Developing a Test Method to Characterise Internal Stress in Tin Coatings: Phase 1

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October 2003

NPL Report MATC(A)148

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ABSTRACT

This report highlights the progress made in Phase 1 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. The work has identified 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 have highlighted a good correlation between whisker growth and residual stress measurements, the later could not be used to predict the occurrence of whisker growth, only to characterise its extent.

ISSN 1473 2734

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NPL, by Dr C Lea, Head, NPL Materials Centre

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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 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 shorts.

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. X-ray diffraction (XRD) has successfully been used to measure residual stress within coatings. This project plans to study this further and formulate a test method. Indeed, XRD analysis of samples from previous NPL Studio Project work demonstrated that tensile stresses were present in coatings that exhibited whisker growth, whilst coatings that produced no whiskers contained zero stress levels.

The aim of this project was to formulate a practical test method based on the XRD technique for measuring stress.

In this project a two phase approach has been adopted. This report covers phase 1 in which just two sample types have been investigated, one with a known high propensity and one with a known low propensity for tin whisker growth. The XRD data have been correlated with direct assessments of whisker growth allowing an evaluation of the potential for the XRD technique to be used for predicting whisker growth. In phase 2, a wider range of finishes will be measured leading to the generation of a practical test method.

2 METHODOLOGY

2.1 SAMPLE DETAILS

Samples for phase 1 of the project were manufactured from stamped Olin 194 lead-frames supplied by CML Microcircuits (UK) Ltd. These samples were plated by one of the partners with two different thicknesses of the same redundant, pure Sn chemistry, nominally 2 and 10µm. The coating thickness was checked by sectioning samples of the lead-frame (see Results - Microsectioning). The chemistry used was a redundant formulation known to promote tin whiskering. The two thicknesses were designed to give two different levels of whiskering, the thinner coating expected to whisker more than the thicker coating. These coatings were designated high and low propensity respectively.

After plating, the lead-frames were taken and manufactured into SOIC components by CML Microcircuits (UK) Ltd. After over-moulding the plastic body 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 1) 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.

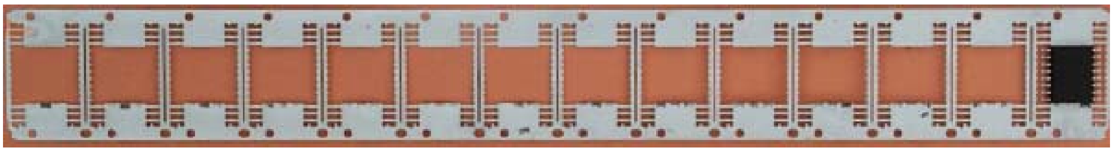


Figure 1 : Example remainder lead-frame with component positioned at right-hand end to show position prior to bend and crop

2.2 SAMPLE AGEING

Both the components and remainder lead-frame produced, were then aged at NPL at room temperature and at 50°C in an air circulation oven. After 28 days, samples were examined using X-ray diffraction and scanning electron microscopy as detailed below. Two samples were checked for each condition and are referred to subsequently as A and B. These results are presented later. However it was found that ageing at 50°C produced marginally more whiskers.

2.3 SCANNING ELECTRON MICROSCOPY INSPECTION

At 0, 28, 56 and 84 days, samples were removed from the remainder leadframe strip and inspected in a Camscan MX2500 scanning electron microscope. Two areas of the top surface of the lead-frame were inspected as detailed in

Figure 2. Three areas of the side of the lead-frame were also inspected, as detailed in Figure 3. In both cases, digital images were acquired at 1000X and 5500X for comparison.

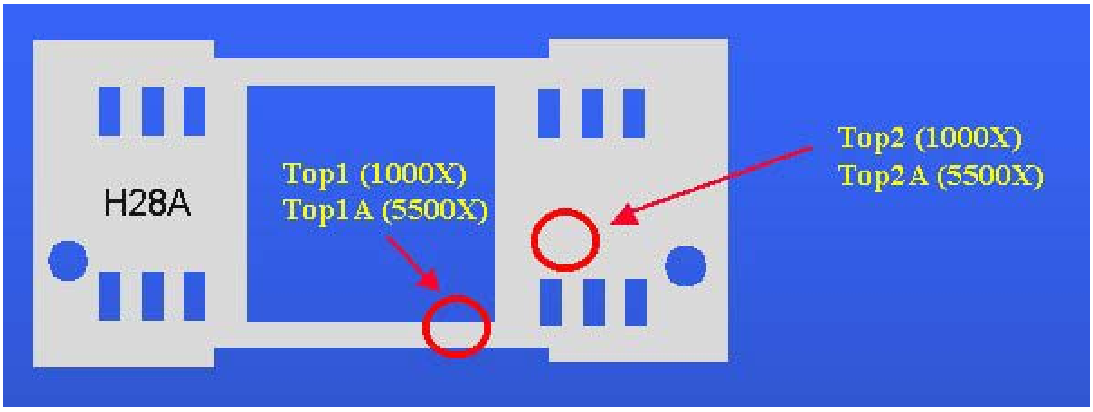


Figure 2 : Inspection areas on top surface of lead-frame samples. The lead-frame is rotated from Figure 1.

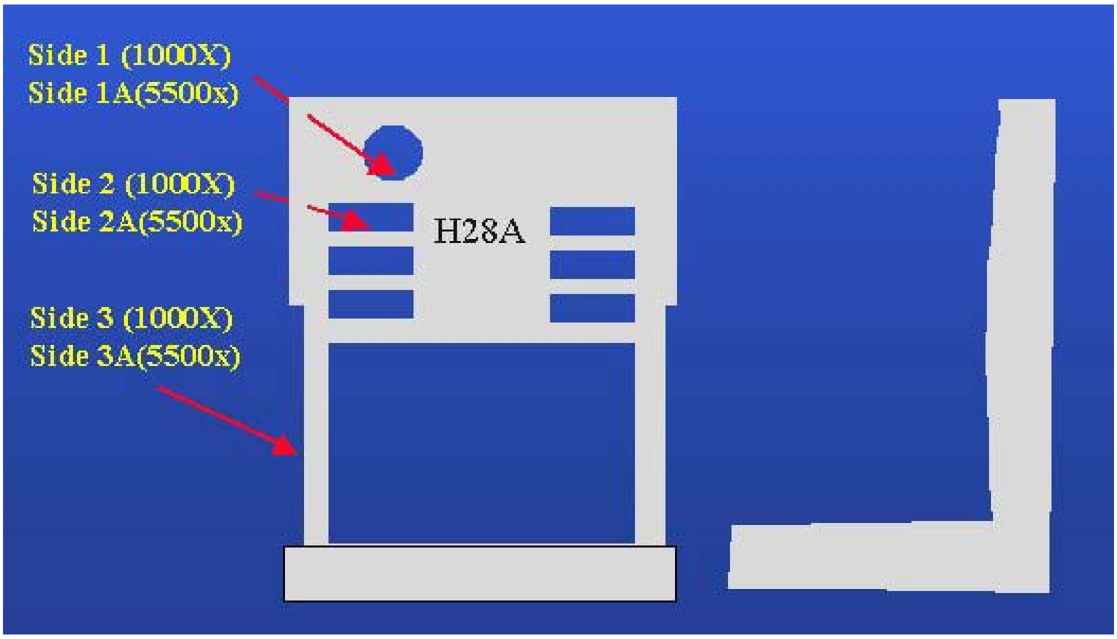


Figure 3 : Inspection areas on side surfaces of lead-frame samples. The side elevation shows that the lead-frame was bent at 90° to present the side wall surfaces to the electron beam more readily.

2.4 WHISKER INDEX CALCULATION

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 2), 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.

After the ageing period of 3 months (84 days), these classifications were used to create a whisker index for each sample using the following formula:

Index = (as received*1) + (1 month*0.75) + (2 month*0.5) + (3 months*0.25)

2.5 RESIDUAL STRESS MEASUREMENTS

The residual stress was determined using a Siemens D500 diffractometer using Cr-Kα radiation[1]. The D500 goniometer was set up in the Bragg-Brentano geometry and used a θ-2θ drive; the X-ray generator was a Kristalloflex model with a solid-state scintillation detector or position sensitive detector attached to the 2θ-drive. Measurements were performed on the top face and the side of each sample batch. For measurements on the side location five lead-frame samples were stacked together so as to present a larger area for the X-ray beam, thereby increasing the counting statistics and reducing the measurement time. For the side measurements the sample was orientated such that its longest dimension was perpendicular to the direction of the X-ray beam.

