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Atomic Layer Deposition (ALD) to Mitigate Tin Whisker Growth and Corrosion Issues on Printed Circuit Board Assemblies

TERHO KUTILAINEN,[1] MARKO PUDAS,[2] MARK A. ASHWORTH,[3,6] TERO LEHTO,[2] LIANG WU,[3,5] GEOFFREY D. WILCOX,[3] JING WANG,[3] PAUL COLLANDER,[1] and JUSSI HOKKA[4]

1.—Oy Poltronic Ab, Espoo, Finland. 2.—Picosun Oy, Masala, Finland. 3.—Department of Materials, Materials Degradation Centre, Loughborough University, Loughborough, UK. 4.—ESA-ESTEC, European Space Agency-European Space Research and Technology Centre, Noordwijk, The Netherlands. 5.—*Present address:* Centre for Manufacturing and Materials Engineering, Coventry University, Coventry CV1 5FB, UK. 6.—e-mail: m.a.ashworth@lboro.ac.uk

This paper presents the results of a research program set up to evaluate atomic layer deposition (ALD) conformal coatings as a method of mitigating the growth of tin whiskers from printed circuit board assemblies. The effect of ALD coating process variables on the ability of the coating to mitigate whisker growth were evaluated. Scanning electron microscopy and optical microscopy were used to evaluate both the size and distribution of tin whiskers and the coating/whisker interactions. Results show that the ALD process can achieve significant reductions in whisker growth and thus offers considerable potential as a reworkable whisker mitigation strategy. The effect of ALD layer thickness on whisker formation was also investigated. Studies indicate that thermal exposure during ALD processing may contribute significantly to the observed whisker mitigation.

Key words: Tin whiskers, corrosion, atomic layer deposition, ALD

INTRODUCTION

RoHS-regulation (The Restriction of the use of certain Hazardous Substances in Electrical and Electronic Equipment) is driving the electronics industry towards the use of lead-free solders. These usually consist of tin with small additions of silver and copper (generally abbreviated as SAC), and sometimes other metals for adjusting the melting temperature and other properties needed for the soldering process and long-term reliability. Among other metals, pure tin and its alloys are known to form whiskers that are conductive crystalline structures of tin. Despite the RoHS exemptions allowed for many specific applications, such as space applications, the availability of tin-lead plated components is decreasing and fewer manufacturers maintain tin-lead soldering processes, as most of the commercial electronics industry is becoming lead-free.

Tin whisker growth, which may occur spontaneously and unpredictably from electroplated Sn finishes and solder joints, can result in electronics failures such as short circuits. Examples of the different growth morphologies that tin whiskers can adopt are shown in Fig. 1a, b, c, d and e. According to the JEDEC standard, a feature is classified as a whisker if it possesses an aspect ratio (length/width) greater than 2 and as being of significance if its length is greater than 10 μm.[1]

Short circuits induced by whisker growth may be permanent if the current is smaller than the melting current of the whisker, or intermittent if the current is sufficient to cause the whisker to melt (typically $>$ 10 mA [2]). In some cases, whiskers may melt and bridge again or break loose and cause short circuits or interfere with sensitive parts of the device. Airborne whiskers may also interfere with sensitive surfaces, such as those in optoelectronic equipment.[3] If the current source is able to provide

