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Developing a Test Method to Characterise Internal Stress in Tin Coatings: Phase 2

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Developing a Test Method to Characterise Internal Stress in Tin Coatings: Phase 2

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Division of Engineering and Process Control

ABSTRACT

This report highlights the progress made in Phase 2 of a Studio Project aimed at formulating a practical test method for measuring the internal stresses in the coatings of electronic components based on the XRD technique. The work is driven by the necessity to be able to assess the potential of whisker growth (generated from internal stresses) from new lead-free finishes for these components. Such finishes are being explored in the light of the forthcoming ban on the use of lead.

XRD measurement did show some correlation of measured compressive residual stress on coatings with whisker growth. Three coatings with high compressive residual stress also exhibited whiskering. However, the coatings evaluated were all at least twice as thick as penetration depth of the XRD measurement system, and the measured compressive residual stress values did not start to increase until after the formation of tin whiskers. However, the XRD method did provide some correlation between residual stress in the coatings and the extent of whisker growth.

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by Dr Jerry Benson, Director, Division of Engineering & Process Control

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1 INTRODUCTION

The electronics industry needs to adopt new component finishes that are lead-free to meet the regulatory requirements of the European ban on the use of lead in electronics from 1st July 2006. The European move has already triggered a global move to remove lead from electronics resulting in significant commercial pressure for UK industry. A tin-lead alloy has traditionally been used as a finish on component terminations and clearly, there is a need to find new alternatives. A finish that has received serious consideration is pure tin. This finish has a number of advantages from both component manufacturing and circuit assembly point of view. However, pure tin finishes can suffer from the spontaneous growth of single crystal whiskers that can cause short circuits.

Although pure tin as a component finish is finding wider acceptance, there remains significant scepticism over full implementation due to the issue of whiskers. Advances in plating chemistries have significantly reduced the propensity of tin coatings to whisker, but whiskering still remains an issue for many end-users. Mechanisms of whisker growth have been proposed, and critical to these is the internal stress within the coatings. In a wide range of applications X-ray diffraction (XRD) has successfully been used to measure residual stress within coatings.

In this project a two phase approach has been adopted. Phase 1 of the work (Reference 1) reported the preferred test ageing conditions, the preferred locations for analysis, and the preferred methodology, thereby allowing a close definition of the work in Phase 2 of the project. Although the results highlighted a good correlation between whisker growth and residual stress measurements, the latter did not predict the occurrence of whisker growth in the single type of coating studied. Phase II of the project aimed to expand the earlier work to a range of finishes from several different plating chemistry suppliers, and to evaluate further the potential for the XRD technique to be used for predicting whisker growth. This project phase also aimed to formulate a practical test method based on the XRD technique for measuring stress.

2 METHODOLOGY

2.1 SAMPLE DETAILS

Samples for Phase 2 of the project were manufactured from stamped Olin 194 lead-frames over-moulded and supplied by CML Microcircuits (UK) Ltd. These samples were plated by three different partners, with up to three different coatings of pure tin chemistry from each partner. Finishes A, B and C were from partner A, finishes D, E and F were from partner B, and finishes G and H were from partner C. in each case the coating thickness was checked by sectioning samples of the lead-frame (see section 3.1).

After plating, the lead-frames were taken and manufactured into SOIC components by CML Microcircuits (UK) Ltd. The components were cropped from the surrounding lead-frame and the leads bent into a gull-wing format. The remainder lead-frame after component removal was used for the majority of the work in this phase, as these were known from previous projects (Reference 2) to have a greater propensity to whiskering than the components themselves. An example of a cropped lead-frame is shown, against a brown background, in Figure 1.

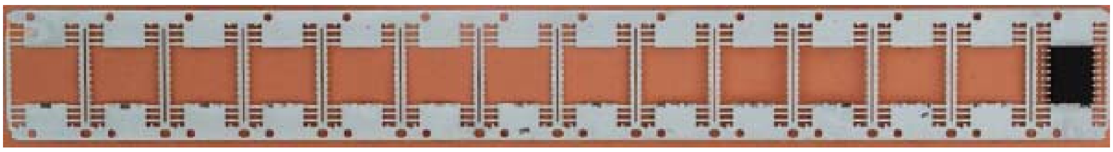

Figure 1 : Example remainder lead-frame with component positioned at right-hand end to show position prior to bend and crop

2.2 SAMPLE AGEING

Both the components and remainder lead-frames were aged at NPL at 50^{o}C in an air circulation oven. After 0, 1, 3 and 6 months, samples were removed for examination using X-ray diffraction (XRD) and scanning electron microscopy (SEM) as detailed below. Two samples were checked for each condition and are referred to subsequently as A and B.

2.3 SCANNING ELECTRON MICROSCOPY INSPECTION

After ageing at the above time intervals, samples were cut from the remainder lead-frame strip and inspected in a Camscan MX2500 scanning electron microscope. As viewed in Figure 1, three areas on the vertical sides of the lead-frame were inspected. This is detailed in Figure 2. In both cases, digital images were acquired at 1000X and 5500X for comparison.

During the SEM inspection, a whisker classification was applied to the side and top of each sample. The classification, which had previously been used successfully by one of the project partners (Reference 3), was as follows:

Class 0 - no observable whisker growth
Class 1 - infrequent, short length (<5µm)
Class 2 - infrequent, moderate length (5-25µm)
Class 3 - more frequent, short or moderate length (<25µm)
Class 4 - long (>25µm), classic whisker shape, 3 - 4µm diameter.

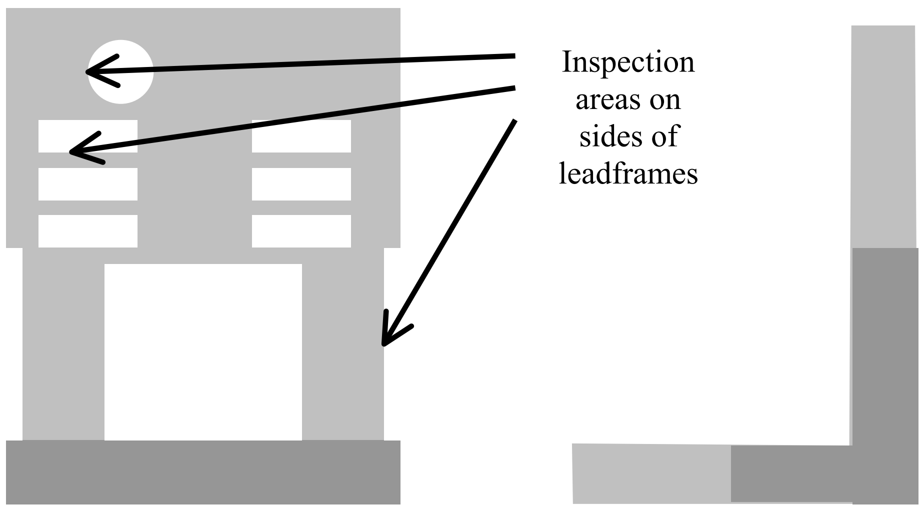

Figure 2: Inspection areas on side surfaces of lead-frame samples. The side elevation shows that the lead-frame was bent at 90° to present the side-wall surfaces to the electron beam more readily.

