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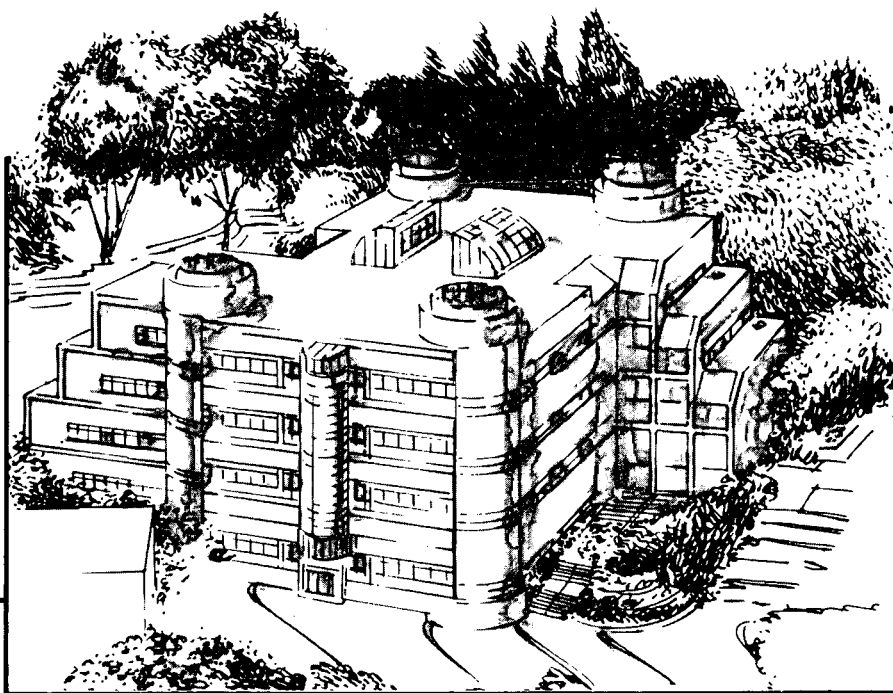
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Effect of Au on the Reliability of Fine Pitch Surface Mount Solder Joints

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The effect of Au on the reliability of 0.65 mm pitch surface mount solder joints between plastic quad flat packs and Cu-Ni-Au FR-4 printed circuit boards was investigated. Cu-Ni-Au is a desirable printed circuit board finish for multi-chip modules or printed circuit boards that would otherwise require a selective Au finish, for example for edge connectors or wire bondable parts. However, Au is known to embrittle solder when it is present in sufficiently high concentrations, creating a concern that solder joint fatigue life in service will also be adversely affected.

This paper reports the results of mechanical shock, mechanical vibration and thermal cycling testing of fine pitch solder joints containing varying amounts of Au. Tests were performed on as-soldered joints and on joints that had been heat-treated to evolve the microstructure toward equilibrium. The tests were designed to accelerate in-service conditions in a typical industrial environment. Under these conditions, the Au concentrations tested did not promote solder joint failures.

Microstructural characterization of the distribution and morphology of the Au-, Ni- and Cu-Sn intermetallics in the joint before and after accelerated testing was also performed. On the basis of these observations it is recommended that the Au concentration in solder joints between plastic quad flat packs and Cu-Ni-Au FR-4 printed circuit boards not exceed 3.0 wt. %.

INTRODUCTION

There is considerable interest in using printed circuit boards (PCBs) with Cu-Ni-Au metalization for some surface mount applications. Cu-Ni-Au is a desirable alternative to the standard hot-air-levelled (HAL) solder board finish for printed circuit boards that would otherwise require a selective Au finish, for example for edge connectors or wire bondable parts. In addition, for thick PCBs, the Ni layer is believed to confer additional resistance of the plated through holes to thermal fatigue failure.

However, a potential reliability problem posed by Au has prevented PCB manufacturers and surface mount assemblers from taking advantage of these benefits in surface mount printed circuit assemblies. Au is highly soluble in molten Sn-Pb solder (see Figure 1) and

it dissolves extremely rapidly.^{1,2} Because Au has essentially no solubility in either Sn or Pb, when the solder joint solidifies, Au is dispersed through the joint in the form of brittle, elongated AuSn_4 or $\text{Au}(\text{Sn,Pb})_4$ intermetallic precipitates (Figures 1 and 2). As a result Au embrittles solder when it is present in sufficiently high concentration, creating a concern that solder joint fatigue life in service would also be adversely affected. Many of studies of the influence of Au on the mechanical properties of bulk solder or solder joints investigated the effect of very thick Au plating ($> 50 \mu\text{in}$) on joints made by solder dipping or wave soldering. Since the Au concentration of the final joints is unknown, these studies are of little value in assessing the degradation that may occur at low Au concentrations. There are a few systematic studies of monotonic tensile and shear behavior in the literature; the authors of these studies have recommended that the Au concentration in the solder not exceed 4-6 wt.%.^{3,4,5,6} Lau, et al. found that the reliability of solder joints to plastic quad flat packs (PQFPs) was not affected by the addition of 0.3-0.5 wt. % Au.⁷ However none of these studies directly investigated actual failure mechanisms such as fatigue that impact long term reliability.

In response to these concerns, the effect of Au on the reliability of Cu-Ni-Au metallized printed circuit boards with fine pitch plastic quad flat packs was investigated from both a process and a reliability standpoint. The goal of the study was to determine an acceptable level of Au in the solder joint. The approach was to establish a definition of reliability and then to define and conduct accelerated mechanical shock and vibration and thermal cycling tests to determine a safe Au concentration in the solder joint. This value can be used in combination with surface mount design rule specifications and Au plating thickness specifications for the PCB to make recommendations about acceptable maximum and nominal PCB Au plating thicknesses. The test plan was based on a target service life of 10 years in a typical industrial office environment.

PROCEDURE

Materials. Fine pitch 0.65 mm (0.0256 in) EIAJ standard 160-pin metric plastic quad flat packs (MQFPs) were surface mounted to FR-4 printed circuit boards. The samples used for shock and vibration testing had Cu leadframes, while those used for the thermal cycling testing had Alloy 42 (Fe-42Ni) leadframes. Different test board designs were used for shock and vibration and for thermal cycling. The boards were 0.031 in (0.8 mm) and 0.062 in (1.6 mm) thick respectively; in addition the boards used for thermal cycling had two patterned Cu inner layers. The PCB metallizations that were tested were Cu-Ni-Au with nominal Au thicknesses of 5, 10, 20 and 50 μin (0.13, 0.25, 0.5, 1.3 μm , respectively) and solder mask-over-bare-Cu (SMOBC) with HAL solder as a control. The Au thickness was measured at the same location on each PCB; however, electrolytic Au plating thickness is sensitive to current density and may vary greatly across the board. The composition of the solder was 63Sn-37Pb (wt %). The solder was applied by stencil printing solder paste with a metal content of 90.3 wt. % (52% by volume). The pad geometry on the PCB was 0.100 x

0.015 inches (2.5 x 0.4 mm). The solder volumes were 8700 mils³ (0.14 mm³) and 5000 mils³ (0.08 mm³), for the joints for vibration and cyclic thermal fatigue, respectively.