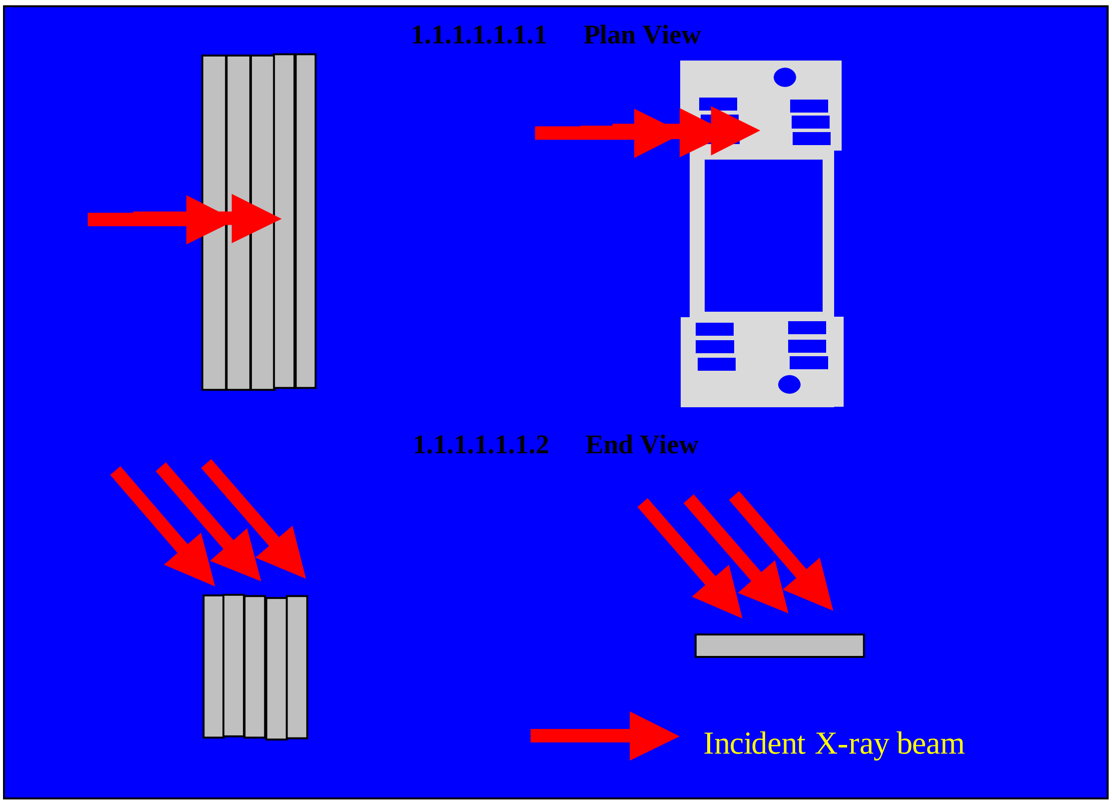


Figure 4 : Diagram of sample orientation during XRD measurement

[1] See Annex 1 for a glossary of XRD terms

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, the resultant diffraction peak being located at a 2-theta angle between 143° and 144°.

For each sample the residual stress was measured using omega geometry, the d vs $\sin^2\psi$ method and the Bruker STRESS program. 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 details are presented in Table 1. The diffraction peak position was identified using a pseudo-voigt fit. This peak position was then converted to the interplanar spacing value d, and plotted against the sin2ψ value. Finally the residual stress and shear stress present in the tin plating was established by fitting a straight line through the data points (if no shear stresses are present), or by fitting an ellipse through the data points (if shear stresses are present).

Table 1 : Typical measurement parameters used for a scintillation detector and PSD

Parameter	**Scintillation Detector**		**Position Sensitive Detector**	
	Top Face	**Side Face**	**Top Face**	**Side Face**
Optical apertures	**1°-1°-1°-0.15°**		1°-1°-7°-7°	
Start 2θ, °	**142**		143	
Stop 2θ, °	146		145.468	
ψ tilts	±40			
Number of tilts	9			
Step size, °	0.020		0.02023	
Scan speed, s/step	3		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. These plating thicknesses were estimated to be 3-6µm for the high propensity samples and 10-14µm for the low propensity samples.

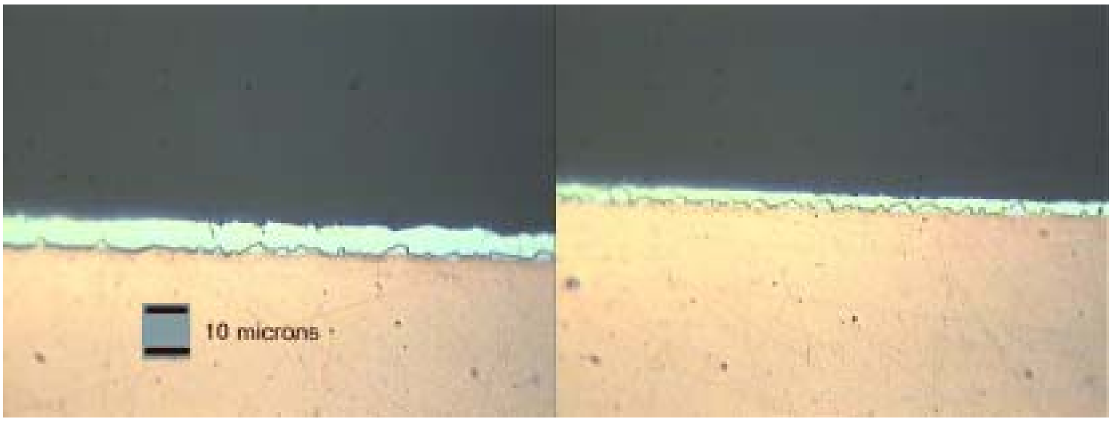


Figure 5: Microsections of high propensity samples

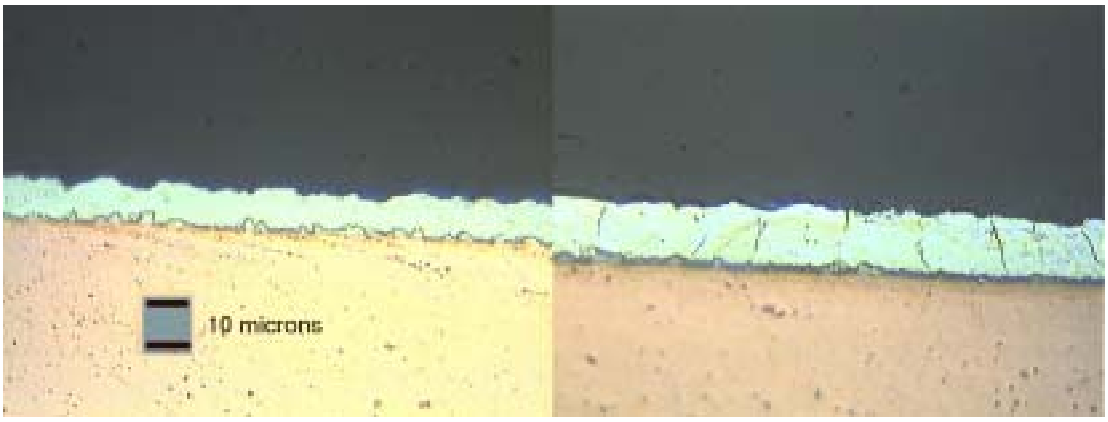


Figure 6: Microsections of low propensity samples

3.2 COMPARISON OF SAMPLES AFTER AGEING AT ROOM TEMPERATURE AND 50^{O}C

To confirm the ageing selection a comparison of whisker classifications for the two ageing conditions after 28 and 84 days was performed and the results are given in Table 2.

Table 2 : Comparison of Whisker Classifications after Ageing at Room Temperature (RT) and 50°C

Age (Days)	Temperature°C	Area	Propensity	Sample	Class
28	RT	Top	High	A	0
28	RT	Top	High	B	0
28	RT	Top	Low	A	0
28	RT	Top	Low	B	0
28	50	Top	High	A	0
28	50	Top	High	B	0
28	50	Top	Low	A	0
28	50	Top	Low	B	0
28	RT	Side	High	A	0
28	RT	Side	High	B	0
28	RT	Side	Low	A	0
28	RT	Side	Low	B	0
28	50	Side	High	A	1
28	50	Side	High	B	1
28	50	Side	Low	A	0
28	50	Side	Low	B	0
84	RT	Top	High	A	0/1
84	RT	Top	High	B	1
84	RT	Top	Low	A	1
84	RT	Top	Low	B	0
84	50	Top	High	A	0/1
84	50	Top	High	B	0/1
84	50	Top	Low	A	1/2
84	50	Top	Low	B	1
84	RT	Side	High	A	1
84	RT	Side	High	B	2
84	RT	Side	Low	A	0/1
84	RT	Side	Low	B	0/1
84	50	Side	High	A	3
84	50	Side	High	B	2
84	50	Side	Low	A	2
84	50	Side	Low	B	3

3.3 COMPARISON OF SAMPLES AFTER AGEING AT 50°C

Table 3 shows the average whisker classification for the tops and sides of samples aged at 50°C. The classification is an average based on SEM inspection of the areas detailed above.

