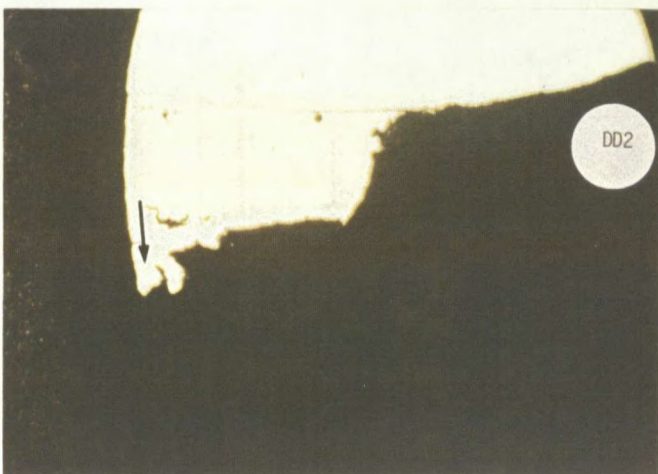
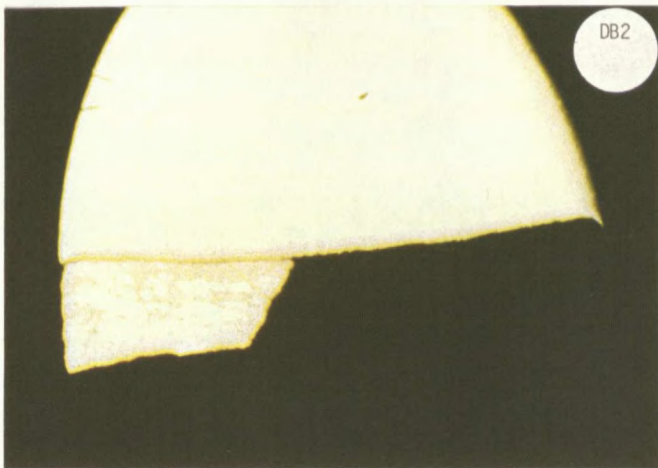


PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM THREE - BASE METAL TO BASE METAL,
WITH GOLD PLATE



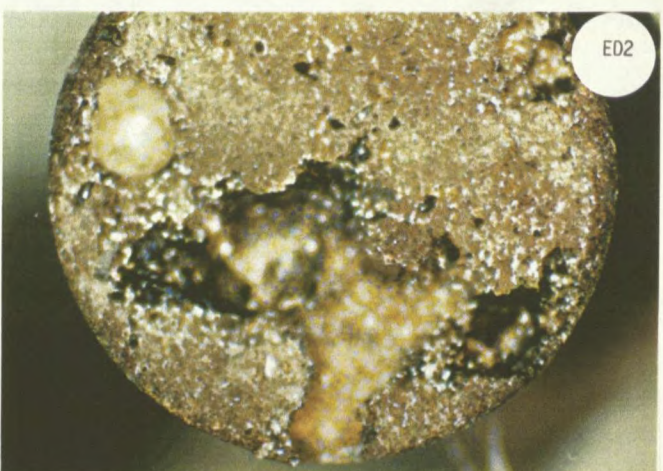