Processing. The joints were made using standard surface mount assembly processes. The solder paste was stencil printed through a metal stencil. Components were placed by a high-precision automated placement machine. The boards were reflowed in an infrared oven using a thermal profile with a dwell of 2 minutes between 130 and 160°C to vaporize the organic solvents in the solder paste and approximately 100 seconds above the 183°C liquidus temperature for the eutectic solder. The average cooling rate between the peak temperature (approximately 220°C) and 100°C was measured at one location on each test board and found to be 0.5°C/sec for the vibration test board and 0.8°C/sec for the thermal cycling test board.

A solder joint may be subjected to three types of thermal exposures during its lifetime: reflow, rework of adjacent components and long-term exposure at the operating temperature of the system. To examine the possible effects, these thermal environments were simulated prior to testing. Joints were tested after three different thermal exposures: as-reflowed, as-reflowed + simulated adjacent rework (170°C/2 min) and as-reflowed + simulated lifetime thermal exposure (110°C/3 days). The simulated adjacent rework thermal cycle is believed to be a worst-case cycle experienced by adjacent components; this cycle causes rapid Cu-Sn and Ni-Sn intermetallic growth and coarsening of the Sn and Pb phases and any voids that may be present. The rework process itself was not simulated because it affects the joint similarly to an additional reflow cycle. The lifetime thermal exposure is designed to simulate long-term exposure to system operating temperatures, which gradually coarsens the solder microstructure. This heat treatment largely eliminates the eutectic lamellae formed during solidification and induces significant Ni-Sn intermetallic growth at the solder/PCB interface. Although the microstructure of any real joint will coarsen over a period of months at the operating temperature, only some of the joints were aged since the effect of high temperatures on the differential coarsening rates of the solder and the intermetallics is unknown. This heat treatment was not performed on joints subjected to thermal cycling because of the extensive coarsening that occurs during the high temperature portion of the cycle.

Au concentration calculations. Accelerated tests of PQFP solder joints sample a distribution of Au concentrations and solder volumes. Even on a single board, the nominal Au concentration in the solder joints may significantly underestimate the Au concentration found in some joints. The nominal Au concentration in the joint can be calculated under the assumption of complete mixing using the nominal Au thickness and the nominal solder volume. However, both the Au thickness and the solder volume will vary in practice leading to a distribution of Au concentrations in the solder joints. Due to differences in current density associated with location on the panel and flight bar and circuit feature density, the electrolytic Au plating thickness on a board (or even a single land pattern) is not uniform. For typical electrolytically plated PCBs, a nominal Au thickness of 5 μin corresponds to a Au thickness range of 3-7 μin with a mean of 4-4.5 μin.

The Au concentrations for the experimental joints were calculated on the basis of the following assumptions about the volume of solder and the amount of Au dissolved into it:

- the solder volume is 50% of the paste volume;
- all Au on the pad is dissolved into the joint;
- the Au thickness on the pad is the measured mean Au thickness.

Table 1 shows the Au concentrations that were actually tested.

Table 1. Actual Au concentration ranges $\bar{x} \pm \sigma$ in wt. % of experimental joints on reliability test boards based on actual mean Au thicknesses and corresponding standard deviations. The actual mean Au thickness for each case is shown in parentheses. (Au concentration values have been rounded to the nearest 0.1 wt. %; Au thickness values are quoted to the nearest 0.5 $\mu\text{in.}$)

Test	Nominal Au thickness ($\mu\text{in.}$)			
	5	10	20	50
Vibration	0.5 wt% (6.5 $\mu\text{in.}$)	0.8-0.9 (12.5)	1.2-1.7 (19)	3.9-4.9 (64)
Thermal cycling	0.5 (4.0)	1.4-2.1 (13.5)	2.1-3.0 (20)	7.1-10.1 (72)

Service environment. The service environment for surface mount assemblies varies widely. Thus the "reliability" of a technology (i.e. fraction of components that remain viable) depends on the environment and desired life. The technology should be sufficiently reliable in any less severe environment than the one it is qualified for, but may or may not exhibit higher failure rates in a more severe environment. For the purposes of this study, the assumed service environment, believed to be typical of an industrial office environment, is:

- 10 years operating life
- 1 on/off cycle per day (5 day week)
- Operating thermal cycle approximately +20°C to +90°C, i.e. $\Delta T = 70^\circ\text{C}$
- Possible mechanical vibrations
- Possible mechanical shock.

The thermal cycle assumed here is relatively large for computer and instrument products; however, the thermal cycle and the environment in general are considerably less severe than that experienced by some assemblies, for example in avionics systems.

Reliability tests. The tests were designed on the assumption that the predominant solder joint failure mechanism is thermomechanical fatigue of the solder joint over the life of the system. To assess the impact of using Cu-Ni-Au boards on the reliability of fine pitch solder joints mechanical shock, mechanical vibration and thermal cycling tests were conducted. The mechanical shock and vibration conditions are similar to those used by Lau, et al.⁸

The following tests were used to evaluate the reliability of the solder joints:

- Mechanical shock Board thickness: 0.031 in (0.8 mm)
 Displacement orientation: Out-of-plane
 Amplitude: 275 in/sec (7 m/sec) at 425 g, 3.6 msec pulse length
 No. cycles: 3 drops
- Mechanical vibration Board thickness: 0.031 in (0.8 mm)
 Displacement orientation: Out-of-plane
 Amplitude: 10 g
 No. cycles: 30 hrs at 60-90 Hz frequency swept sine wave (resonates samples)
- Thermal cycling Board thickness: 0.062 in (1.6 mm)
 Amplitude: 0°C-109°C; 83 minutes/cycle
 (heating and cooling rates ~ 5.2°C/min and 3.3°C/min, respectively; minimum dwell, 6 mins)
 No. cycles: 1100 cycles simulates 10 years.

The test board for mechanical vibration and shock was designed to deflect during testing to induce cyclic stress on the solder joint. In thermal cycling the displacement is imposed as a result of the differential thermal expansion between the component and the PC board. Differential heating and cooling rate effects were minimized by choosing the component density so that the heat capacities of the board and the components were approximately matched.