Table 3 : Whisker Classification for Samples after Ageing at 50°C

Ageing (days)	Propensity	Sample	Position	Class	Propensity	Sample	Position	Class
0 days	High	A	Top	0	Low	A	Top	0
0 days	High	A	Side	0	Low	A	Side	0
0 days	High	B	Top	0	Low	B	Top	0
0 days	High	B	Side	0	Low	B	Side	0
28 days	High	A	Top	0	Low	A	Top	0
28 days	High	A	Side	1	Low	A	Side	0
28 days	High	B	Top	0	Low	B	Top	0
28 days	High	B	Side	1	Low	B	Side	0
56 days	High	A	Top	0	Low	A	Top	0/1
56 days	High	A	Side	2	Low	A	Side	1/2
56 days	High	B	Top	0	Low	B	Top	0/1
56 days	High	B	Side	1/2	Low	B	Side	2
84 days	High	A	Top	0/1	Low	A	Top	1/2
84 days	High	A	Side	3	Low	A	Side	2
84 days	High	B	Top	0/1	Low	B	Top	1
84 days	High	B	Side	2	Low	B	Side	3

3.4 WHISKER INDEX :

The whisker classifications in Table 3 were used to calculate whisker indexes for the samples, as shown in Table 4.

Table 4 : Sample Whisker Indexes

Age	**High Side**	**High Top**	**Low Side**	**Low Top**
0	0	0	0	0
28	1	0	0	0
56	2	0	1.75	0.5
84	2.5	0.5	2.5	1.25

3.5 RESIDUAL STRESS RESULTS :

From the d vs. $\sin^2\psi$ plots values of the residual stress and shear stress present in the tin plating were evaluated using the Bruker Stress software, and these results are presented in Figure 7 and Table 5.

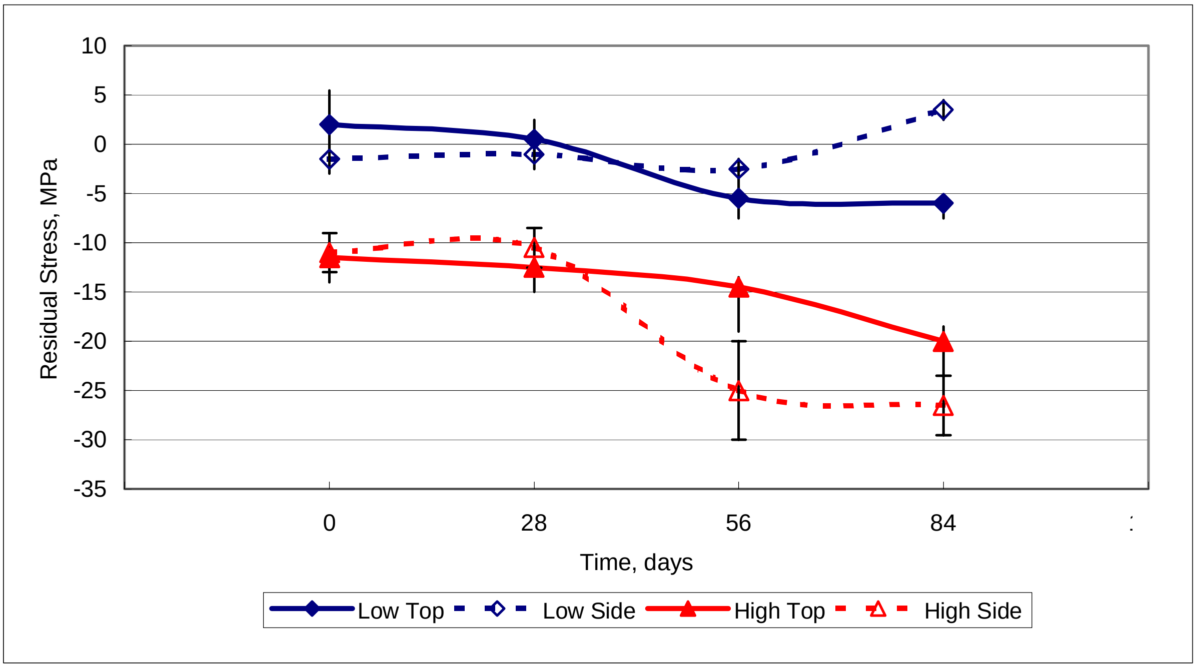


Figure 7: Average normal residual stresses for low and high propensity samples

Table 5: Normal Stresses and Shear Stresses present in the tin plating

Ageing (days)	Propensity	Sample	Position	Normal Stress, MPa	Error, ± MPa	Shear Stress, MPa	Error, ± MPa
0	High	A	Top	-12	1	-4	0
			Side	-6	3	10	1
		B	Top	-11	1	-3	0
			Side	-16	2	8	0
	Low	A	Top	4	1	-3	0
			Side	-1	4	13	1
		B	Top	0	2	-5	0
			Side	-2	3	8	0
	High	A*	Top	-1	1	-1	1
			Side	6	3	11	1
		B*	Top	3	0	0	0
			Side	-6	2	7	0
	Low	A*	Top	0	1	1	0
			Side	-8	3	9	1
		B*	Top	3	1	0	0
			Side	-2	2	6	0
28	High	A	Top	-12	1	-1	0
			Side	-11	3	-13	1
		B	Top	-13	1	-4	0
			Side	-10	2	6	0
	Low	A	Top	-3	1	-3	0
			Side	2	2	8	0
		B	Top	4	2	-3	0
			Side	-4	2	6	0
56	High	A	Top	-16	1	-4	0
			Side	-23	4	14	1
		B	Top	-13	1	-4	0
			Side	-27	5	12	1
	Low	A	Top	-8	2	-4	0
			Side	0	2	6	0
		B	Top	-3	0	-1	0
			Side	-5	2	7	0
84	High	A	Top	-28	2	1	0
			Side	-23	4	11	1
		B	Top	-12	1	-1	0
			Side	-30	3	11	1
	Low	A	Top	-5	1	-4	0
			Side	3	3	12	1
		B	Top	-7	1	-3	0
			Side	4	-4	14	1

* Additional cure after plating

4 DISCUSSION

4.1 COMPARISON OF AGEING TEMPERATURES

To reduce the number of samples to be examined, it was decided to age samples at 50°C and room temperature, and then after 28 days decide which ageing regime to pursue. Reference to the 28 day results in Table 2, indicates that only two samples exhibited any whiskering. These were the sides of the two high propensity samples aged at 50°C. An example of the whiskers observed is shown in Figure 8. Based on these results, it was decided to monitor only samples aged at 50°C for the remainder of this phase of the project. This finding is agreement with work reported by a number of other workers (Reference 2).

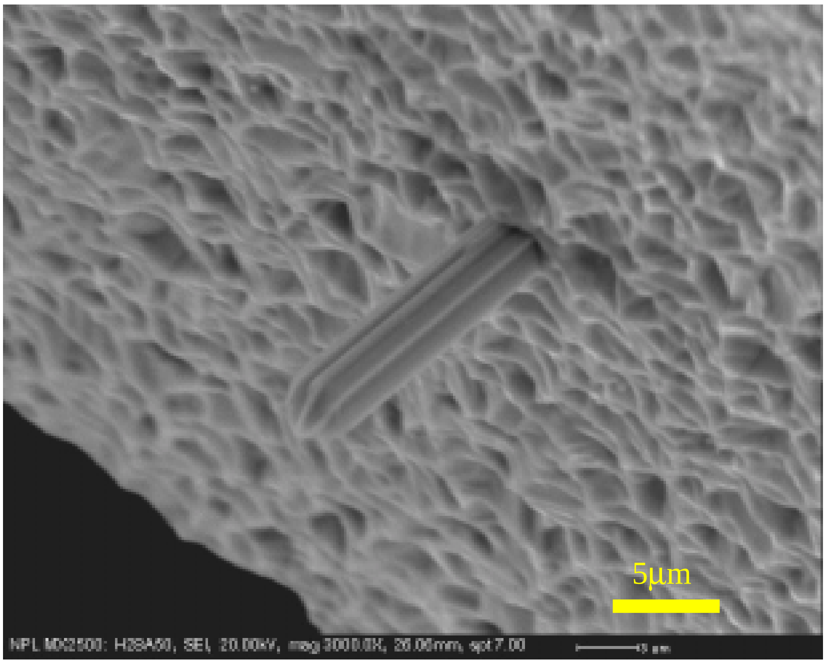


Figure 8 : Example of whiskering seen on high propensity sample aged at 50°C for 28 days

After 84 days of ageing, this decision was checked by examining high and low propensity samples aged at room temperature and comparing them with samples aged at 50°C. The results of this comparison can be seen in Figure 9. These data show that samples aged at 50°C still produced a higher class of whiskering than those aged at room temperature. Examples of the whiskers produced are presented in
Figure **10** and Figure 11. The whisker shapes and sizes are similar. The conclusion from this work is that 50°C should continue to be used for the ageing in phase 2 of the project.