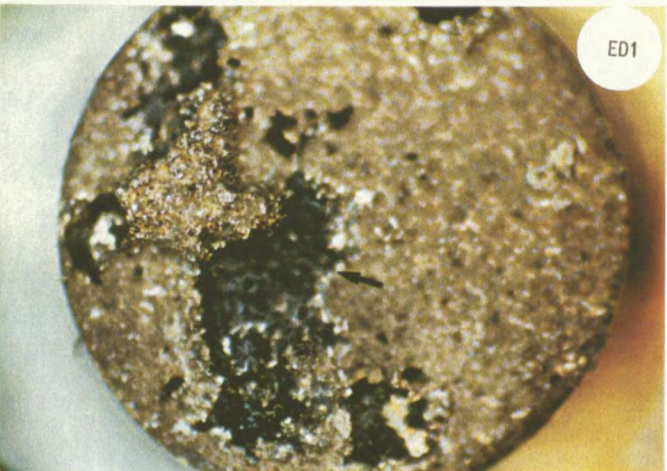
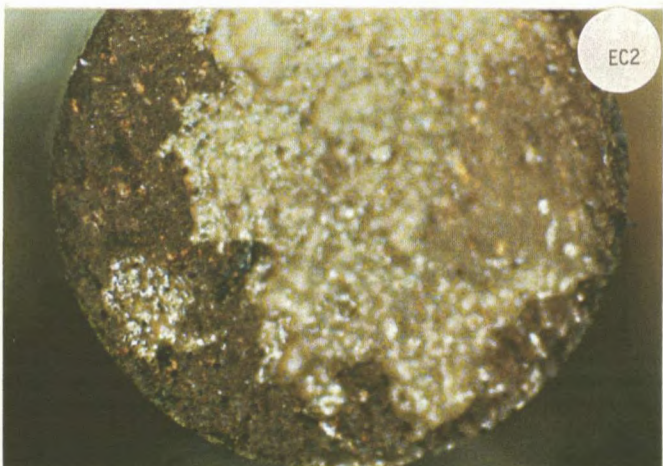
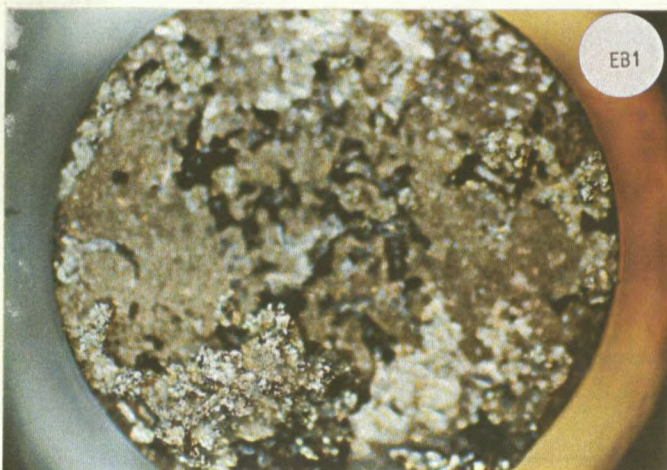


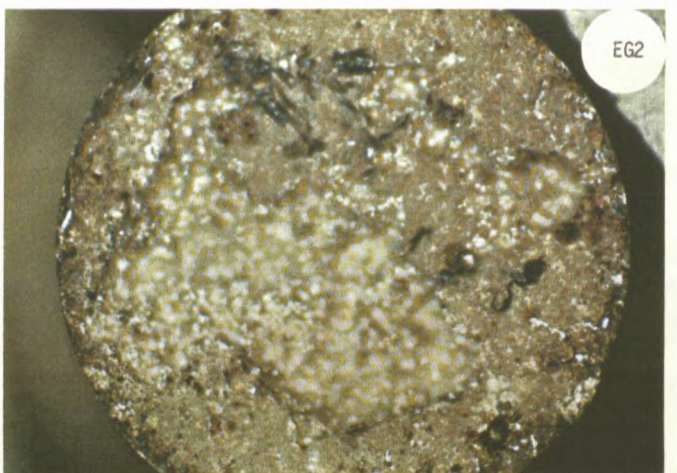
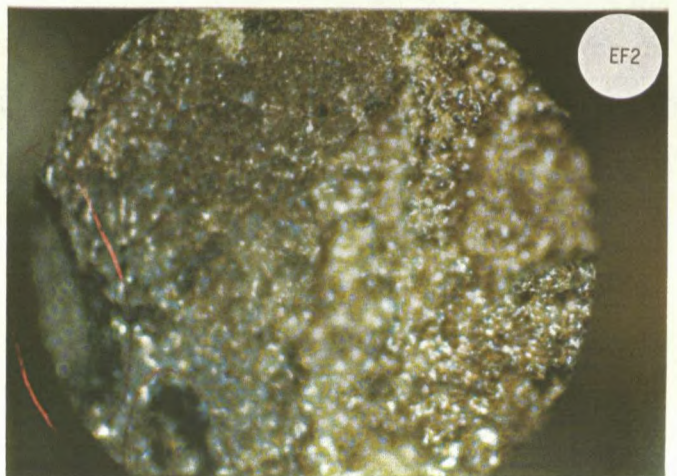
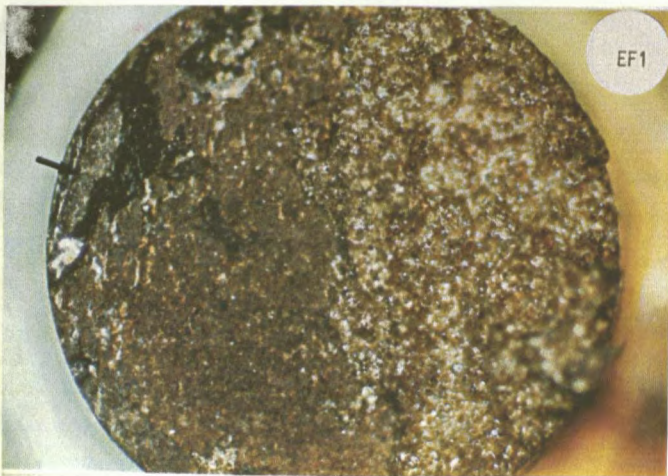