The solder joints were electrically monitored for integrity during the reliability testing. Components were obtained with alternate pairs of leads wire-bonded together inside the package. The PC boards were designed so that the solder joints were the final links of a daisy chain through the component and the PCB. A single solder joint failure (open) opens the circuit and allows detection of a failure at that component. The components were monitored during testing for solder joint failures using an HP 3497 data acquisition controller attached to an HP 9000/236 computer that checked the individual daisy chains for continuity. A solder joint failure was defined as a ten percent increase in measured resistance that was repeated in later cycles.

Accelerated testing strategy. Actual joints were selected over bulk samples of similar nominal composition to ensure that as many of the possible compositional and microstructural inhomogeneities that may exist in the real joints were included. In addition, fatigue of PQFP solder joints differs from fatigue of experimental solder joint samples designed for controlled testing of the fatigue or mechanical response of solder in three major respects.

The stress and strain distributions that arise during cycling of PQFPs are qualitatively different from those experienced by experimental samples. Finite element analyses have shown that the solder joints that attach a PQFP have a grossly non-uniform distribution of stress and strain. (It is for this reason that most experimental samples are leadless and mimic more accurately the behavior of leadless chip carriers.) The compliance of the lead means that the stresses on the solder joint and the imposed cyclic displacement are relatively low. In contrast to leadless geometries, creep of the solder decreases the stress on the joint slowly (the loading on the solder joint is closer to being stress-controlled than displacement-controlled). Consequently, the effect of creep on the total plastic strain in the joint is limited, even over a 24 hour period, except in regions of high stress concentration. When cracks do initiate, the complexity of the loading geometry means that stress/strain distribution and the driving force for crack growth are affected.

The joints may have an inhomogeneous Au distribution, in addition to the inhomogeneous microstructure that results from variations in cooling rate and the inhomogeneous strain distribution during cycling. Au dissolves very rapidly into solder. For all of the Au thicknesses investigated in this study, once the joint was formed there was no pure Au remaining at the interface. However, while the nominal Au concentration ($[\text{joint volume}]/[\text{land area}]$) is a useful descriptor, it may be too simplistic a measure to define the joint microstructure. The initial Au concentration at the interface is very high and the Au-Sn phase(s) that form are kinetically rather than thermodynamically determined until complete mixing of Au into the molten solder occurs. Furthermore, initial experiments showed that solder does not necessarily wet the entire pad if the paste brick is much smaller than the pad. A beneficial consequence of this phenomenon is that a smaller amount of Au is dissolved into the joint. However, the result is that the Au concentration will be locally high at the periphery of the joint, where there is a solder/Au interface. A preponderance of brittle phases there may provide a crack initiation site that greatly shortens fatigue life.

Finally, because of the small solder volume in the joint, fatigue life is strongly dependent on the distribution of initial flaws that provide initiation sites for fatigue cracks that propagate to failure in subsequent cycles. In a few cases, cracking has been observed at the solder/AuSn₄ interface; these cracks may have formed as a result of high tensile stresses generated by differential cooling rates after reflow.

In the regime of Au concentrations under study, the variation in the mechanical and fatigue properties of the solder with Au concentration is expected to be monotonic. The shear and tensile properties of bulk Au-containing solder fit this description. If the fatigue life of the solder is affected by Au in this regime, it is likely to be monotonically decreasing. The basis for this statement is that the solder is a phase mixture of Pb, Sn and AuSn₄ for all the Au concentrations of interest in this study; only their proportions will vary. Thus if specimens of a certain Au concentration are found to be reliable, specimens with lower concentrations are expected to be reliable as well.

RESULTS AND DISCUSSION

Mechanical shock. There were no failures of solder joints or component leads during shock testing. This result is consistent with those of Lau, et al.,⁸ who observed only a few mechanical shock failures in tests conducted after mechanical vibration, which probably introduced fatigue cracks.

Mechanical vibration testing. Only four samples of the 240 tested failed during the test. Approximately one-half of the vibration samples were subjected to mechanical shock first. The small number of samples for the individual conditions makes it difficult to draw conclusions with statistical weight. However the samples that failed were not strongly biased toward thicker Au, prior long-term low temperature aging or exposure to prior mechanical shock. All of the samples that failed had received the simulated adjacent rework heat treatment. All of the failures occurred in the leads at the top of the solder joint rather than in the foot of the joint.

It is difficult to make a quantitative correlation between the results of this test and expected reliability. Lau, et al.⁸ did similar tests and analyzed the results using a Weibull distribution, the most conservative assumption from a reliability perspective. They concluded that more than 90% of the joints would survive more than 10 million military general vibration cycles. The failure rate observed in this test was lower than in Lau's; the vibration fatigue life is therefore predicted to be even longer.

Thermal cycling. When the test was stopped after 1450 thermal cycles, no solder joint failures had been observed. While there may still be an effect of Au on the fatigue life of these joints, all of the joints seem sufficiently resistant to thermal fatigue for the target service environment. The results are consistent with the results of thermal cycling tests of 0.5 mm pitch components conducted by Lau, et al.,⁹ in which the first failures occurred after more than 2000 cycles with a ΔT of 165°C (cf. with the qualification test used in this study of 1100 cycles with a ΔT of 110°C).

The thermal fatigue test was designed so that 1100 cycles simulated 10 years of exposure to the specified thermal cycle (20-90°C, one cycle/day). The relative severity of larger thermal cycles can be estimated by relating the desired number of cycles with $(\Delta T)^2$. This correlation has some fundamental basis in the relationship between displacement, shear strain in the solder, and crack initiation in creep fatigue and seems to be relatively good for relatively small shear strains.¹⁰ For leaded components, it is probably conservative. Increasing ΔT from 70 to 110°C reduces the number of thermal cycles by a factor of approximately 2.5. Thus the test simulates life exposure of more than 13 years.

Solder joint microstructures. The solder joint microstructures were characterized before and after both vibration and thermal cycling and at intervals during the thermal cycling test. The microstructures were examined using optical microscopy of metallographic cross sections and a Four Pi™ x-ray laminography system (which provides an x-ray cross section or "laminograph"). The key observations were: 1) under identical solidification conditions,

the Sn-Pb microstructure becomes coarser with increasing Au content of the joint; 2) the needle-like AuSn_4 intermetallics become aligned with the lines of shear during vibration fatigue tests; and 3) extremely thick Au plating promotes the formation of voids within the solder joint. The last observation, of large voids in joints made on 50 μin Au boards, may be of considerable importance to solder joint reliability. Each of these observations is discussed in further detail below.

The coarsening of the Sn-Pb microstructure with increasing Au concentration is illustrated in Figure 3. As the Au concentration is increased, more Sn is incorporated into Au intermetallics, resulting in an increase in the amount of pro-eutectic Pb. In addition, Au appears to alter the eutectic solidification kinetics, resulting in a coarser Sn-Pb microstructure.