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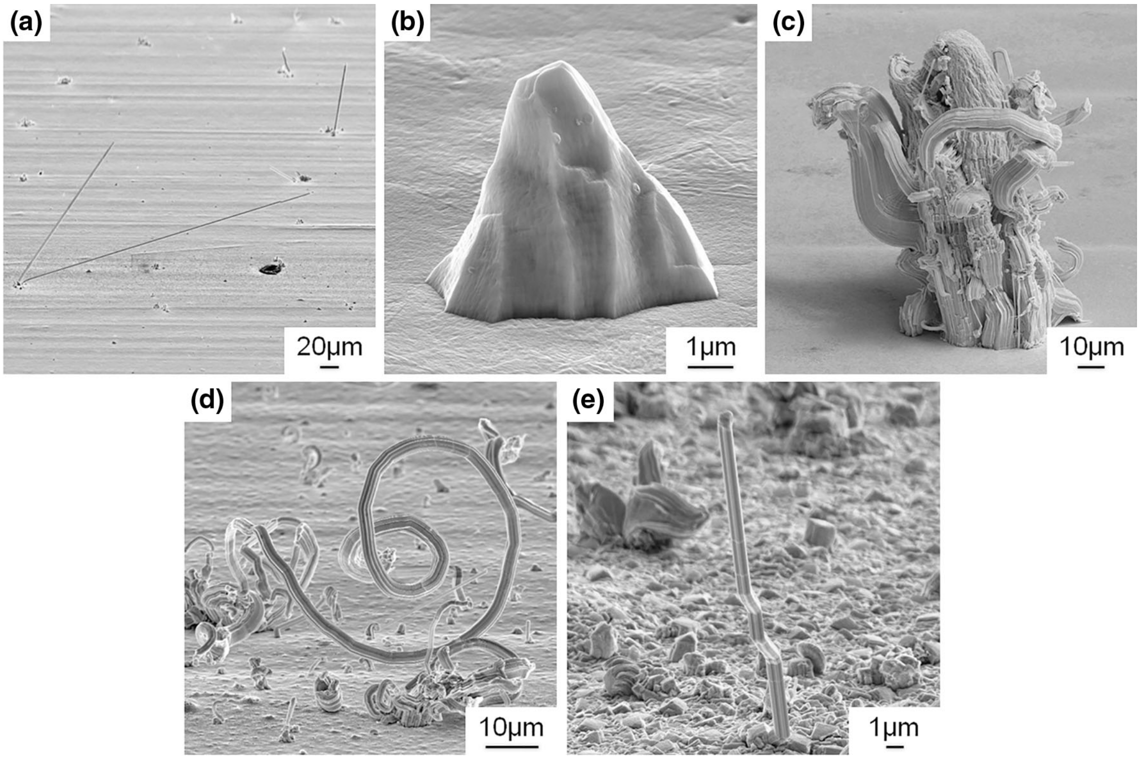

Fig. 1. SEM images illustrating some of the various forms of whisker growth that are possible: (a) filament, (b) nodule, (c) odd shaped eruption, (d) spiral and (e) kinked.

sufficient current, a short circuit may result in the formation of a conductive plasma of metal ions, which may cause severe damage to the devices and the surrounding structures.[2]

Compressive stress, caused by volumetric expansion as a result of Cu-Sn intermetallic growth, is widely accepted as the primary driving force for the growth of whiskers from Sn coatings on Cu substrates.[4–6] Moreover, the morphology and crystallographic structure of the intermetallic compound has been shown to influence the extent of whisker growth.[5] In addition to intermetallic formation, which occurs even at room temperature, mismatch in the coefficient of thermal expansion (CTE) may result in the generation of stresses during thermal cycling.[7,8] Elevated temperatures and high humidity conditions that result in corrosion are also a potential additional mechanism for stress formation in a tin layer [9,10], as are directly applied external mechanical loads.[11–14] There are, however, many factors that may influence the growth of whiskers, including the physical characteristics of the Sn layers (e.g. thickness,[7,15] grain size and crystallographic structure), and despite an increased understanding of the processes that give rise to whisker growth the phenomenon is still not fully understood.[4,16] The influences on whisker growth of other factors such as current load [17–19] and electrostatic forces [20] have also been considered.

Usually, conformal coatings are applied to improve the capability of electronic boards to withstand various environmental conditions such as humidity, external particles, liquid and gaseous impurities. Such coatings also provide a degree of protection from whisker growth.[21–23] Atomic later deposition (ALD) coatings are currently utilised for corrosion protection in many industrial and commercial applications, and therefore represent an interesting option for the protection of assembled electronic circuit boards and modules, especially in specific high-end applications. ALD has been studied previously alongside other coating methods and materials, and has been shown to give promising results with regards to tin whisker mitigation, see e.g.[2,24,25]

ALD is a gas-phase coating method used to produce ultra-thin films of high uniformity and conformality and is based on surface-controlled and self-saturating adsorption reactions between gaseous precursors and the surface, as shown in Fig. 2. The film growth proceeds sequentially, which inherently leads to precise control of film thickness and results in a highly conformal coating that is transparent, practically massless and pin-hole free. The ALD process takes place in vacuum (0.1–10 mbar in typical production tools) and typically at elevated temperature (e.g. 40–400°C), but it can be performed at room temperature for some materials. In addition to the deposition of single elements, alloys or compounds, it is possible to deposit film laminates by alternating layer composition, even on a monolayer by monolayer basis. However, there are