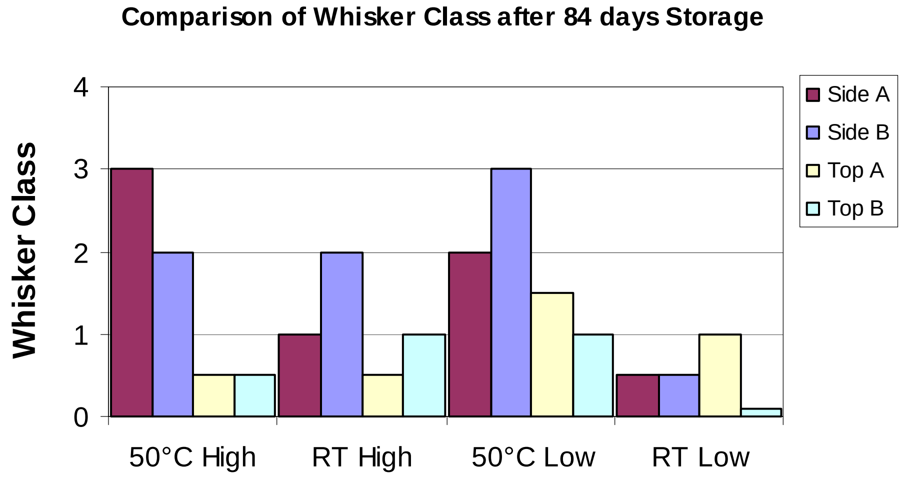


Figure 9 : Comparison of whisker classification of high and low propensity samples after storage at room temperature (RT) and 50°C

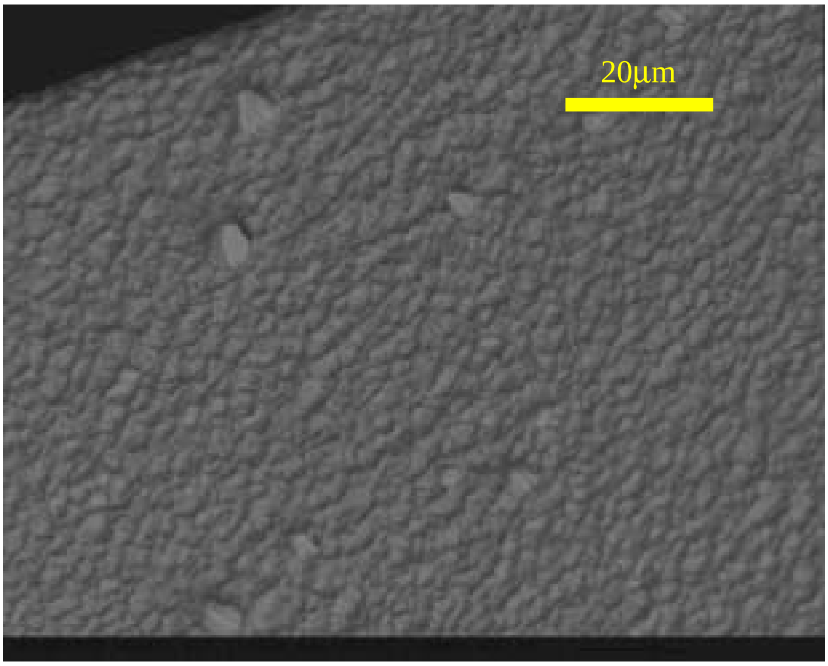


Figure 10 : Back scattered SEM image of the side of a high propensity sample after ageing at 50°C for 84 days

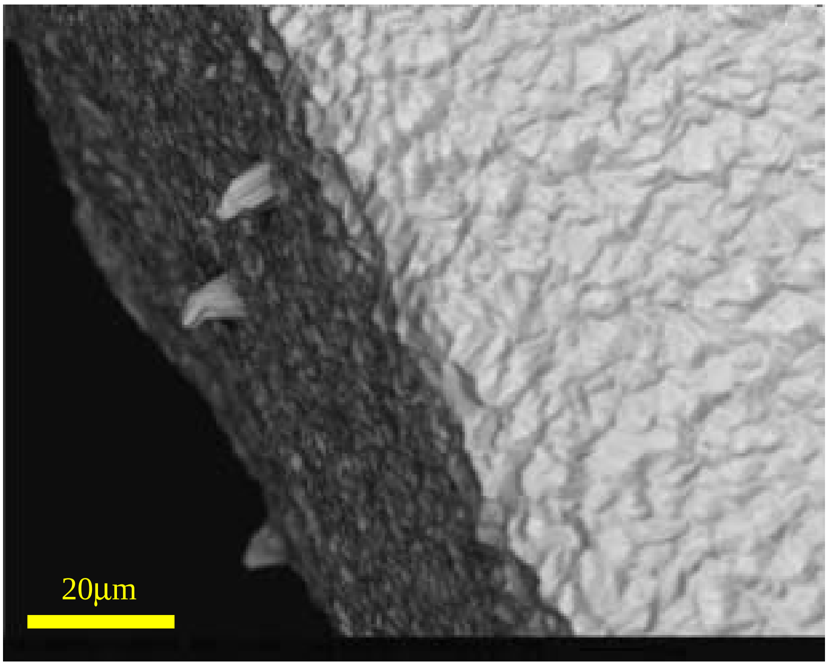


Figure 11 : Back scattered SEM image of the side of a high propensity sample after ageing at room temperature for 90 days

4.2 WHISKER DEVELOPMENT AT 50°C

Figure 12 and Figure 13 show the whisker development on the sides and tops of both high and low propensity samples at 50°C. On the sides of the samples. It can be seen that the high propensity samples develop whiskers earlier but after 84 days, the low propensity samples have developed whiskers to a similar level. Whisker development on the tops of the samples is significantly slower than for the sides, and after 84 days there is insufficient growth to determine whether high or low propensity samples develop whiskers faster.

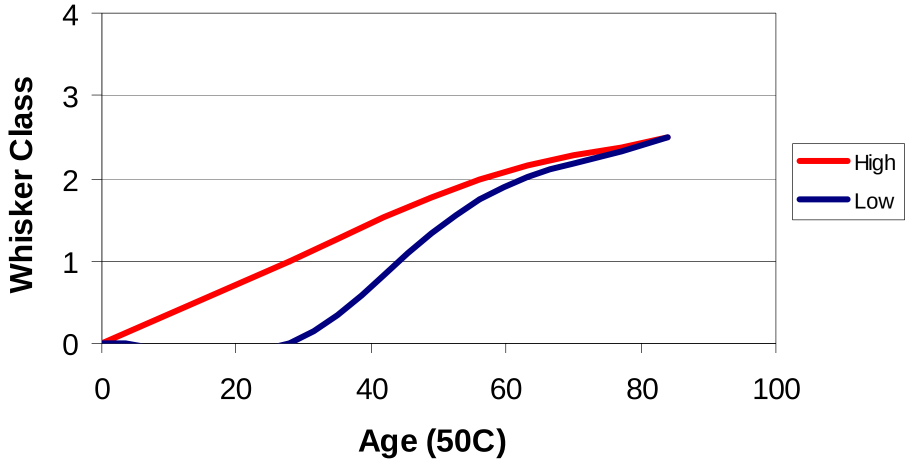


Figure 12 : Comparison of whisker class development on the sides of high and low propensity samples