2.4 RESIDUAL STRESS MEASUREMENTS

As in Phase I of the project, the residual stress in Phase II was determined using a Siemens D500 diffractometer using Cr-Kα radiation and a position sensitive detector. For each sample measurements were performed on the side of five lead frames, which were stacked together on edge and held vertically, to increase the irradiated surface area. The sample was orientated such that its longest dimension was perpendicular to the direction of the X-ray beam.

A full 2θ scan was initially conducted on the plating to establish a suitable high angle peak on which to perform the residual stress measurement, and the resultant diffraction peak was from the [312] family of planes which could be located at a 2-theta angle between 143° and 144°.

For each sample, the residual stress was measured using omega geometry. The $\sin^2\psi$ method and the Bruker STRESS program were used to analyse the diffraction data. The residual stress measurements were performed in accordance with the NPL Measurement Good Practice Guide No. 52 – Determination of Residual Stresses by X-ray Diffraction (Reference 3). Typical measurement parameters are presented in Table 1. The diffraction peak position was identified using a psuedo-voigt fit peak fitting method. This peak position was then plotted against the $\sin^2\psi$ value. Finally, the residual stress and shear stress present in the tin plating were established by fitting a straight line through the data points (if no shear stresses were present); or by fitting an ellipse through the data points (if shear stresses were present).

Table 1: Typical measurement parameters used in Phase II

Parameter	**Values**
Optical apertures	1°-1°-7°-7°
Start 2θ, °	143
Stop 2θ, °	145.468
ψ tilts	±40
Number of tilts	9
Step size, °	0.02023
Scan speed, s/step	0.5
ARX	1
E, MPa	41400
ν	0.33

3 RESULTS

3.1 MICRO-SECTIONING

The plating thickness of the samples was checked using micro-sectioning techniques and the results are shown in Figure 3 below. Plating thicknesses of the samples were in the range of 4 to 10 μm and these represented standard commercial thicknesses.

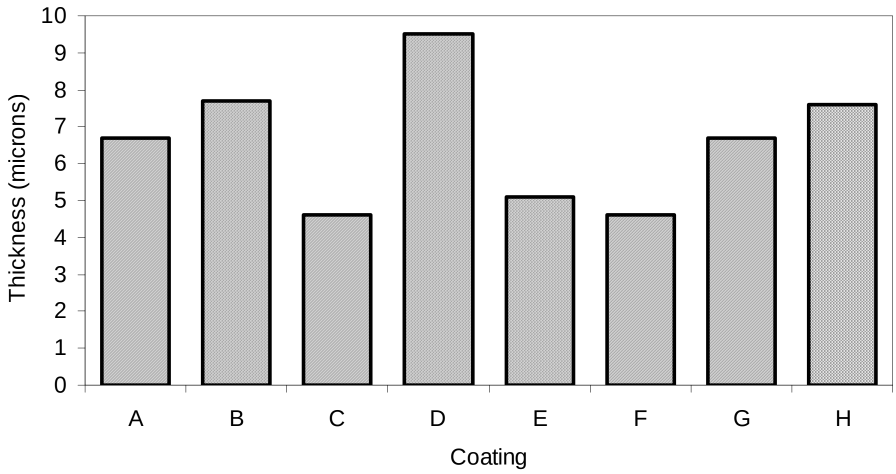

Figure 3: Average coating thickness for the eight plated samples

3.2 COMPARISON OF SAMPLES AFTER AGEING AT 50°C

Figure 4 shows the average whisker classification for the sides of samples aged at 50°C. The classification is an average based on SEM inspection of the areas detailed above.

The finishes can be separated into two groups. Finishes B, C, D and F showed little or no inclination to whisker during the test period. Finishes A, E, G and H did whisker but whiskering was restricted to class 2 without any long (>20μm) whiskers forming or the density of whiskers increasing.

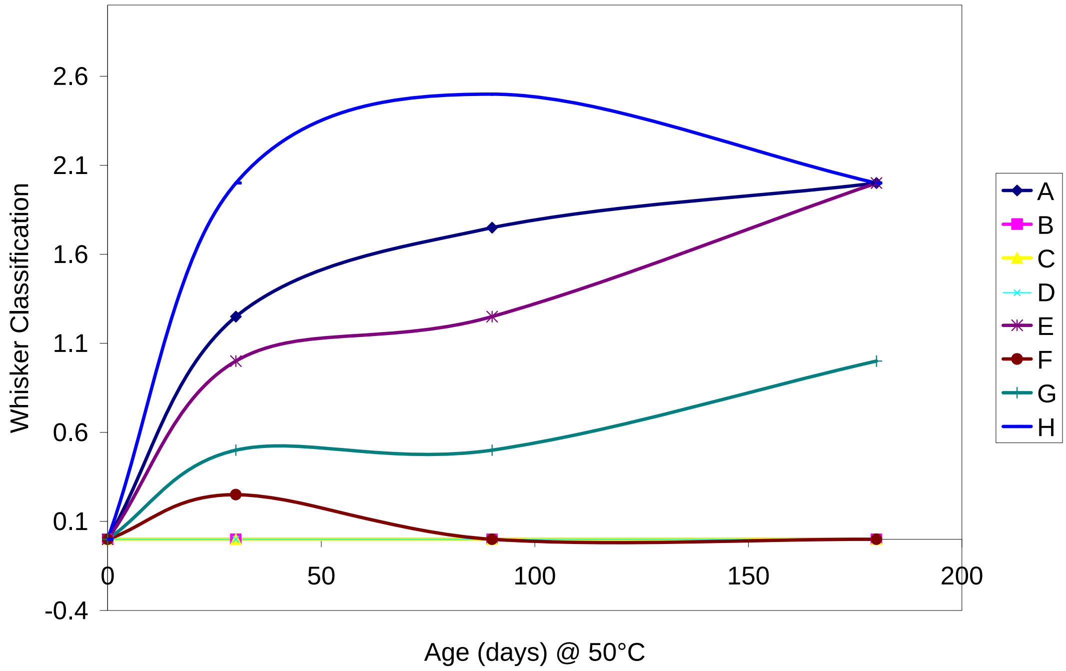

Figure 4: Comparison of average whisker classification for 8 tin finishes aged at 50^{o}C

3.3 RESIDUAL STRESS RESULTS

The Phase II samples consisted of 8 sets with measurements being conducted at time = 0, 1, 3 and 6 months. The results are presented in Figure 5.