During vibration fatigue testing, the needle-like AuSn_4 intermetallics change their orientation. Figure 4 (top) shows the orientation of the rods in an as-solidified specimen. The orientation of the rods does not appear random; for example, near the solder-PCB interface the rods appear to have been frozen perpendicular to the interface. Figure 4 (bottom) shows the appearance of the identical location in another specimen after vibration testing. The intermetallics are now aligned at right angles to their original orientation. In this orientation, they lie along the lines of shear experienced by the specimen during fatigue cycling. In this position, they provide minimum resistance to plastic deformation of the solder. The mechanism for reorientation is presumably largely grain and phase boundary sliding, known to be prevalent in solder even at room temperature.

No re-orientation of the intermetallic phase was discernible in the thermally-cycled specimens. Samples microstructures before and after cycling are shown in Figure 5. The AuSn_4 intermetallics appear randomly dispersed throughout the joint in as-soldered specimens. During thermal cycling, the high temperature portion of the thermal cycle coarsens the Sn-Pb microstructure, but the size and distribution of the AuSn_4 intermetallics does not change significantly. There are several differences between the thermal and mechanical vibration fatigue cases that might explain why no reorientation was observed. The components assembled to the thermal cycling boards had Alloy 42 rather than Cu leadframes. This compositional difference along with the greater board thickness caused the initial Sn-Pb microstructure to differ. An equiaxed grain structure was promoted over the globular microstructure observed in the vibration specimens, and blocky Ni-Sn intermetallics are dispersed through the joint. In addition to these microstructural differences, the number of fatigue cycles and the strain rate at which they were performed are substantially different. The vibration specimens received more than 8 million cycles at 60-90 Hz, while the thermal cycled joints pictured received less than 1000 cycles at about 2×10^{-4} Hz.

Increased voiding was observed in solder joints containing more than 4 wt. % Au, i.e. in joints made on 50 μin Au boards. Although the number of small voids seemed to increase with Au content in metallographic cross sections of as-soldered joints, it is difficult to do

enough cross sections to get statistically significant data. However, in joints that were subjected to the adjacent rework simulation treatment of 170°C/2 min, these small voids coalesced into much larger ones, as illustrated in Figure 6. This process is theoretically similar to the coarsening of a distribution of small particles; energy is decreased for a sparser distribution of particles with a lower surface-to-volume ratio. These voids were large enough (>5 mils) that they were easily observable with x-ray laminography as light areas (through which x-rays are transmitted) in the fillet region, which made it possible to compare large numbers of joints. It was confirmed that large voids were much more prevalent in joints with high Au concentrations.

The source of voiding in joints with high Au concentrations is not definitively understood. Because these voids were not observed in joints that contained little or no Au, it is believed that they do not result from solvent entrapment caused by a poor reflow profile, but are associated with the high Au concentration in the joints. Unusually high porosity has been observed before in Au joints,¹¹ but no reasons for the phenomenon were suggested. However at high Au concentrations there is a high volume fraction of AuSn₄ intermetallics in the melt that, along with the component lead, provide surfaces on which any gas bubbles present in the melt may be entrapped. The Au itself may be a source of voiding. Organic molecules incorporated into the plating may decompose and vaporize when the Au dissolves into the molten solder. Another possible explanation is the large negative volume change associated with the reaction between solid Au and Sn (in this case the metallic, or white, body-centered tetragonal form referred to as β -Sn) to form AuSn₄. The AuSn₄ structure is orthorhombic with prototype PdSn₄. This structure is morphologically similar to the structure of the low temperature form of Sn (gray or α -Sn, which has a diamond cubic structure) with two Au atoms added to the interior of the diamond cubic cell.¹² However, the volume change associated with this solid state transformation can only explain the observed voiding if the as-solidified solder joint is supersaturated with Au that transforms to AuSn₄ by solid state reaction; it does not explain the voiding if the AuSn₄ forms during solidification from the melt. Although small voids are common in solder joints and are not believed to substantially degrade joint reliability,¹³ voids of the size observed in these experiments have implications for both the reliability and the durability (e.g. under shock conditions) of the solder joint.

CONCLUSIONS

The accelerated tests carried out as part of this study did not indicate any increased reliability risk in the defined service environment associated with the presence of Au in eutectic Sn-Pb solder joints to plastic quad flat packs. The tests were not severe enough to cause a significant portion of the solder joints to fail, with or without added Au. Leaded packages such as PQFPs provide a highly compliant system in which the solder joint is minimally stressed. It cannot be emphasized enough that the results might be different if the package material and geometry and/or the imposed displacement were altered so that the cyclic strain in the solder joint was significantly increased (e.g. leadless ceramic chip carriers in avionics applications). The microstructural observations performed as part of

this study nevertheless present a strong case for limiting the Au concentration to less than 3.0 wt. % Au even in solder joints to PQFPs. In this experiment, Au concentrations above 4.0 wt. % resulted in large numbers of voids that could have a detrimental effect on in-service reliability during long-term exposure to both mechanical and thermal stresses. The 3.0 wt. % Au concentration limit can be used to set acceptable Au plating thickness levels. The effect of Au on the mechanism of solder joint fatigue and failure is under investigation.

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FIGURE CAPTIONS

Figure 1. Pseudo-binary phase diagram for 60Sn-40Pb (wt. %) and Au. Au is highly soluble in molten Sn-Pb solder. 60Sn-40Pb solder can dissolve more than 11 wt.% Au at 225°C. (From ref 1).

Figure 2. Optical micrographs illustrating the geometry and microstructure of the solder joint investigated in this study. The joints were made using 63Sn-37Pb solder (wt. %). The Au concentration after reflow is approximately 4.5 wt. %. Left: Geometry of solder joint between a fine-pitch quad flat pack and a Cu-Ni-Au printed circuit board. Right: Microstructure of a solder joint prior to vibration testing.

Figure 3. Effect of Au content on eutectic Sn-Pb solder microstructure. a) No Au. b) 0.8 wt%. c) 4.4 wt%. Micrographs are taken from the heel region of as-soldered vibration testing samples.

Figure 4. Reorientation of AuSn₄ intermetallics during vibration testing. Top) As-soldered microstructure in a joint containing approximately 4.4 wt. % Au. Bottom) Microstructure after vibration testing. Note that the needle-like intermetallics are now primarily horizontally oriented.

Figure 5. Solder joint microstructure (toe region) before and after thermal cycling. a) As-soldered joint on hot-air-levelled solder (0 wt. % Au), b) 0 wt. % Au, after 785 thermal cycles, c) As-soldered joint containing approximately 8.6 wt. % Au, d) 8.6 wt. % Au, after 785 cycles.

Figure 6. Voiding in thermal cycling specimens with 8.6 wt. % Au. Top) As-soldered. Bottom) After heat treatment of 170°C/2 minutes to simulate adjacent rework.