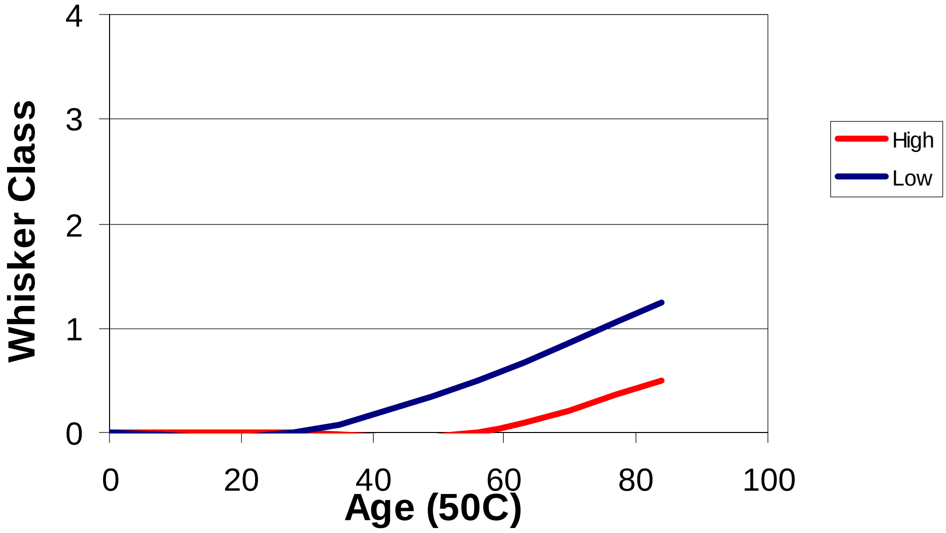


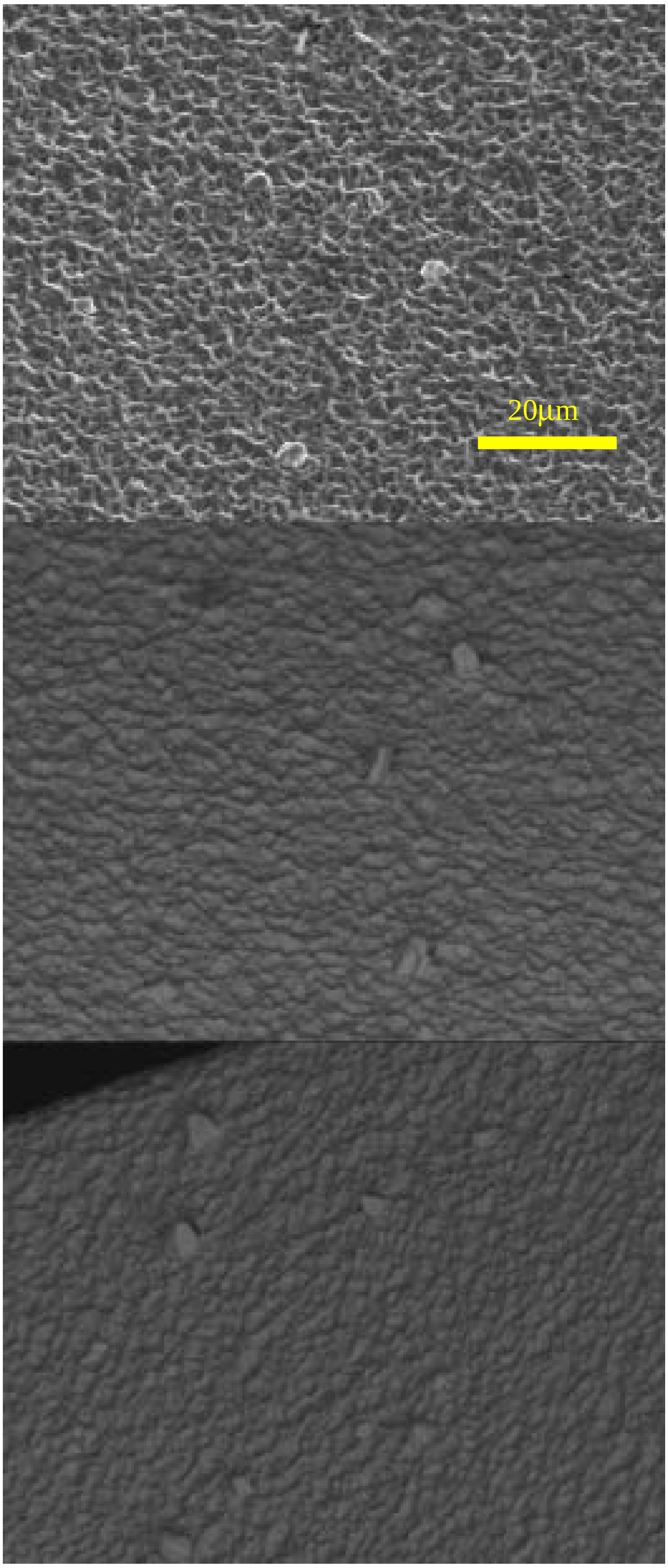
Figure 13 : Comparison of whisker class development on the tops of high and low propensity samples

Figure 14 and Figure 15 show SEM images typical of the whisker development for high and low propensity samples. In both images the 28 day results are shown using the normal SEM mode, but the 56 and 84 days were taken using the back scattered mode. It was found, subsequent to the 28 day results, that in the back scattered mode whiskers were more easy to detect. These images indicate that there is little change in the apparent size of whiskers occurring between 56 and 84 days. Hence it is recommended that the current samples be further inspected after 6 and 12 months to determine if any further whisker growth occurs.

It is also apparent that very few of the whiskers are greater than 10 µm in length. It should also be noted that the whiskers are not classical whisker shape (i.e. needle-like) and are of a lower aspect ratio than might be expected.

After 84 days, there was very little evidence of whisker growth on the tops of both high and low propensity samples. Figure 16 shows a typical view of the top of a high propensity sample after ageing at 50°C for 84 days.

Because of the increased whisker growth on the sides of samples, it is recommended that only the sides of samples be inspected for phase 2 of the project. This will enable a greater diversity of samples to be inspected.