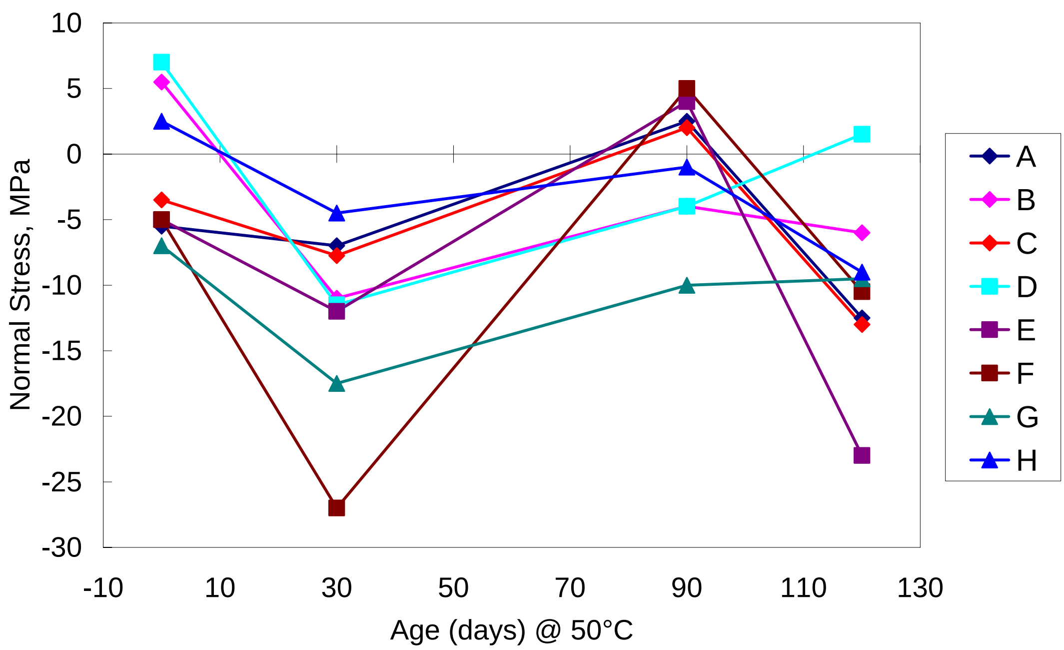

Figure 5: Average normal stress results

Initial indications from the XRD measurements were that all the samples were showing signs of the evolution of increasing compressive residual stresses after an ageing time of 1 month. The measurements made after 3 months of ageing would appear to show the residual stresses in all of the samples relaxing and tending towards zero. However, the measurements made after 6 months in some cases contradict this observation. To make the changes in the residual stress in the plating clearer, the data were re-plotted excluding the 3-month data. On examination of this plot, it was clear that the datasets could be split into two groups. The first group comprises samples which demonstrate a trend for increasing compressive stress with time in the plating, whilst the other group has samples showing an initial increase in compressive stress, but after further ageing, a relaxation. Figures 6 and 7 show the results from these two groups respectively. The sample groupings as described above are presented in Table 2.

Table 2: Behaviour grouping of the Phase II samples

Sample ID	Group 1 Increasing compressive stress	Group 2 Relaxing with time
A	×	
B		×
C	×	
D		×
E	×	
F		×
G		×
H	×	

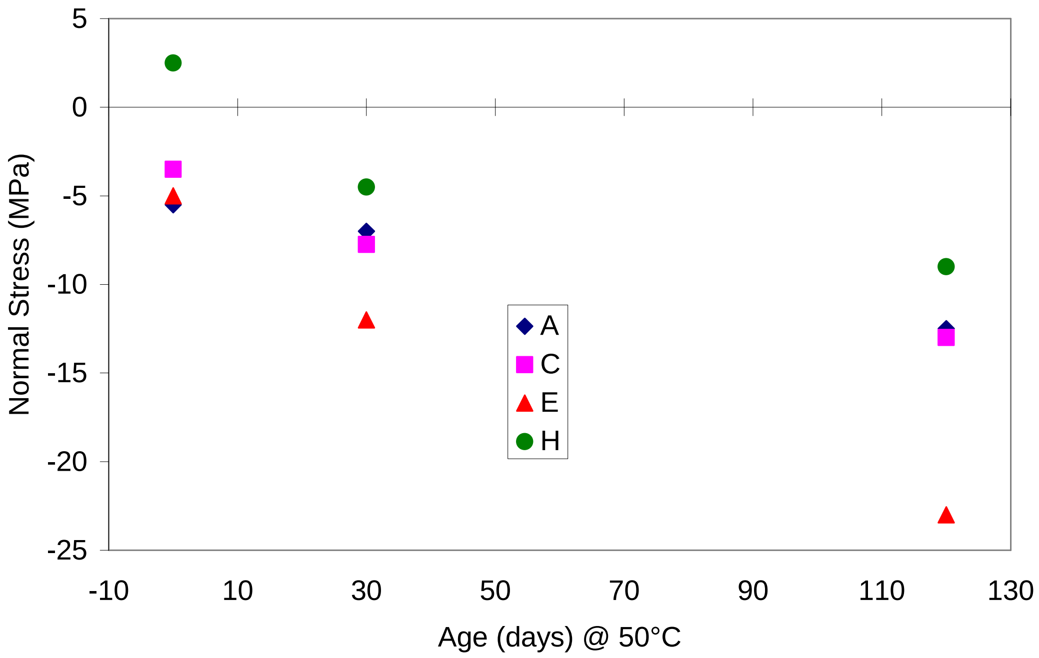

Figure 6: Samples which show increasing levels of compressive residual stress with time