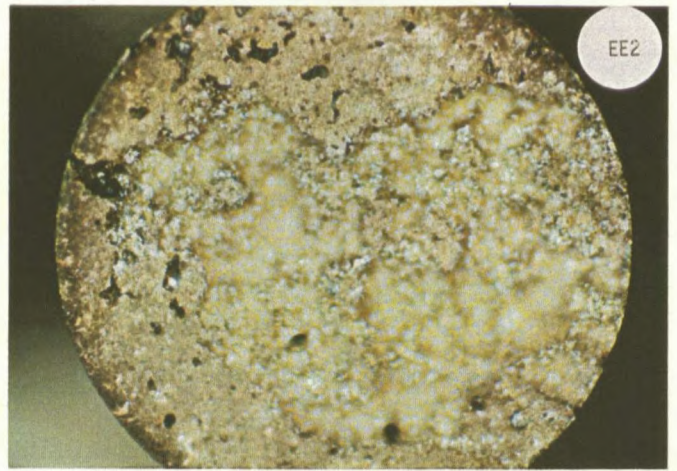
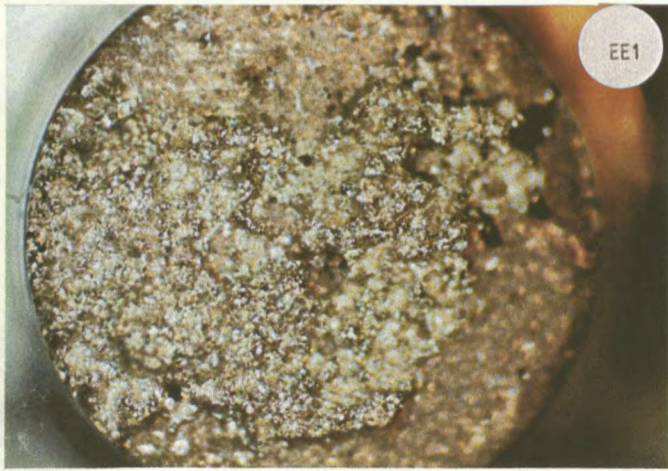


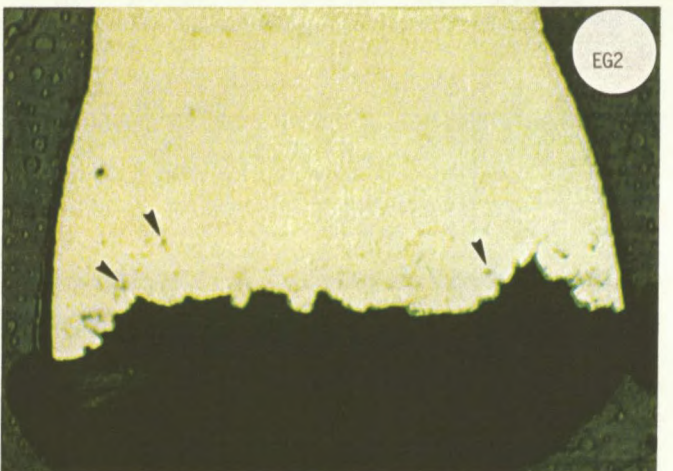
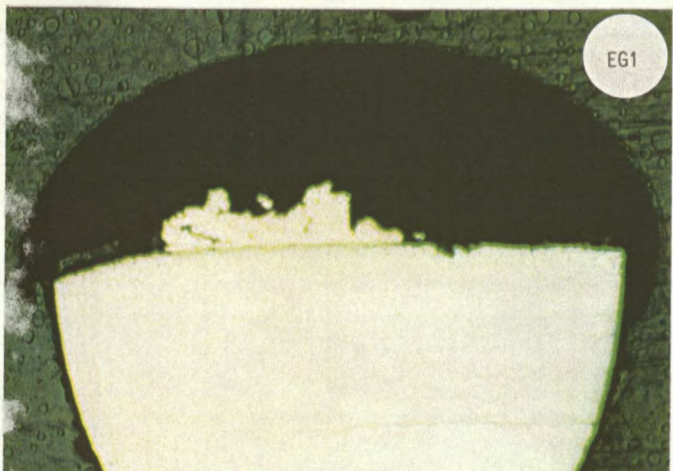
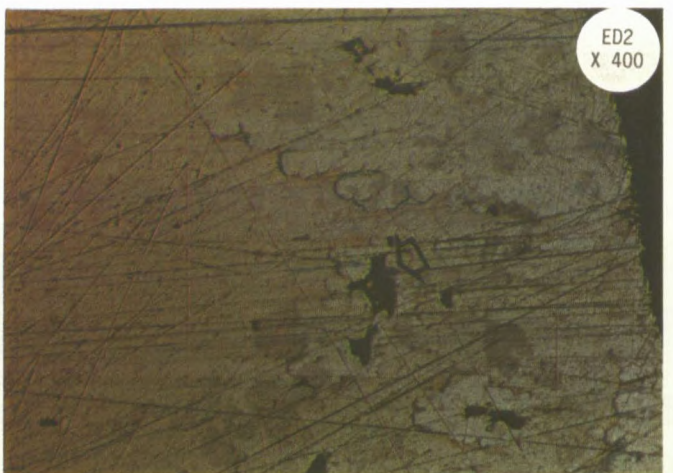