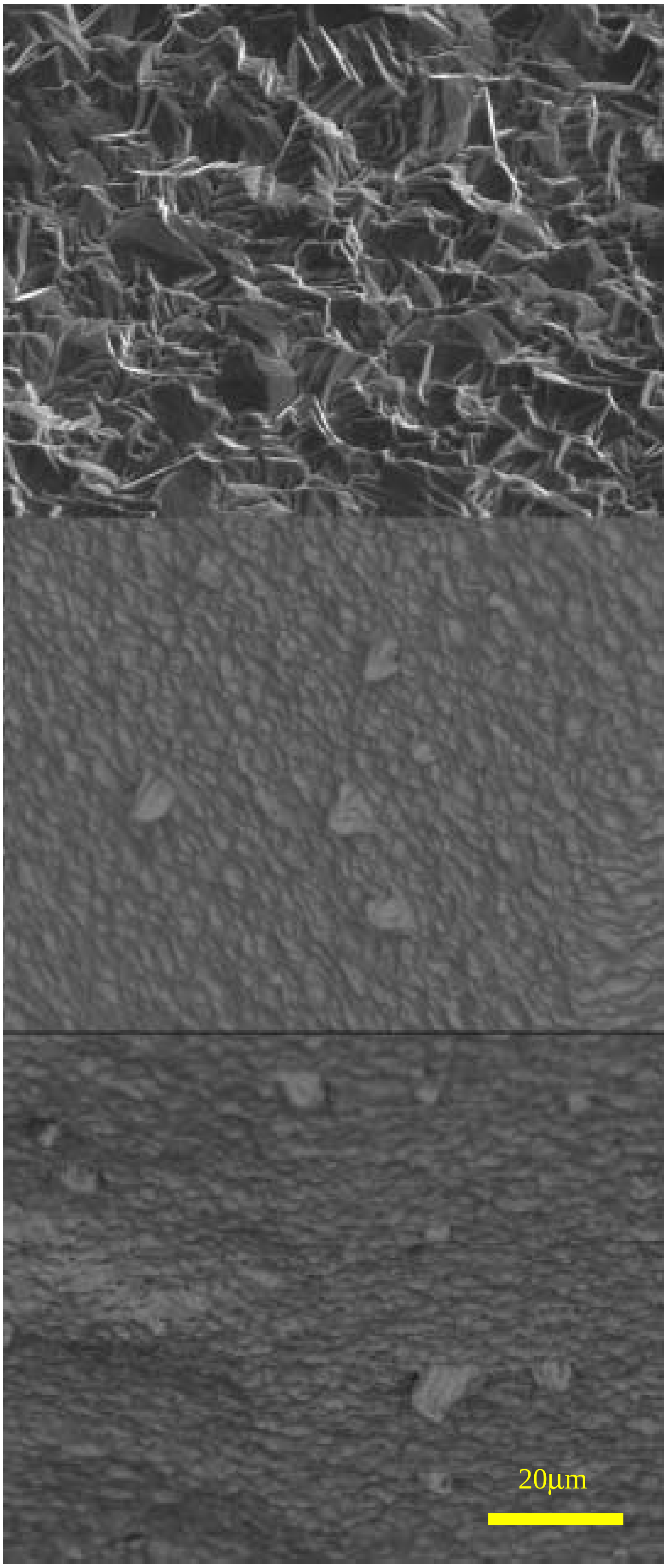


28 days

56 days

84 days

Figure 14 : Whisker development over 84 days for high propensity samples



28 days

56 days

84 days

Figure 15 : Whisker development over 84 days for low propensity samples

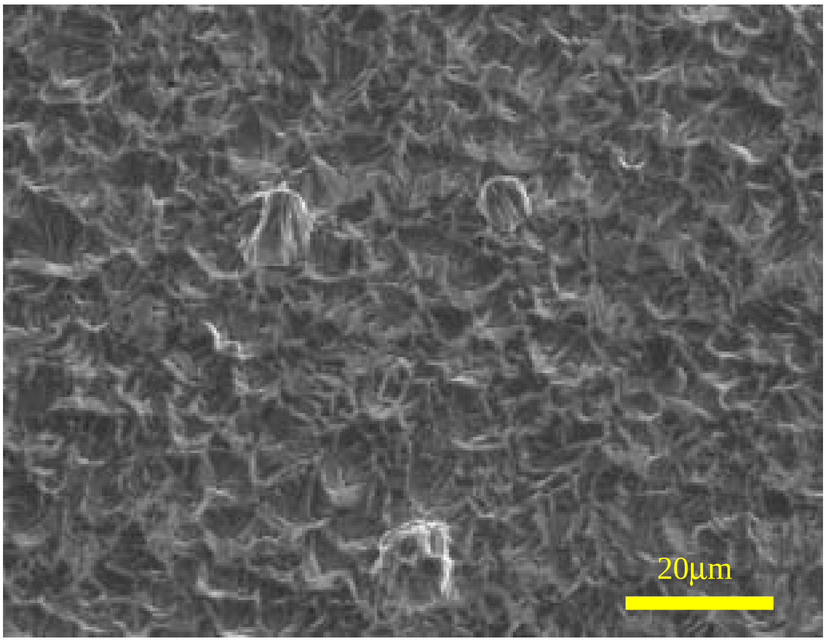


Figure 16 : Typical SEM of top of high propensity sample after ageing for 84 days at 50°C

4.3 COMPARISON OF TYPES OF WHISKERS PRODUCED BY HIGH AND LOW PROPENSITY SAMPLES

Figure 17 presents a comparison of typical whiskers developed from high and low propensity samples after 84 days at 50°C at the same magnification. The aspect ratio of the different whiskers is very similar but the size of the whisker for the low propensity sample is significantly greater. Perhaps the most interesting aspect is the different diameters of the whiskers. Further investigation of 6 and 12 month old samples may provide more information on these differences between the whisker types.

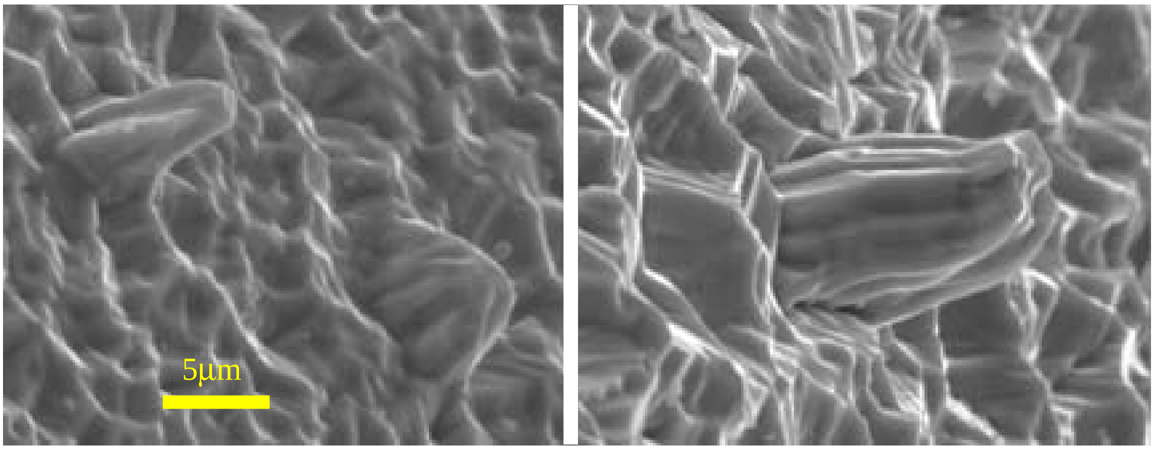


Figure 17 : Comparison of typical whiskers developed from high propensity (left) and low propensity (right) samples after 84 days at 50°C at the same magnification

4.4 AREAS WITH LARGER WHISKERS

On a number of both high and low propensity samples, some small areas of higher whisker growth were evident. These areas were associated with interrupted plating where electrical connection had been made to the lead-frame for plating purposes and thus the plating is likely to have been very thin. These interrupted areas would not occur in normal plating, but occurred here since the plating was carried out in a laboratory environment. Examples of the whiskers observed are shown in Figure 18 and an example of an interrupted plating area is shown in Figure 19.

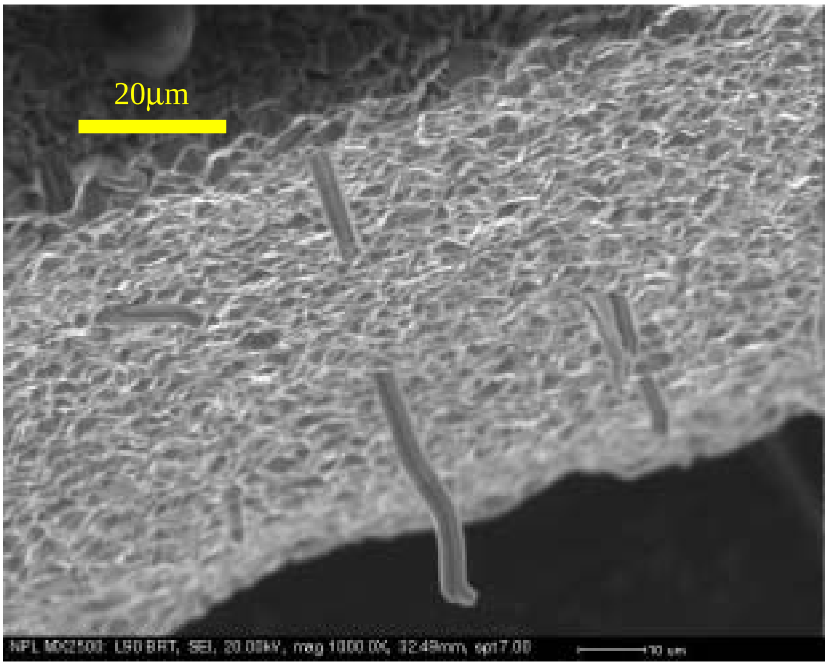


Figure 18 : Typical area of longer whisker growth (low propensity, 90 days at room temperature)

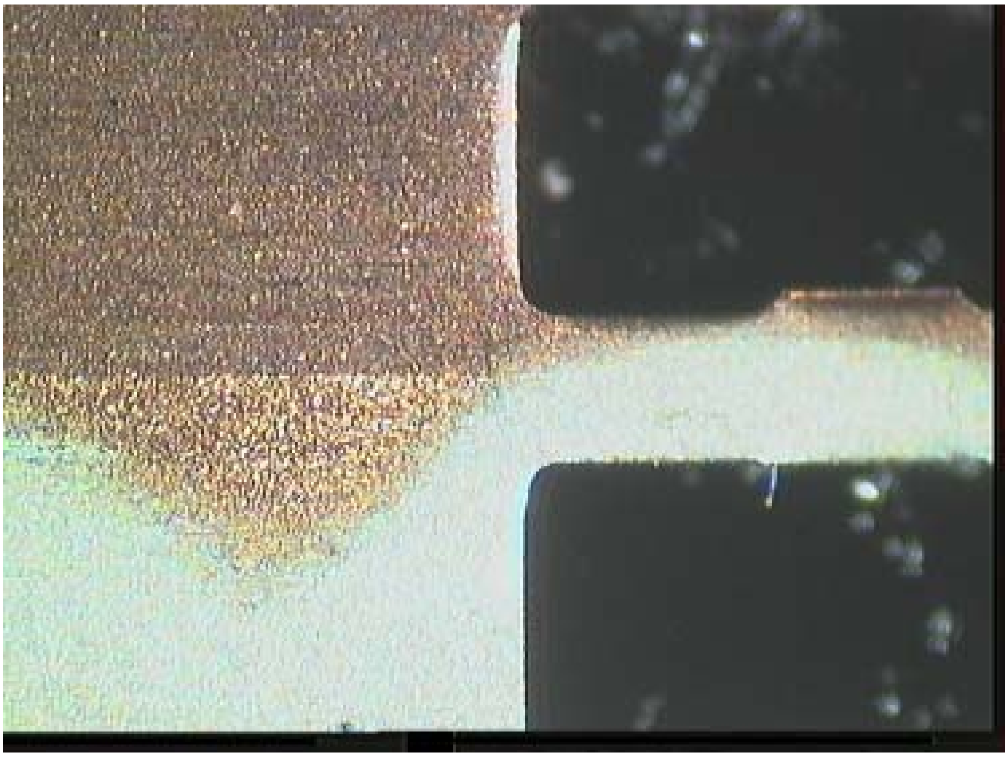


Figure 19 : Example of interrupted plating area showing large whisker lower right

4.5 WHISKER OCCURRENCE ON COMPONENTS

In addition to the remainder lead-frames, a number of standard components cropped from these lead-frames were also aged at 50°C. These samples also exhibited whiskering, but at a lower level than that observed on the remainder lead-frames. On the limited number inspected, it appeared that the whiskering was largely restricted to the sides of the outermost leads of the devices. No whiskers were seen on the top surfaces of component samples. Whiskering was generally more advanced on the high propensity samples. Figure 20 and Figure 21 illustrate typical examples of whisker occurrence on components.