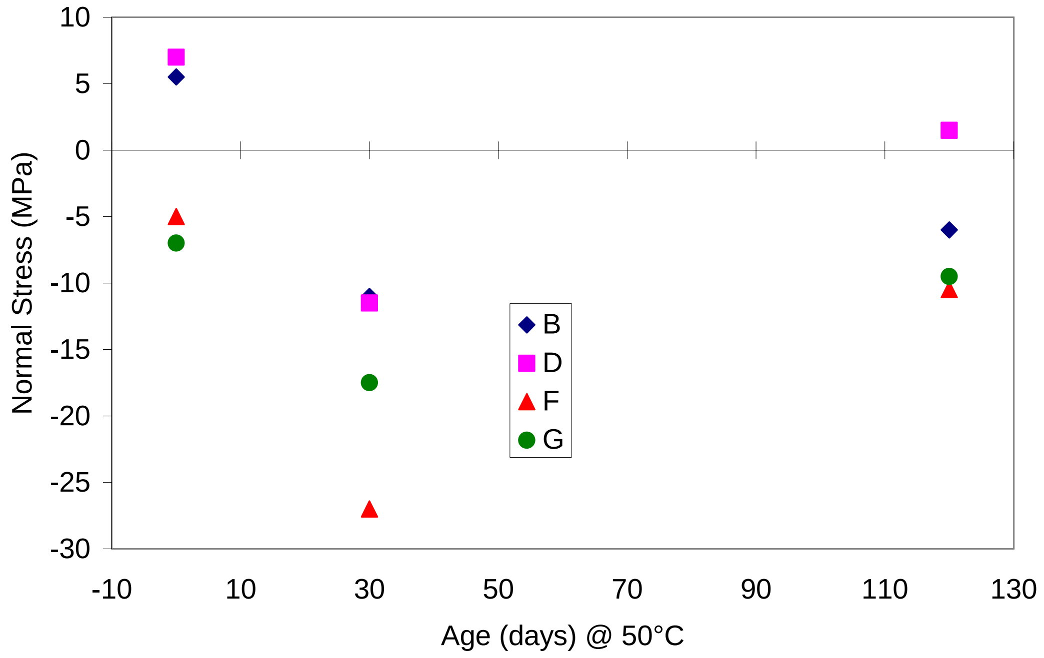

Figure 7: Samples which show an initial increase in residual stress, but this then relaxes with time

4 DISCUSSION

4.1 SEM RESULTS

Figures 8 to 11 present representative images of samples aged over 6 months at 50°C. These finishes B, C, D and F, did not show any significant whiskering, and are without any apparent major surface features.

Figures 12 to 15 illustrate the images of the surface finishes that did whisker i.e., finishes A, E, G and H. These finishes showed similar levels of whiskering although the form of the whiskering is often different. Finishes A and H showed low aspect ratio whiskers, which were tapered towards the tip. Finish E had whiskers of similar size, although the whiskers were of constant thickness throughout their length. Finish G formed much smaller whiskers of differing shapes.

With all samples, little additional whiskering was noted after 3 months of ageing at 50°C.

No sample exhibited whiskers longer than 20µm after 6 months, with few samples showing whiskers greater than 10µm in length.