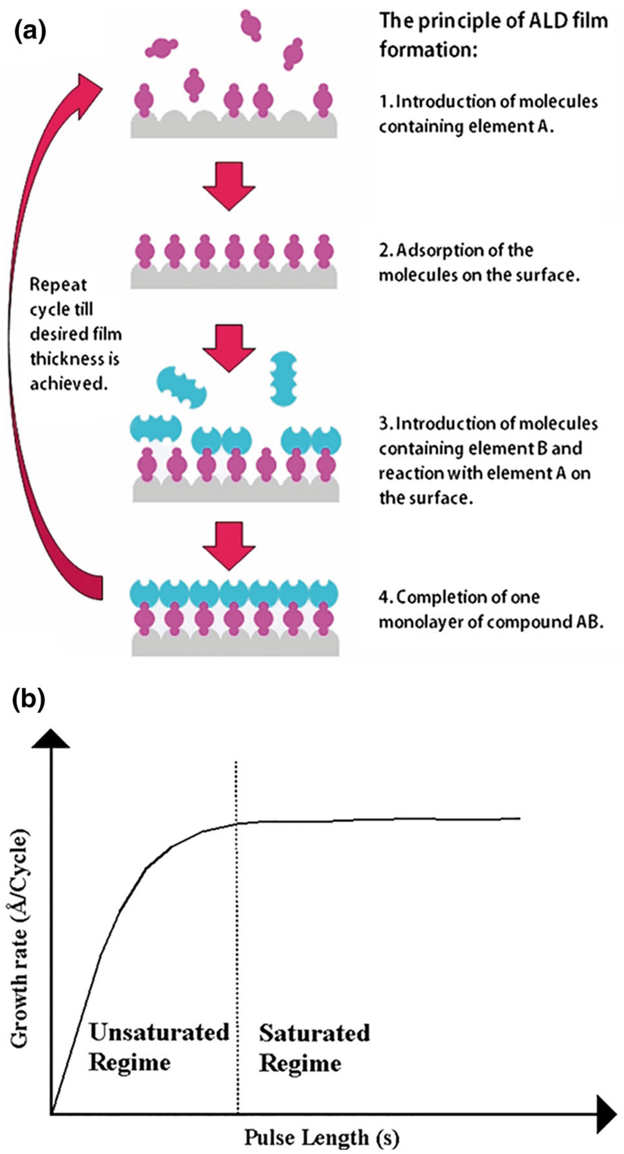

Fig. 2. ALD of a binary compound A-B using self-limiting surface chemistry: (a) reaction sequence with molecules containing elements A (e.g. Al) and B (e.g. O) and (b) saturation of growth rate with sufficient precursor pulse length. Figure courtesy of Picosun/Dr T. Suntola.

certain practical restrictions and it is important to optimise layer compositions, thicknesses and structures to reach the desired properties and performance. Laminate structures have previously been shown to provide very good corrosion protection (e.g. [26]) and offer the potential to improve the flexibility of the coating. For a more in-depth introduction to the ALD process and its applications, the reader is referred to the following reviews.[27–29]

In this paper, we concentrate on tin whiskers and show results from a project whose main aims were to assess the capability of ALD coatings to mitigate the growth of tin whiskers using dedicated test vehicles and develop the ALD processes for the coating of a typical component board structure.

MATERIALS AND METHODS

Preparation of Test Coupons

Electroplated Sn-Cu deposits on Cu (0.4 mm thick, 99.9% purity, Advent Research Materials) were used to evaluate the ability of ALD coatings to mitigate whisker growth. Two-μm thick Sn-Cu coatings were deposited at a current density of 10 mA cm^{-2} from a commercial bright Sn electroplating bath modified by the addition of 10 mmol L^{-1} copper (II) sulphate. Test coupons had dimensions of 20 × 40 mm and the electroplated area was 20 × 20 mm. Except for batch 1, control samples were also electroplated for each batch; these did not undergo ALD processing but were otherwise subjected to the same environmental conditions.