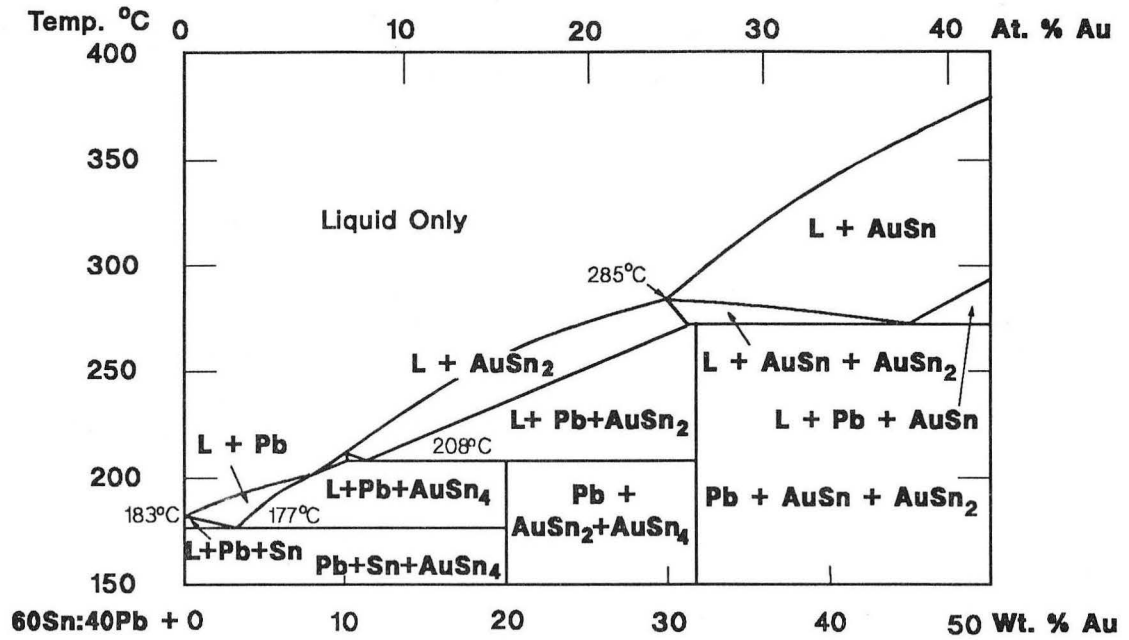


Figure 1

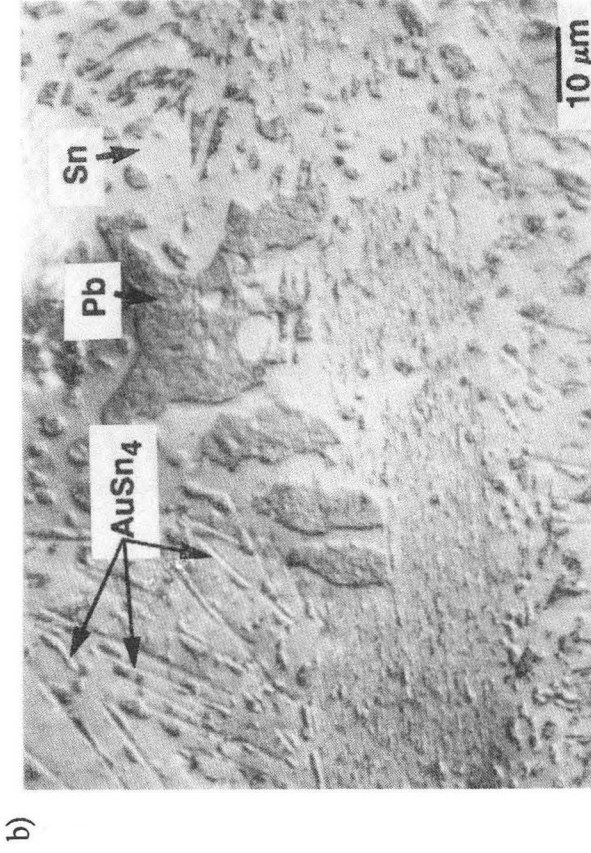
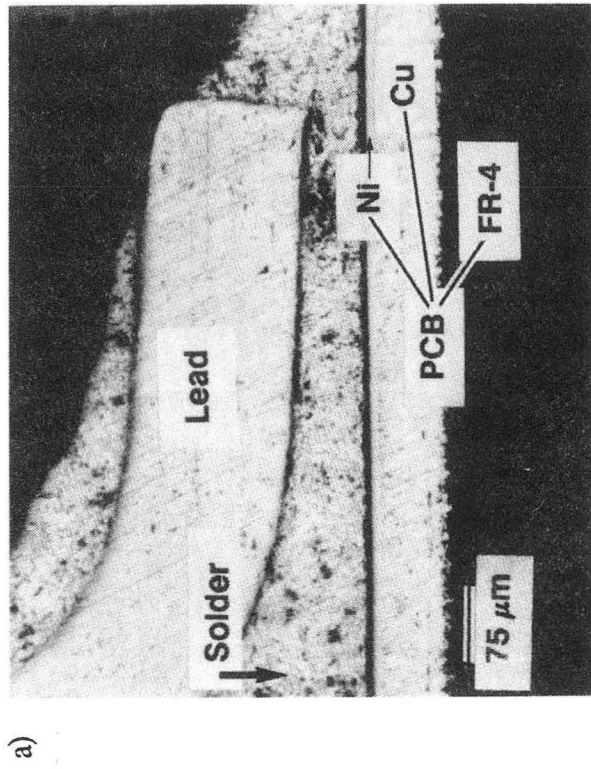
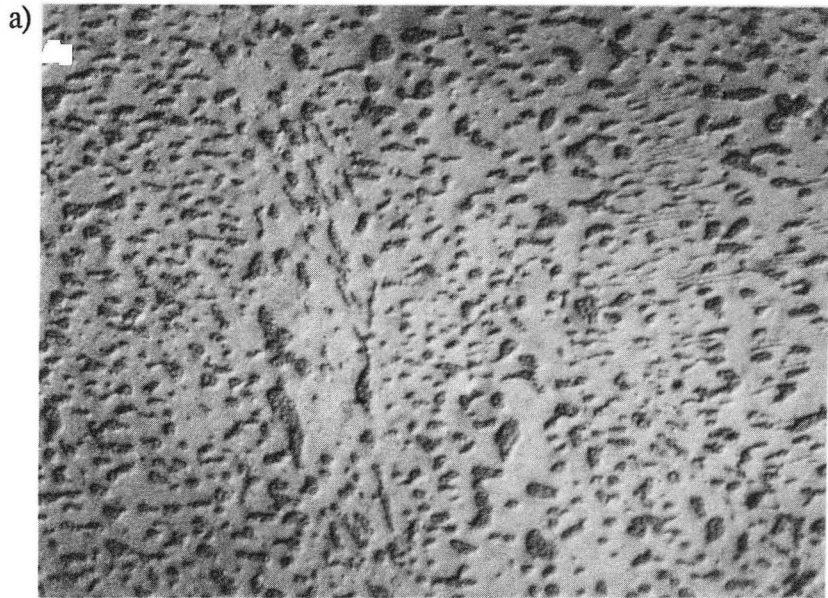
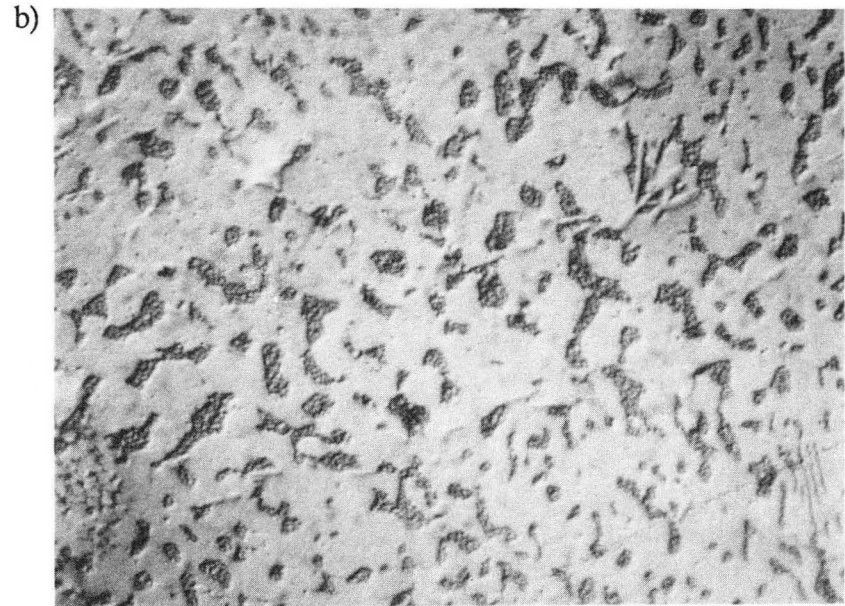