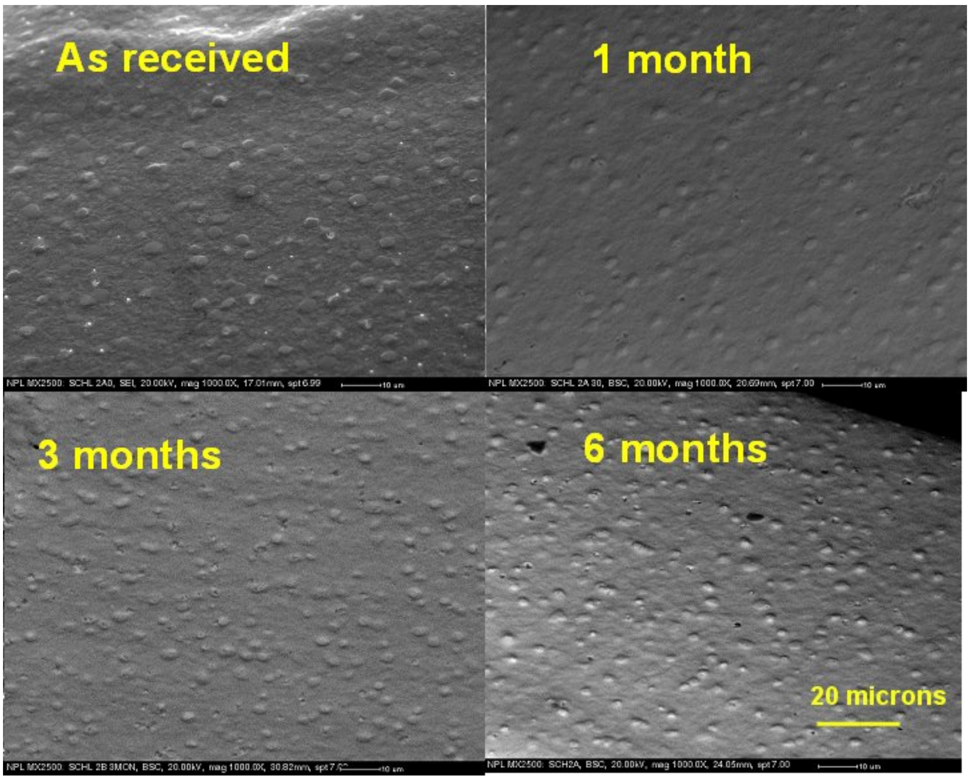

Figure 8: Comparison of SEM images of aged samples of finish B

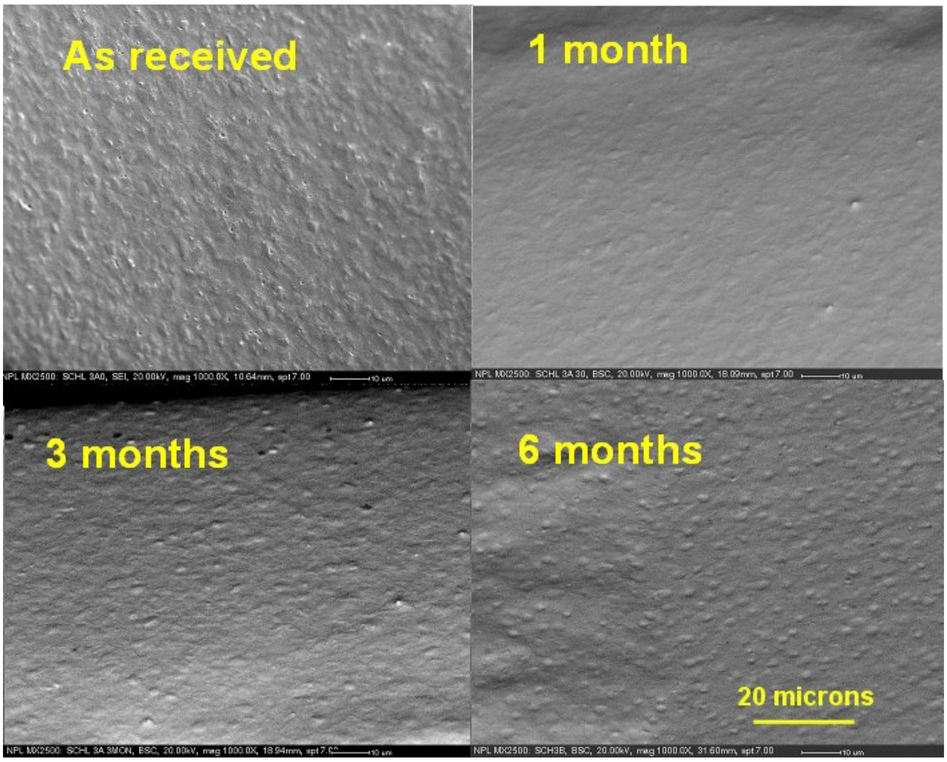

Figure 9: Comparison of SEM images of aged samples of finish C

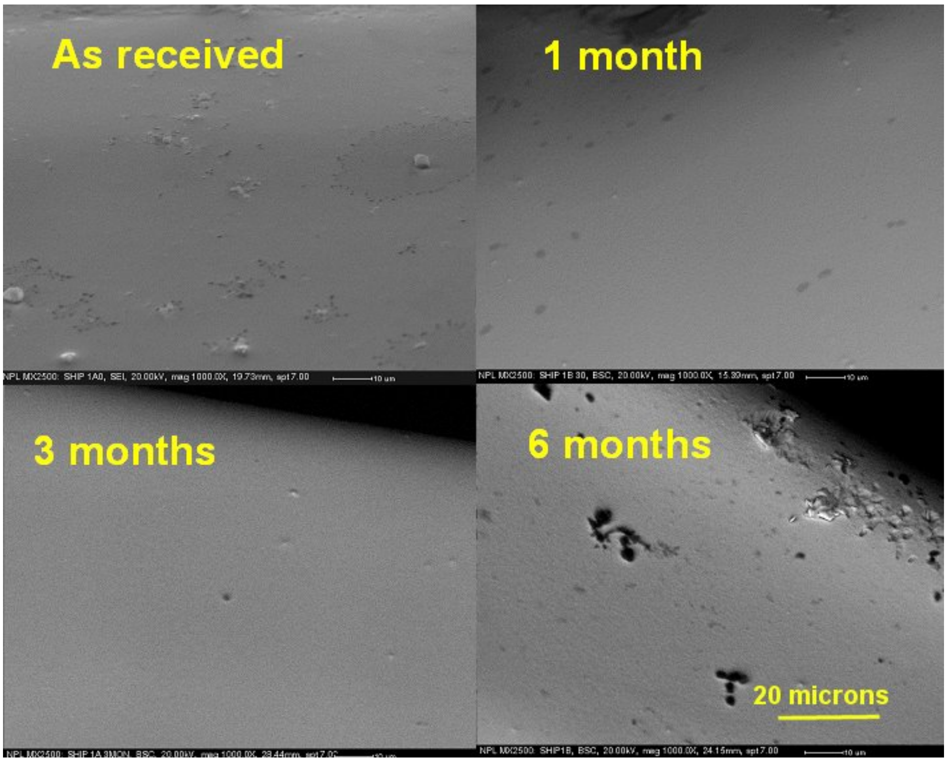

Figure 10: Comparison of SEM images of aged samples of finish D

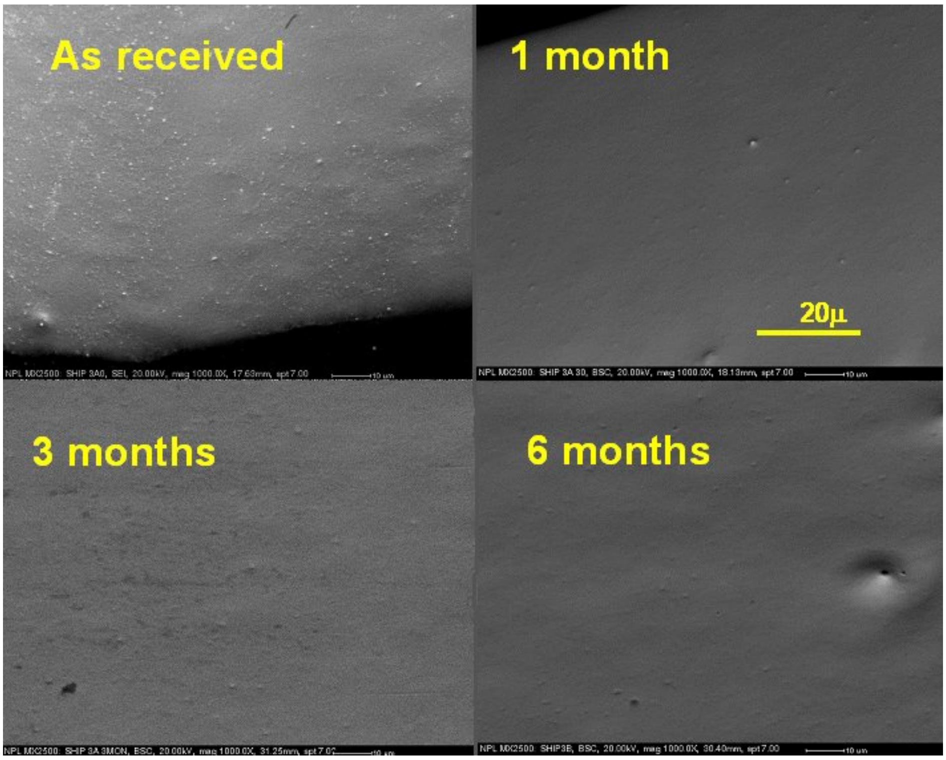

Figure 11: Comparison of SEM images of aged samples of finish F

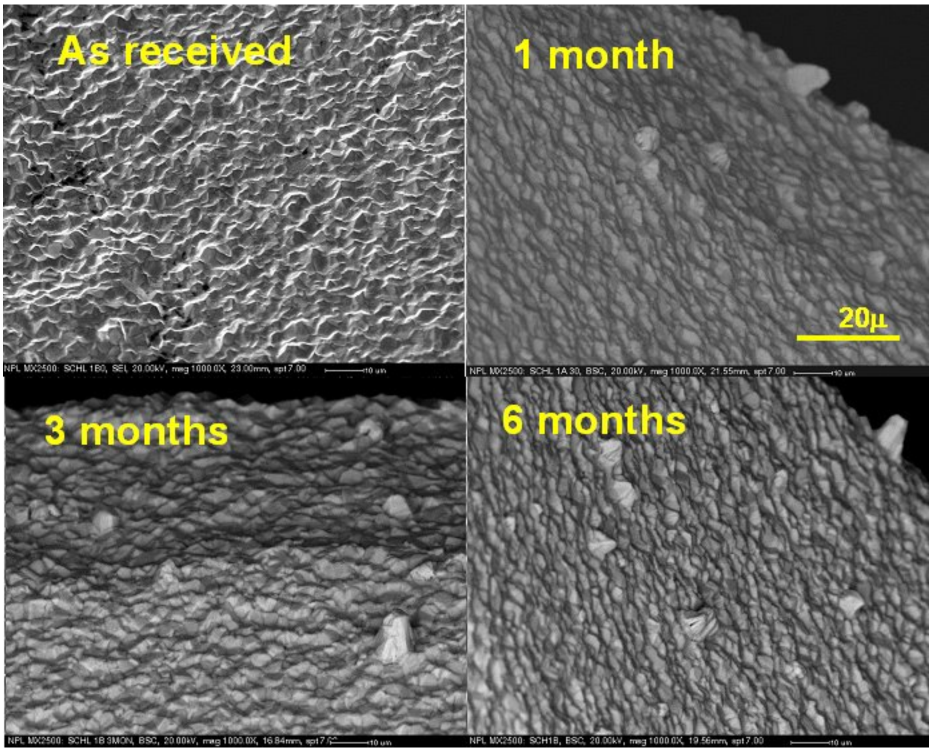

Figure 12: Comparison of SEM images of aged samples of finish A

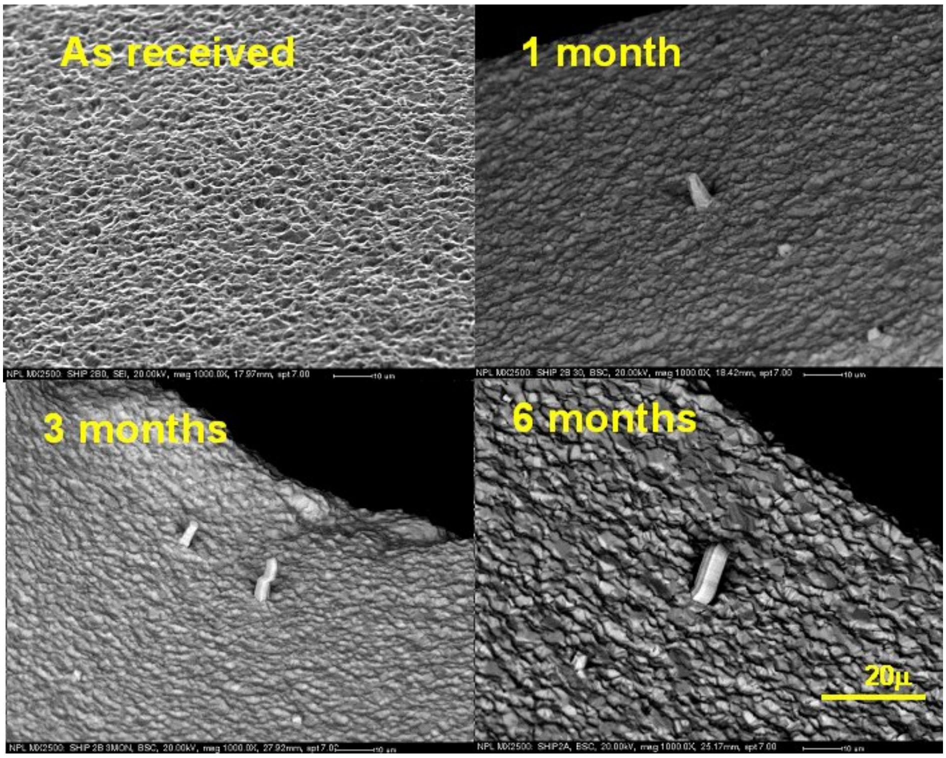

Figure 13: Comparison of SEM images of aged samples of finish E

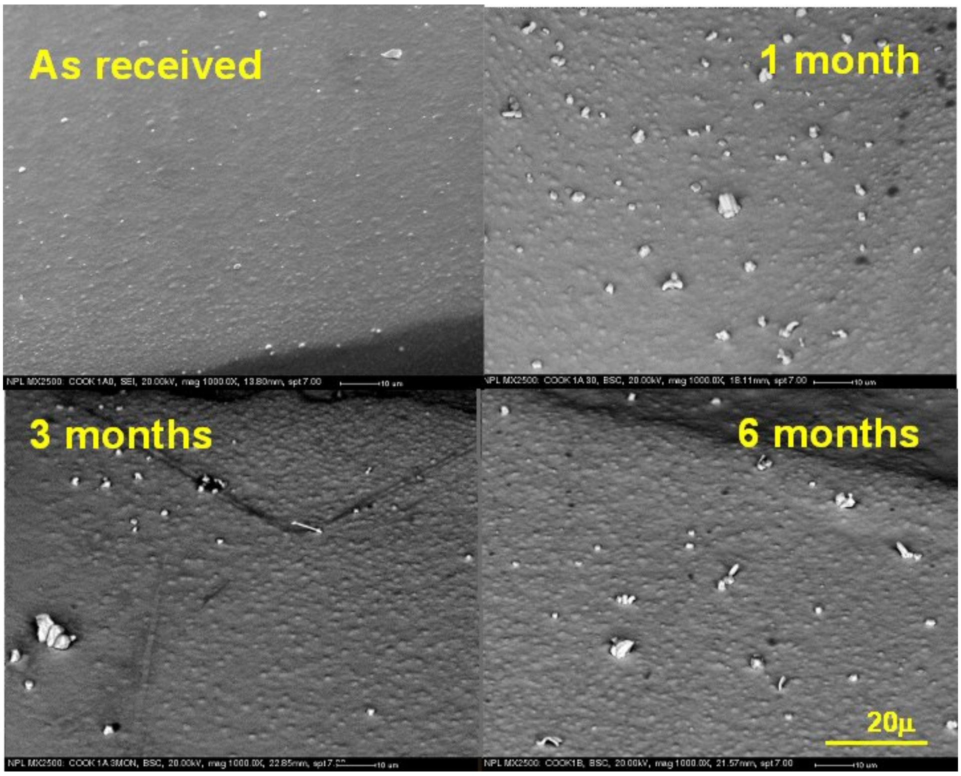

Figure 14: Comparison of SEM images of aged samples of finish G

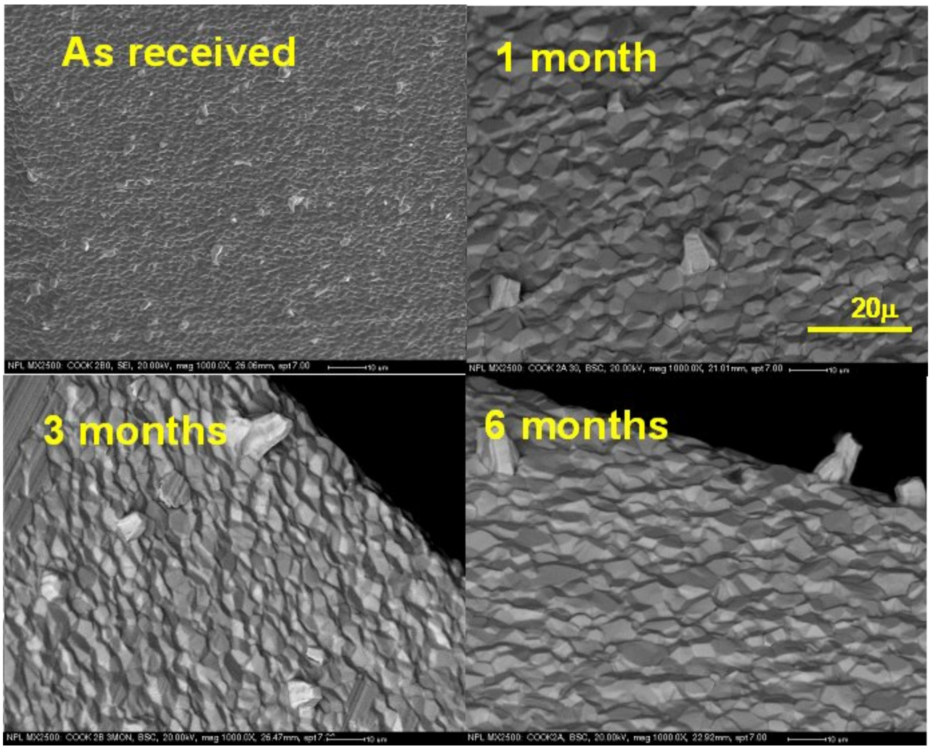

Figure 15: Comparison of SEM images of aged samples of finish H

4.2 XRD RESULTS

Initially the diffraction data from all the platings indicated that there was an increasing level of compressive residual stress developing with time. Some of the samples continued to show increasing levels of stress, whilst in others the stress relaxes. This apparent relaxation could be a true indication of the stress in the plating, or it could be due to the stress field varying with ageing and coating thickness. It is useful to compare the range in plating thickness and the penetration depth of the X-rays. If the residual stress is developing at the interface between the substrate and the plating then it is important that the measurements are taken from as close to this area as possible. Figure 