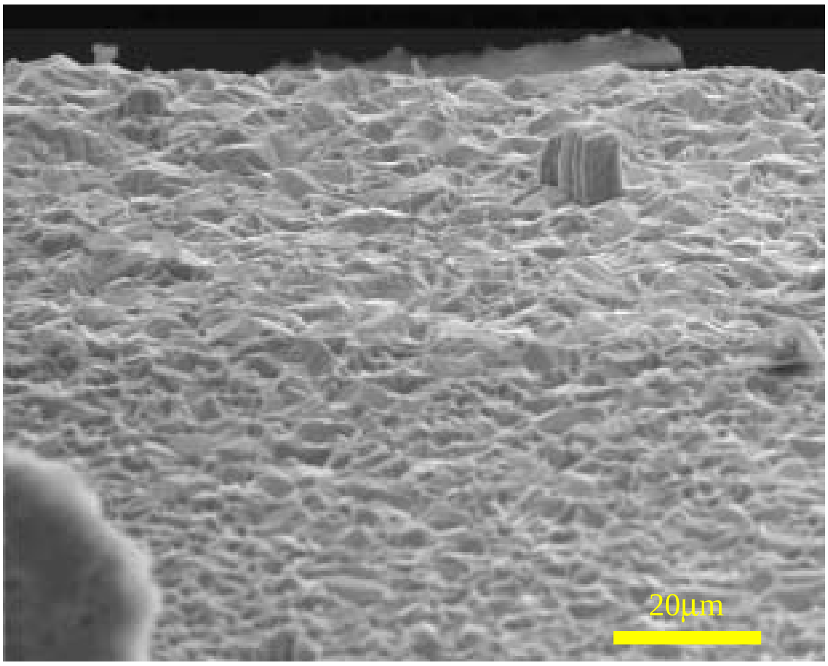


Figure 20 : Whiskering on high propensity component sample

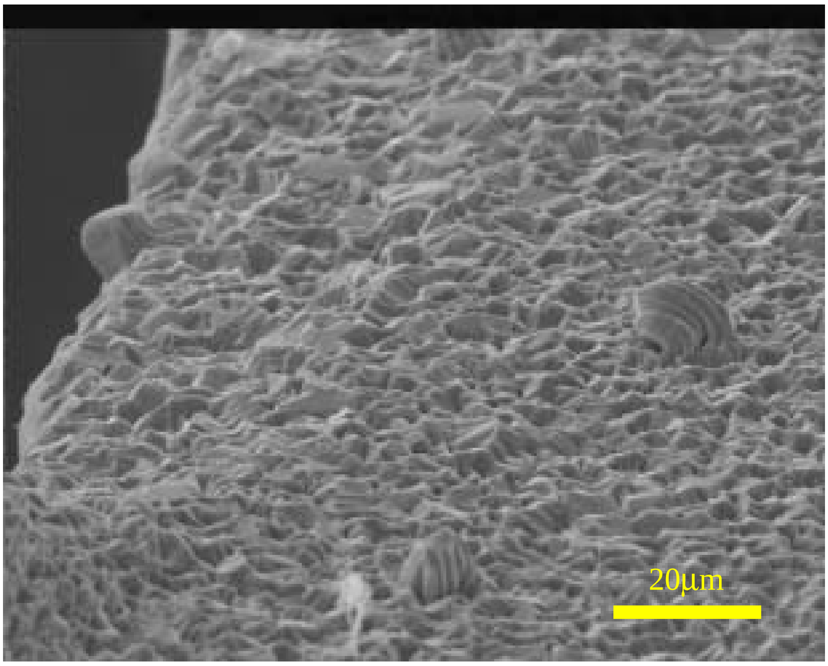


Figure 21 : Whiskering on low propensity component sample

4.6 XRD RESULTS

Initially the diffraction peaks obtained from both the high and low propensity samples were reasonably well defined with good intensity. As the samples were aged the definition of the peaks became progressively worse. In particular with the high propensity samples overlapping peaks and orientation effects became more prominent, with diffraction peaks at psi angles around ±10° and 0° becoming either non-existent or so broad that they could not be used for the stress evaluation. Given the thickness of the samples this is not wholly surprising. Consider Figure 22, which shows the X-ray path and penetration for a thin coating at a low psi angle and a high psi angle. At high angles, for example 40°, the X-rays penetrate the coating at a more oblique angle thereby sampling a greater volume of the coating than for low angles, for example 0°, where the X-rays pass through the coating and much of the resultant diffracted signal originates from the substrate material.

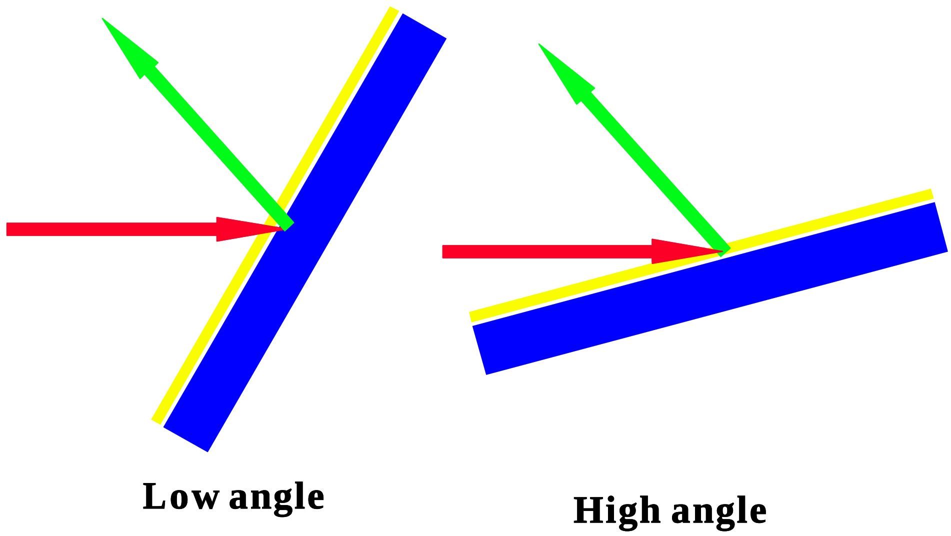


Figure 22 : Effect of psi tilt on X-ray penetration

This effect did not, however, adversely affect the residual stress evaluation as sufficient high angle data were collected to enable reliable analysis to be carried out.

The data, as presented in Table 5 and Figure 7, show that measurable residual stresses were developed within the high propensity samples almost immediately. As the samples were aged the level of stress increased. This trend was similar for both the top face and the side face. In the low propensity samples insignificant stress was measured at the initial stages. The stress appeared to become more compressive for the top face, although the actual increase was very small, and could represent sample-to-sample variation. The side face showed an increase in the average measured stress that became tensile, although this too is considered due to sample variation than associated with whisker development.

The results therefore demonstrate that residual stresses were present in the high propensity samples. This compressive stress increased as the samples were aged. It is possible that such stresses also developed in the low propensity samples, but due to the different levels of thickness between the two batches, the X-rays were unable to penetrate a sufficient distance into the plating, i.e. down to the plating-substrate interface, for the low propensity samples. If we compare the calculated penetration depth of the Cr Kα X-rays into tin (1.5 – 2 µm) with the nominal thickness of the plating on the low propensity (10 – 14 µm) and high propensity samples (3 – 6 µm) then it is clear that the collected diffraction data arise from very different locations, relative to the coating / substrate interface. Hence it is possible to conclude that the stress is likely to be developing at the interface.

4.7 COMPARISON OF SEM INSPECTIONS AND XRD MEASUREMENTS

Figure 23 shows a comparison of the results for whisker classification and residual stress for the sides of high propensity samples aged at 50°C. A good inverse correlation can be seen with the degree of residual stress decreasing as whisker growth increases. 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 Sn grains, leading to whisker formation (References 4 and 5). An observation from these results is that the residual stress measurement does not predict whisker growth, but does reflect the level of formation.