Figure 2

XBB 917-5144

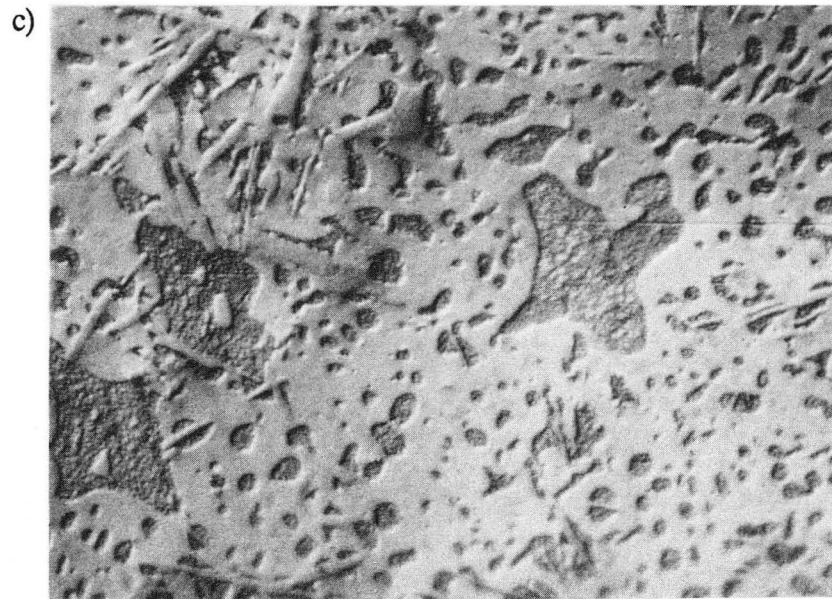


XBB 914-2483



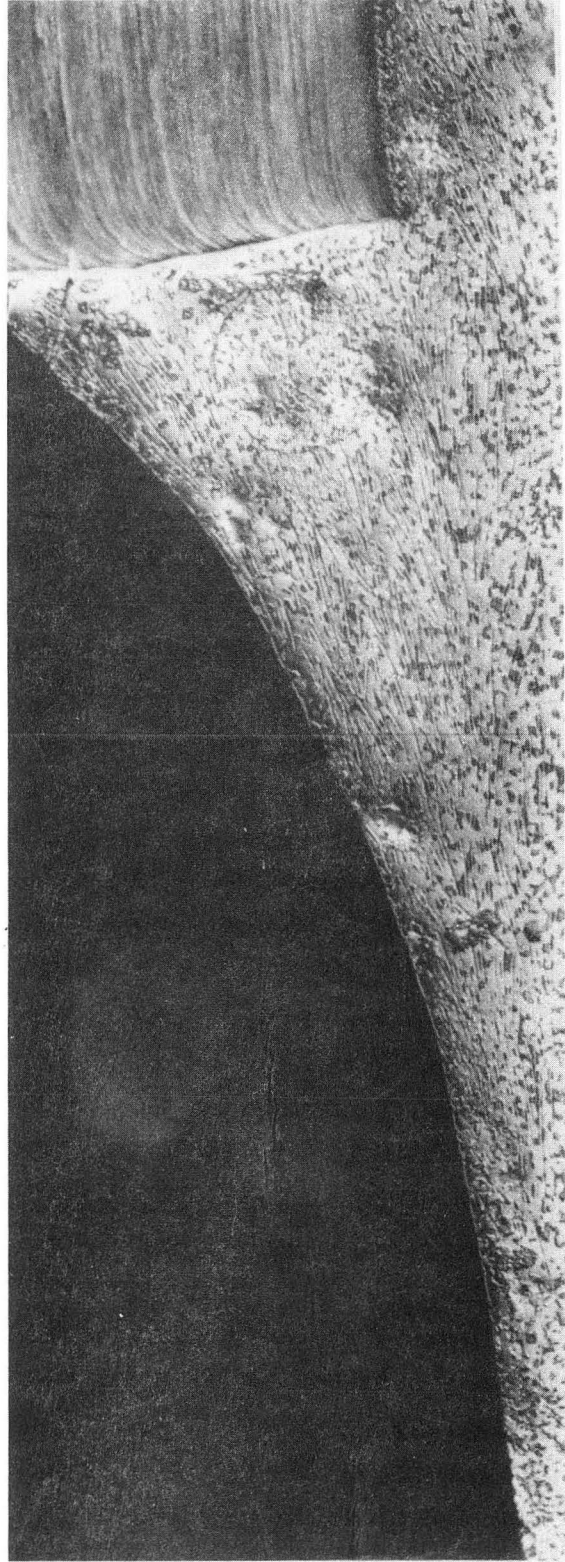
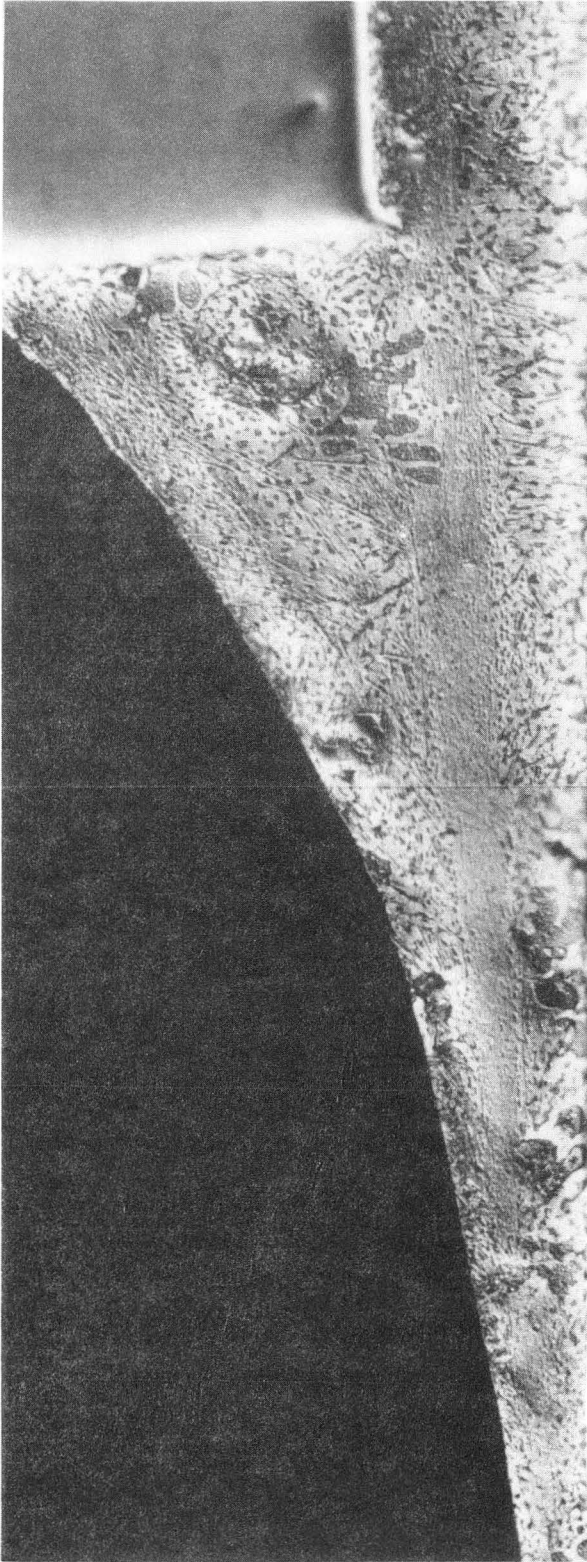
XBB 914-2482