16 shows the penetration depth of Cr-Kα and Cu-Kα radiation as a function of the ψ tilt. The maximum penetration of the Cr-Kα radiation in tin is calculated to be in the order of 2μm. If the plating thickness is much greater than this then the measurement will be made at some distance from the interface, which may reduce the sensitivity of these measurements to increases in the residual stress. Conversely, if the residual stress increases as we move away from the interface then this too will affect the results as the measurement depth is essentially fixed.

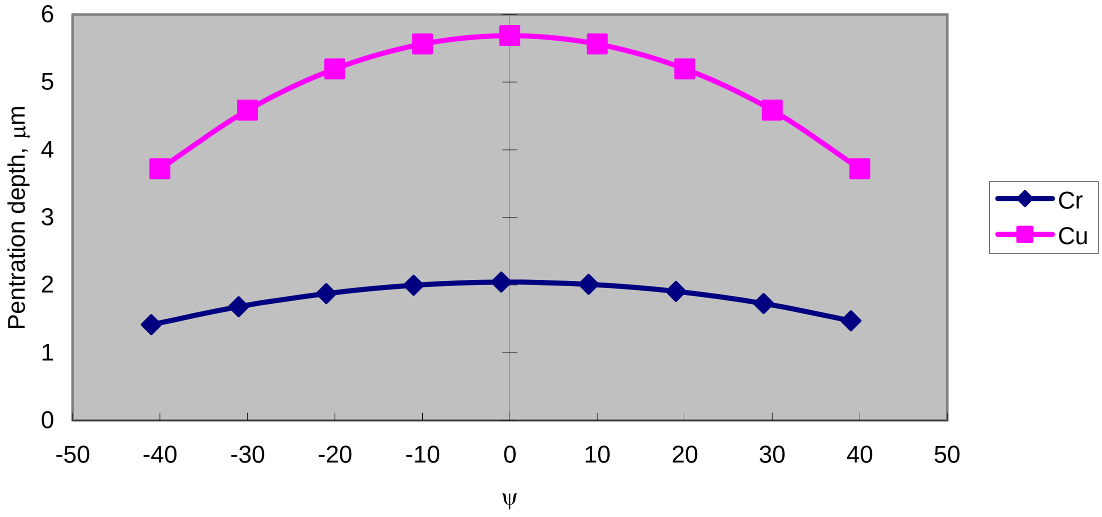

Figure 16: Penetration depth for 90% contribution to the diffracted beam vs. ψ for tin with an assumed density of 7.310 g/cm^3

4.3 COMPARISON OF SEM INSPECTIONS AND XRD MEASUREMENTS

Figure 17 presents a comparison of the results for whisker classification and residual stress for the low whisker density samples aged at 50°C. For samples B, C and D, there was little compressive (negative) residual stress build-up and no whiskering after 6 months. With sample F, some compressive residual stress is noted initially, and indeed, as can be seen in Figure 4, some samples of this finish did exhibit a low level of whiskering initially.

Figure 18 provides a comparison of the results for whisker classification and residual stress for the higher whisker density samples aged at 50°C. Again, there was little evidence of high levels of compressive residual stress initially, with measured levels for most samples at around –10 MPa. Finish G had the highest initial compressive residual stress but was actually the sample which whiskered least of these four finishes. However, at the end of the 6 month evaluation period, compressive residual stress values in the samples can be seen to be increasing significantly.

Thus, there is a reasonable inverse correlation towards the end of the measurement period, with the degree of compressive residual stress increasing for higher whisker growth samples. This is in agreement with the most popular theory of whisker development in which the formation of intermetallics at the Sn/Cu interface causes stress in adjacent tin grains, leading to whisker formation (References 4 and 5). ***An important observation from these results is that the residual stress measurement does not predict whisker growth, but does reflect the level of formation.***

As discussed in Section 4.2 above, this delayed detection of stress may be associated with the thickness of the plating. Compressive residual stress may be being generated due to intermetallic growth, sufficient to generate whiskering, but this may be too deep in the plating to be measured using X-ray diffraction. It is estimated that the measurement depth of the X-ray technique is limited to 1-2µm, whilst the plating thickness on these samples is around 4-10µm. Thus the observed increase in compressive residual stress will only be detected when intermetallic growth has occurred to a point where the resultant stress field, that grows from the substrate interface in the coating, overlaps the detection depth of the XRD technique.