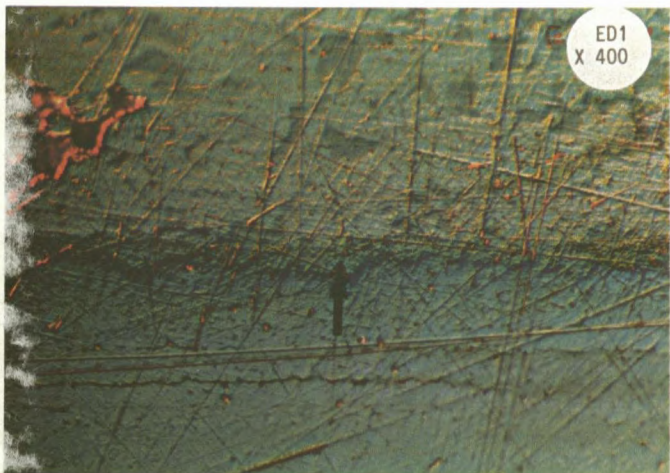
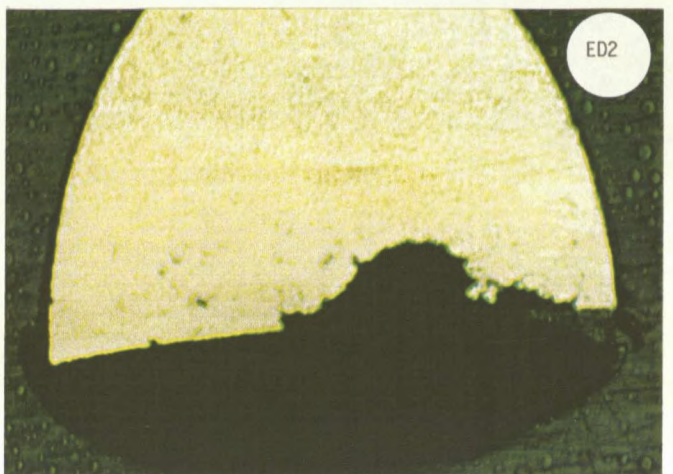
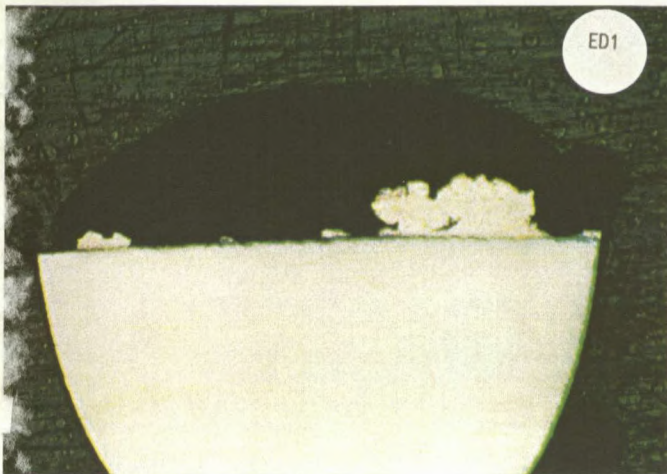
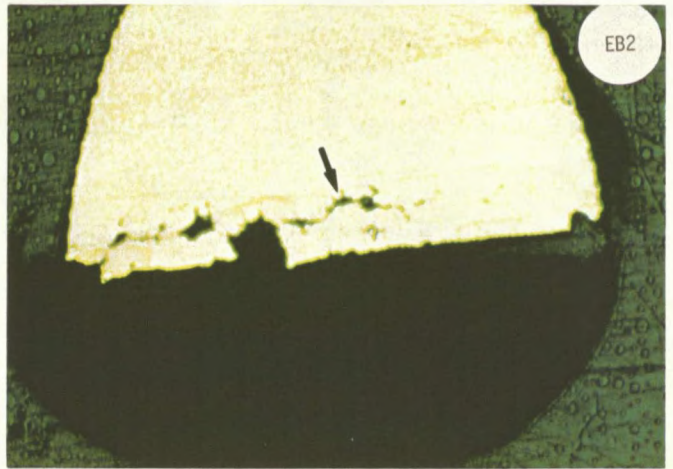
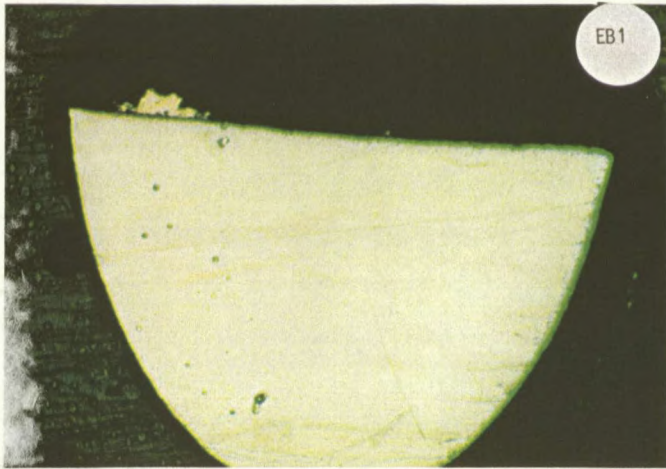


ANNEXURE G

PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM THREE - BASE METAL TO SEMI-PRECIOUS,
WITH GOLD PLATE







PHOTOMICROGRAPHS OF END-ON VIEWS AND SIDE-ON
VIEWS OF SOLDER JOINT BREAKS
SUBPROBLEM THREE - BASE METAL TO PRECIOUS,
WITH GOLD PLATE