25μm



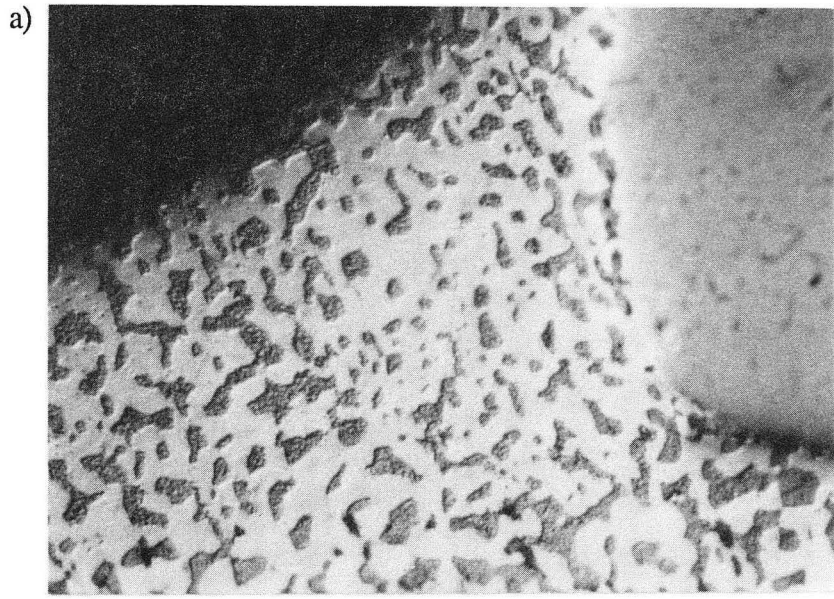
XBB 914-2481

Figure 3

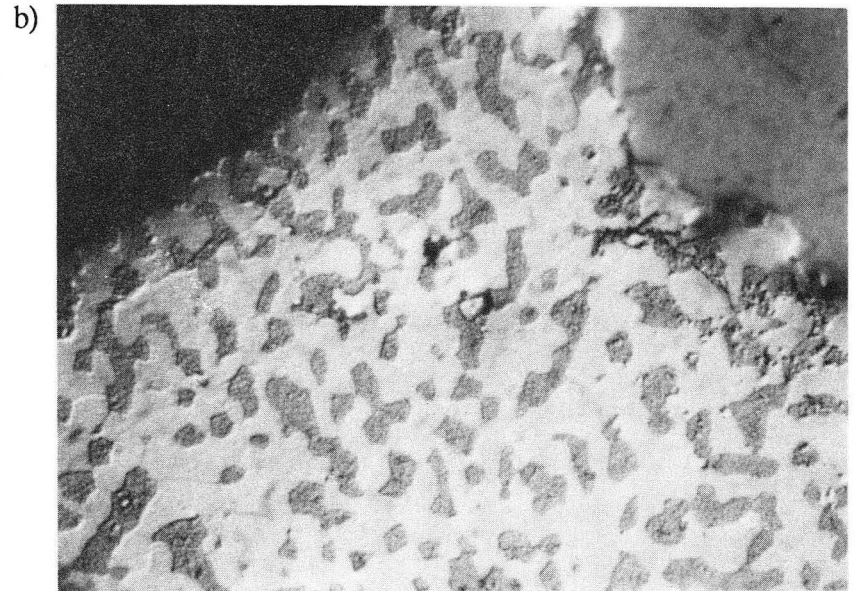


XBB 911-245

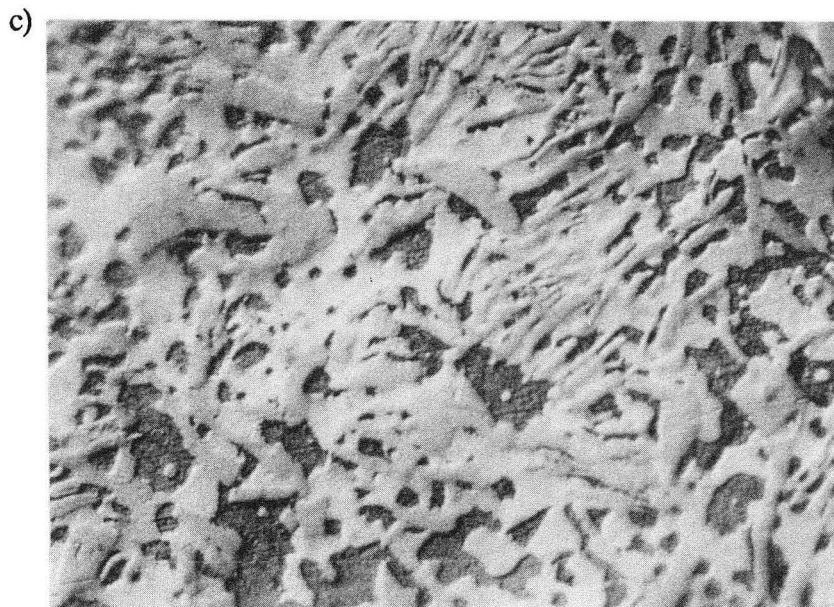
Figure 4



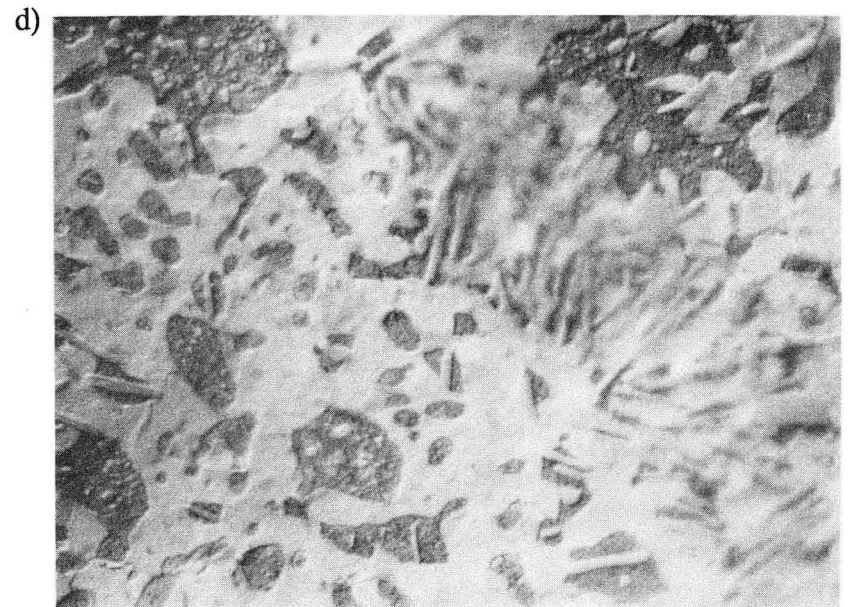
XBB 914-2534