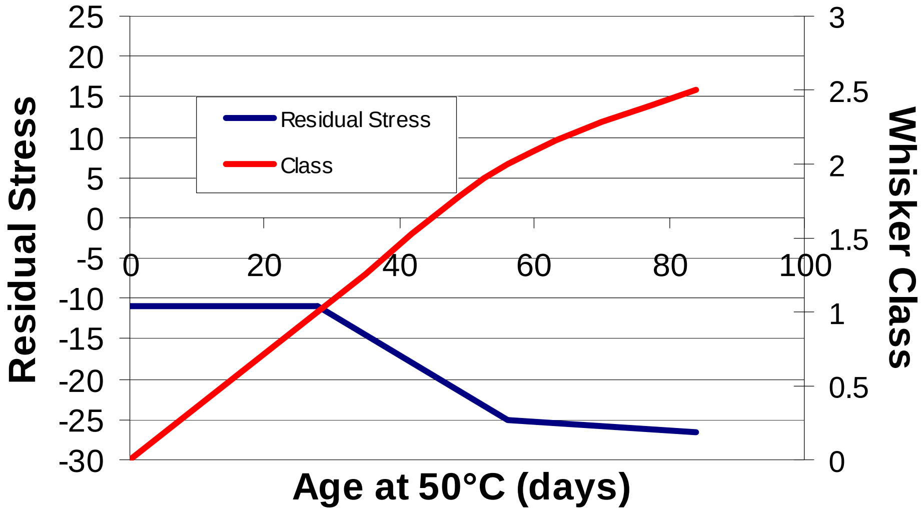


Figure 23 : Comparison of whiskering classifications with residual stress over an initial 84 days of ageing at 50°C for the sides of high propensity samples

Figure 24 presents a similar comparison for low propensity samples. However, here there is no clear correlation between whisker development and residual stress. This may be associated with the increased thickness of the plating. 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 depth penetration of these samples by the incident X-ray beam is limited to 1-2µm, whilst the plating thickness on these samples is around 18µm.

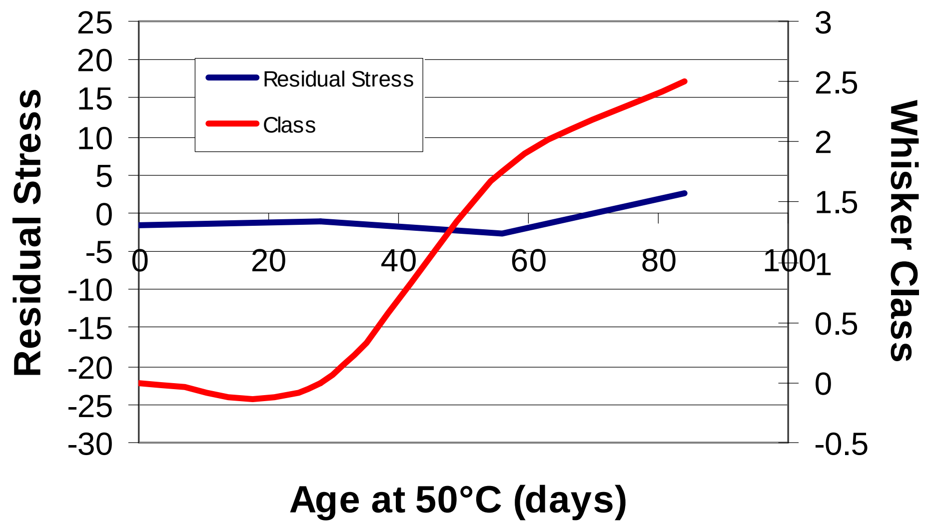


Figure 24 : Comparison of whiskering classifications with residual stress over an initial 84 days of ageing at 50°C for the sides of low propensity samples

Figure 25 and Figure 26 provide similar comparisons for the top surfaces of high and low propensity samples. However, although a similar correlation appears to be starting for the high propensity samples, insufficient data are currently available to confirm this.

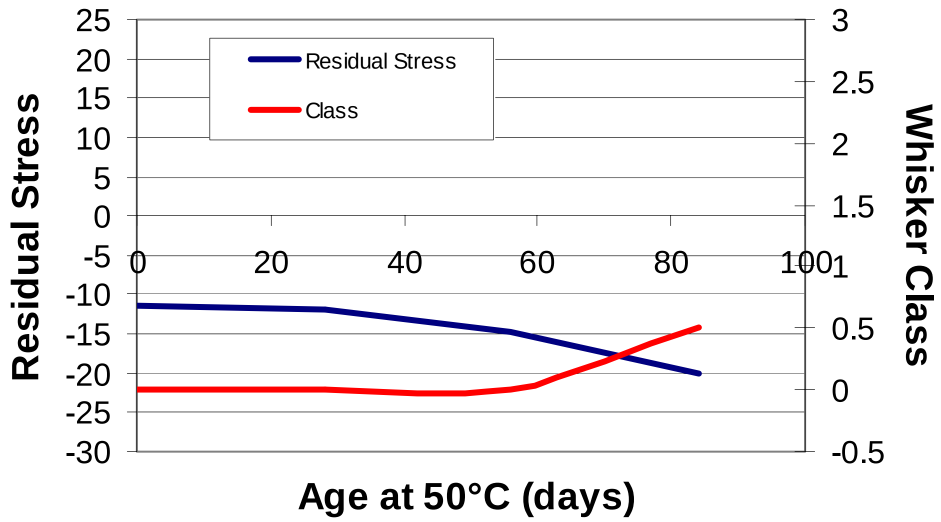


Figure 25 : Comparison of whiskering classifications with residual stress over an initial 84 days of ageing at 50°C for the tops of high propensity samples

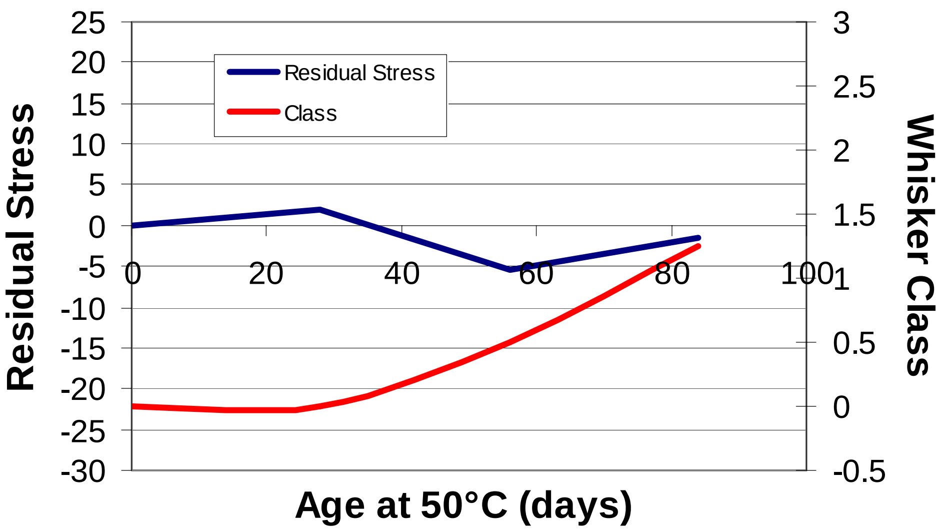


Figure 26 : Comparison of whiskering classifications with residual stress over an initial 84 days of ageing at 50°C for the tops of low propensity samples

5 CONCLUSIONS

A general comment on the coatings studied here, none of the samples produced whiskers greater than 20µm in length, and most were characteristically half this size, (except for the untypical areas of very thin coatings, not normally encountered). It is very unlikely whiskers of this size will cause any problems.

Phase 1 of this project has enabled a number of conclusions to be made.

- Both high and low propensity remainder lead-frame samples exhibited whisker growth over a 84 day period of aging at 50°C
- Whiskers developed faster on the sides of the remainder lead-frame samples than on the top surfaces.
- Low propensity samples exhibited slower whisker formation; although the whiskers generated were generally of larger diameter, they exhibited the same aspect ratio
- Very few whiskers were observed that were longer than 10µm
- Larger whiskers were noted in areas on interrupted plating
    - This is not typical of a production environment
- Small whiskers were noted on the sides of components after 84 days of ageing at 50°C
- Ageing at 50°C initially developed whiskers faster than at room temperature on remainder lead-frames, although after 84 days, results were very similar
- A good correlation between residual stress and whisker growth was found for thin coatings but not for thicker coatings

  - o In these latter samples, residual stress may still be accumulating, but because of the greater coating thickness, may only be measurable using XRD at a later stage
- Residual stress measurement is not predictive in nature for the coatings used in this phase of the project

These observations enabled a number of decisions to be made about phase 2 of the project.

- Ageing at 50°C will be used
- SEM inspection and XRD measurement will only be undertaken on sides of samples
- A limited further investigation of phase 1 samples will be undertaken for ageing beyond 84 days

6 ACKNOWLEDGEMENTS

The work was carried out as part of a project in the MPP Programme of the UK Department of Trade and Industry. We gratefully acknowledge the support and co-operation of the following companies without whose help this project would not have been possible.

CML Microcircuits (UK) Ltd

Cookson Electronics

European Space Agency

Schloetter Company Ltd.

Shipley Europe Ltd.

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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