Atomic Layer Deposition (ALD)

In this work, 22 different ALD coatings with variations in pre-treatment or thickness were investigated. Table I shows an overview of the processing parameters and the time interval between electroplating and ALD processing for the sample batches discussed in this paper. Examples of ALD coated whisker test coupons are shown in Fig. 3. All depositions and thermal treatments were carried out at 125°C, which was defined as the maximum allowable temperature for electronic assemblies in this programme.

To serve as a control a batch of samples was subjected to a thermal treatment (batch 13) to simulate the conditions experienced during ALD processing; this was done to decouple the effect of the thermal cycle from that of the thermal cycle and oxide coating combined. The thermal treatment was undertaken within the ALD reactor using the same treatment time, process temperature and atmosphere as that utilised for ALD coating, i.e. the only difference was that the chemicals used to form the ALD coating were not admitted into the reactor.

A series of samples were also prepared to investigate the effect of ALD coating thickness on whisker growth (batch 16). The thickness of the applied coating varied between 0.9 nm and 87.2 nm. Whisker growth for these samples was evaluated after 36 months storage at room temperature.

Characterisation Methods

After ALD coating, whisker growth was evaluated at periodic intervals using both optical and scanning electron microscope techniques. For each batch, average whisker densities were evaluated from 3 randomly selected ALD coated samples using an optical microscope at 20 random locations, distributed across the sample, using a 20× objective lens (corresponding to an area of ~ 0.57 mm^2); 2 control samples were also examined for each batch. In this study, only filament type whiskers and large eruptions having a high aspect ratio were considered. At the time of the initial analysis, storage time

Table I. Summary of the processing details for the different sample batches

Batch	Recipe	Pre-treatment	Coating material	Coating thickness (nm)	Interval between electroplating and ALD coating (days)
1	R150508A	None	'ALD 1'	87	16
3	R150502A	Reductive			2
4	R150618A	–		500	4
5	R150715	–	'ALD 2'	87	7
6	R150717	–		500	4
7	R150914B	–	'ALD 3'	87	5
8	R150915	–		500	4
13	R150502A	Thermal treatment under N_2 flow at 0.1–1 mbar			5
16	R151002A	–		0.9–87	6–13

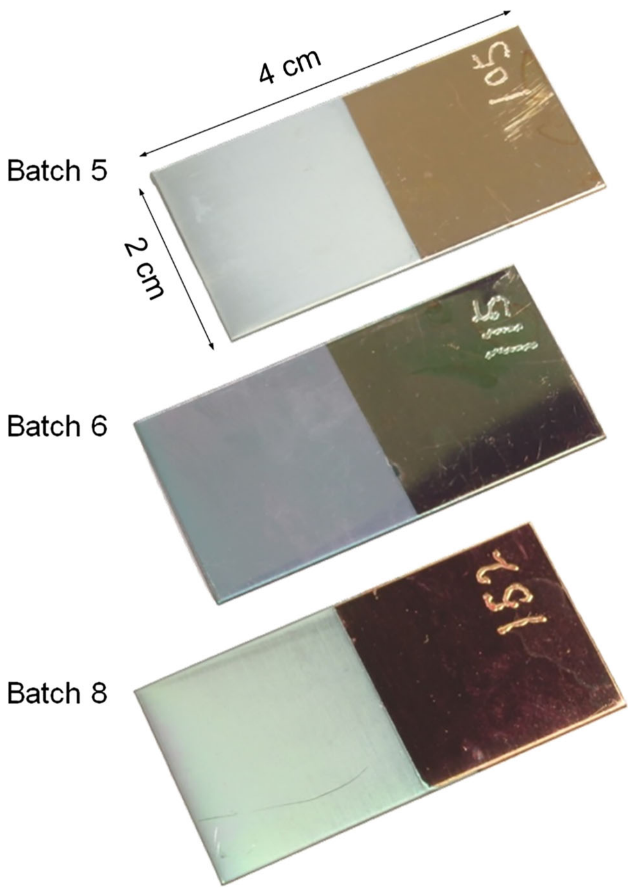

Fig. 3. Photograph showing the dimensions and appearance of selected ALD coated test coupons.

at room temperature (20°C) ranged from 12 months for batch 1 to 8 months for batch 8. A further examination of whisker growth was undertaken for selected sample batches after 3 years.