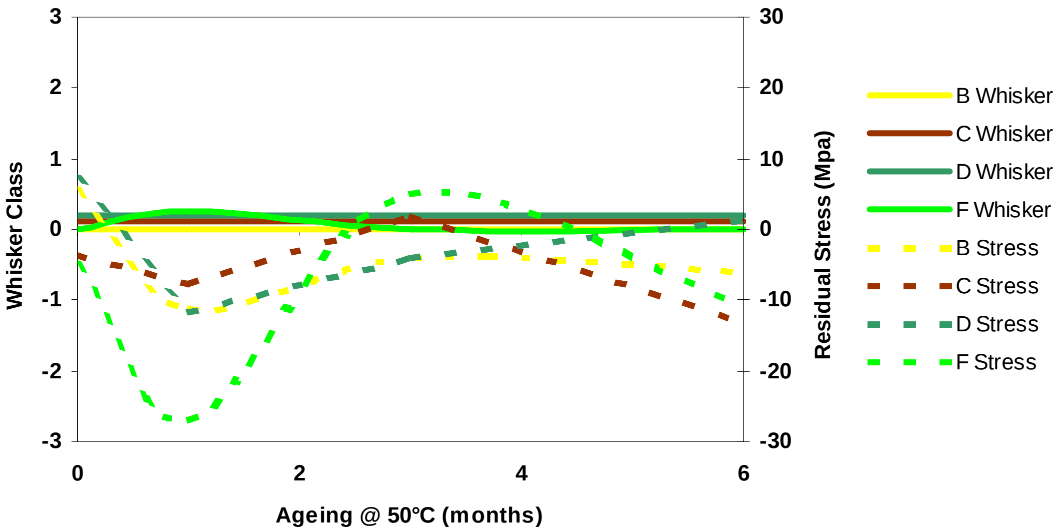

Figure 17: Comparison of whiskering classifications with residual stress for 6 months of ageing at 50°C for low whiskering samples (B, C, D, F)

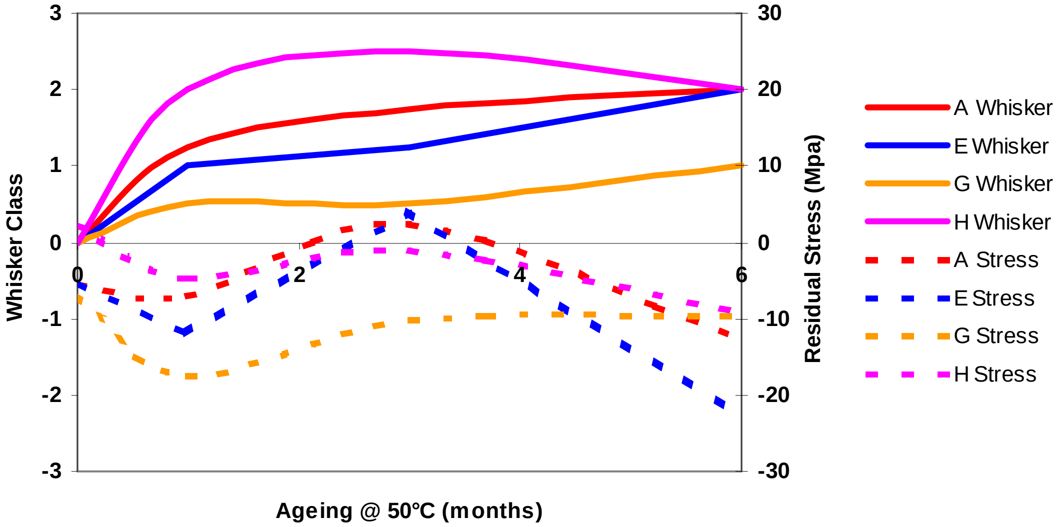

Figure 18: Comparison of whiskering classifications with residual stress for 6 months of ageing at 50°C for high whiskering samples (A, E, G, H)

5 CONCLUSIONS

None of the samples exhibited significant whiskering. In the six months of ageing at $50^{O}C$ none of the samples produced whiskers greater than 20 µm in length, and most were characteristically half this size, except for the untypical areas in very thin coatings. It is thought that whiskers of this size will not cause any problems in the vast majority of electronics applications. Surface mount components with the largest numbers of lead-ins will typically have a minimum pitch of 400 µm i.e. a 200 µm gap. Hence whiskers 20 µm in length do not pose a direct threat for short circuits. Moreover, half the samples exhibited no whiskering at all.

The XRD technique is not suitable for predicting potential whisker growth. The XRD method did provide some correlation between residual stress in the coatings and the extent of whisker growth. However, the penetration depth of the X-rays is considerably less that the thickness of the coatings, by at least a factor of two in this work, and this is significant since the consensus view is that internal stress is generated at the coating-substrate interface with intermetallic growth. In addition, the measured compressive residual stress did not start to increase until after the formation of the tin whiskers.

6 ACKNOWLEDGEMENTS

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7 REFERENCES

1. Developing a Test Method to Characterise Internal Stress in Tin Coatings, Phase 1: Martin Wickham, Tony Fry and Chris Hunt: NPL Report MATC(A) 148, (October 2003)
2. Processability of Lead-free Component Termination Materials: Martin Wickham, Deborah Lea, Jaspal Nottay and Chris Hunt: NPL Report MATC(A)47, August 2001
3. Whisker Evaluation of Tin-Plated Logic Components Leads: Douglas W. Romm, Donald C. Abbott, Stu Grenney and Muhammad Khan: Texas Instruments Application Report SZZA037A (February 2003)
4. M.E. Fitzpatrick, A.T. Fry, P. Holdway, F.A. Kandil, J. Shackleton and L. Suominen: *NPL Good Practice Guide No. 52: Determination of Residual Stresses by X-ray Diffraction.* March 2002
5. An Empirical Study Into Whisker-Growth of Tin and Tin Alloy Electrodeposits: Keith Whitlaw and Jeff Crosby: Shipley Europe Ltd., Coventry, England
6. Tin Whiskers: Attributes and Mitigation: Jay A, Brusse, Gary J. Ewell and Jocelyn P. Siplon: QSS Group, Incorporated, NASA Goddard Flight Centre

8 ANNEX 1

Glossary of definitions for X-ray diffraction:

Cr-Kα - Characteristic X-ray for a Cr anode tube, with a wavelength of 2.290 Å [1].

θ - The Bragg angle, this is the angle between the diffracting lattice planes and the incident beam [2].

2θ - The diffraction angle, this is the angle between the incident (transmitted) and diffracted X-ray beams [2].

d - Inter-planar spacing (d-spacing) - the perpendicular distance between adjacent parallel crystallographic planes [1].

ψ - Angle between the normal of the sample and the normal of the diffracting planes (bisecting the incident and the diffracted beams) [2].

1 M.E. Fitzpatrick, A.T. Fry, P. Holdway, F.A. Kandil, J. Shackleton and L. Suominen: *NPL Good Practice Guide No. 52: Determination of Residual Stresses by X-ray Diffraction.* March 2002

2 prTC 138 WI 097:2001, Non destructive testing – Test method for residual stress analysis by X-ray diffraction