XBB 914-2537



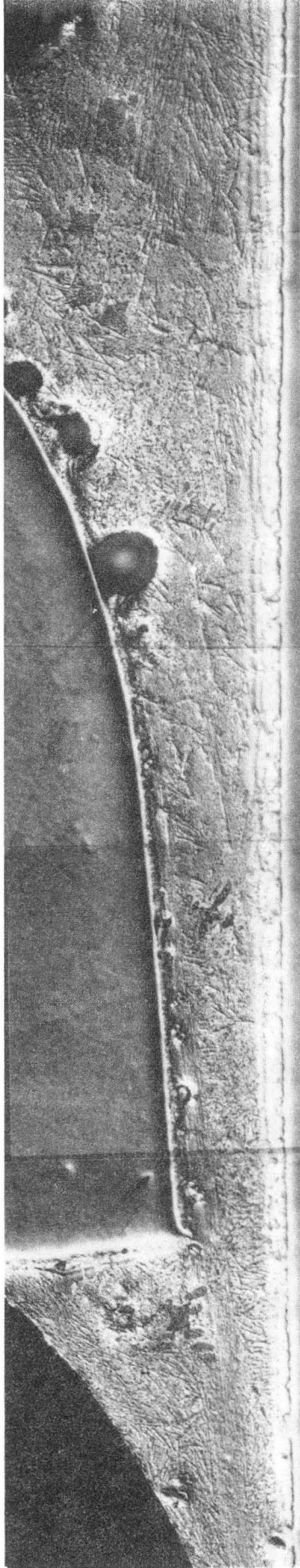
XBB 914-2536



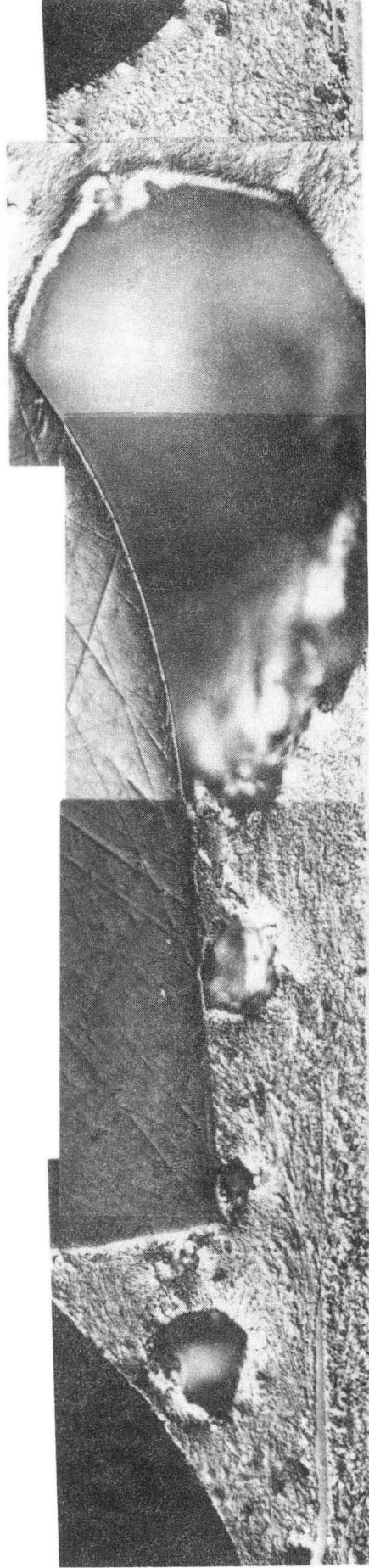
XBB 914-2539

Figure 5

25µm



50μm



50μm

XBB 911-246

Figure 6

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