RESULTS AND DISCUSSION

Evaluation of Whisker Growth on ALD Coated Samples: 1 year After Processing

The measured average whisker density, determined from 20 areas, is shown in Fig. 4 for each of the sample batches investigated. From the graph, it is apparent that whisker densities are very significantly reduced for the ALD processed samples compared with uncoated control samples, except for samples from batches 7 and 8. The lowest filament whisker growth was observed for batches 3, 4, 5 and 6 with, on average, no more than 2 filament type whiskers observed over 20 frames for any of the batches (corresponding to 20 whiskers cm^{-2}). Indeed, no filament whiskers or large eruptions were recorded for any of the samples from batch 3 (ALD 1, 87 nm coating thickness), which had the shortest delay (2 days) between electroplating and ALD coating. In comparison, the whisker density measured for the unprocessed control samples from these batches was 3000 cm^{-2}. Representative SEM images comparing whisker growth from ALD processed samples with those from control samples are shown in Fig. 5 for batches 4 and 6.

For batch 1 samples, processed using recipe ALD 1, the average whisker density was 159 $\pm$ 15 cm^{-2}. Although this value is somewhat higher than those recorded for ALD processed samples from batches 3-6, they are still greatly reduced compared with those recorded for unprocessed control samples from other batches (typically 3000 whiskers cm^{-2}). The increased interval between the deposition of the Sn-Cu coating and ALD processing (see Table I) is the likely reason for the increased whisker growth in this batch; i.e. the observed whiskers may already have been present at the time of ALD processing due to the propensity of the Sn-Cu coating to rapidly develop whiskers.

Results showed that the least effective coating treatments, regarding whisker formation, were those used for batches 7 and 8 (ALD 3). For these batches increased whisker densities were recorded, compared with batches 1 and 3–6, despite the comparable interval between electroplating and ALD processing. The whisker density for ALD coated samples from batch 8 was comparable with that measured on the unprocessed control samples.

The approximate length of the longest whisker present within each measurement frame was also determined for all batches. For ALD coated samples from batches 3–6, all the recorded whisker lengths

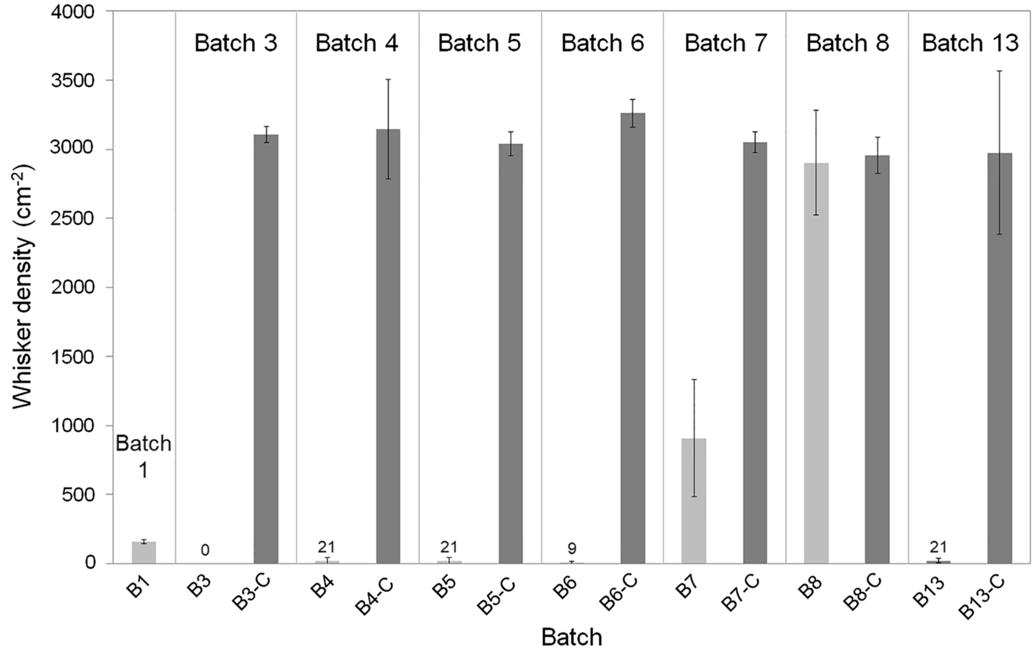

Fig. 4. Graph comparing whisker growth data after 1-year storage at room temperature for ALD processed and control samples.

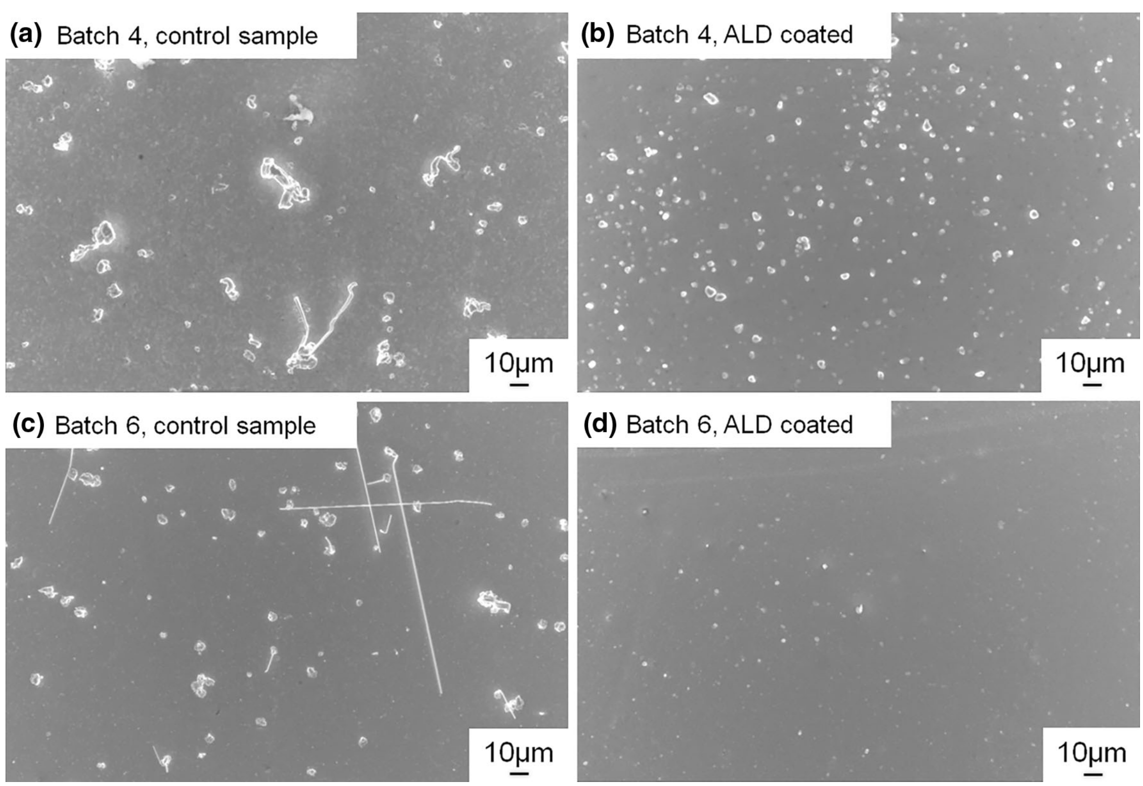

Fig. 5. Scanning electron micrographs comparing whisker growth on ALD processed samples with that on unprocessed control samples: (a) control sample and (b) ALD coated sample from batch 4 and (c) control sample and (d) ALD coated sample from batch 6.

were less than 75 μm. In comparison, many whiskers with lengths in the range 100–500 μm were recorded for the control samples. Whisker lengths were somewhat increased for batch 1 due to the greater interval between electroplating and ALD coating. For batches 7 and 8 the distribution of whisker lengths was comparable with those of the uncoated control samples. For illustration, histograms showing the distribution in whisker lengths for batches 5 and 7 are shown in Fig. 6.

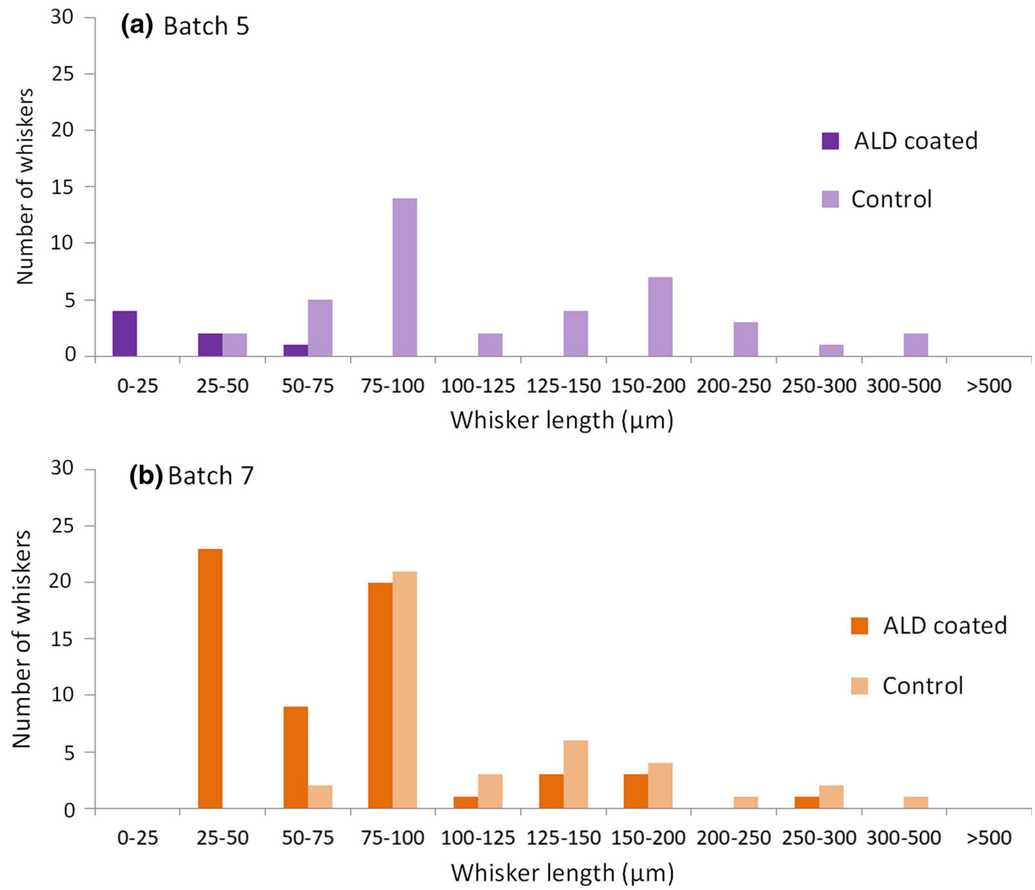

Fig. 6. Histograms comparing the distribution of the longest whisker observed in each measurement frame: (a) ALD coated and unprocessed control samples from batch 5 and (b) ALD coated and unprocessed control samples from batch 7.

The most significant finding from SEM observations was that all the whiskers observed on the ALD processed samples were encased within a shell of the applied ALD coating (Fig. 7). This assessment is based on the appearance of the whisker surface on the ALD coated samples compared with that typically observed on unprocessed control samples from this study. The presence of an oxide shell covering the whiskers confirms that their growth occurred prior to the application of the ALD coating. In many cases, such as the example shown in Fig. 7a, fracture of the ALD oxide shell is observed, possibly as a result of continued whisker growth during storage or built-in residual stresses within the ALD coating material.

SEM analysis of coated samples from batch 8 (500 nm film processed using ALD 3), which possessed a high whisker density, revealed numerous examples of coating failure, as shown in Fig. 8, especially for samples that were previously examined in the SEM and thus subjected to a high vacuum environment and exposure to an electron beam. No signs of underlying whisker growth were apparent in regions where the coating had failed (Fig. 8c), which suggests that whisker growth is not the cause of the coating failure. This supposition is supported by the observation that whiskers present on samples from batches 7 and 8 were all found to be encased within an oxide shell (e.g. Figure 8d). Based on these results it is evident that not all ALD deposited layers produced an optimal film quality for the application.

Since there is no evidence of whisker growth occurring after the application of the ALD coating, it is unclear why whisker growth for batches 7 and 8 should be increased so significantly compared with batches 3-6, especially given that the delay between electroplating and ALD coating was comparatively short for these samples. Moreover, whisker densities for unprocessed control samples were generally consistent, which makes it unlikely that such large variations were related to batch-to-batch variations in whisker growth rates.

Evaluation of Whisker Growth on ALD Coated Samples: 3 years After Processing

A further evaluation of whisker growth was undertaken for batches 1, 3, 4 and 13 after storage at room temperature for 3 years. The results of this analysis are summarised in Fig. 9. For all batches, the whisker density of the unprocessed

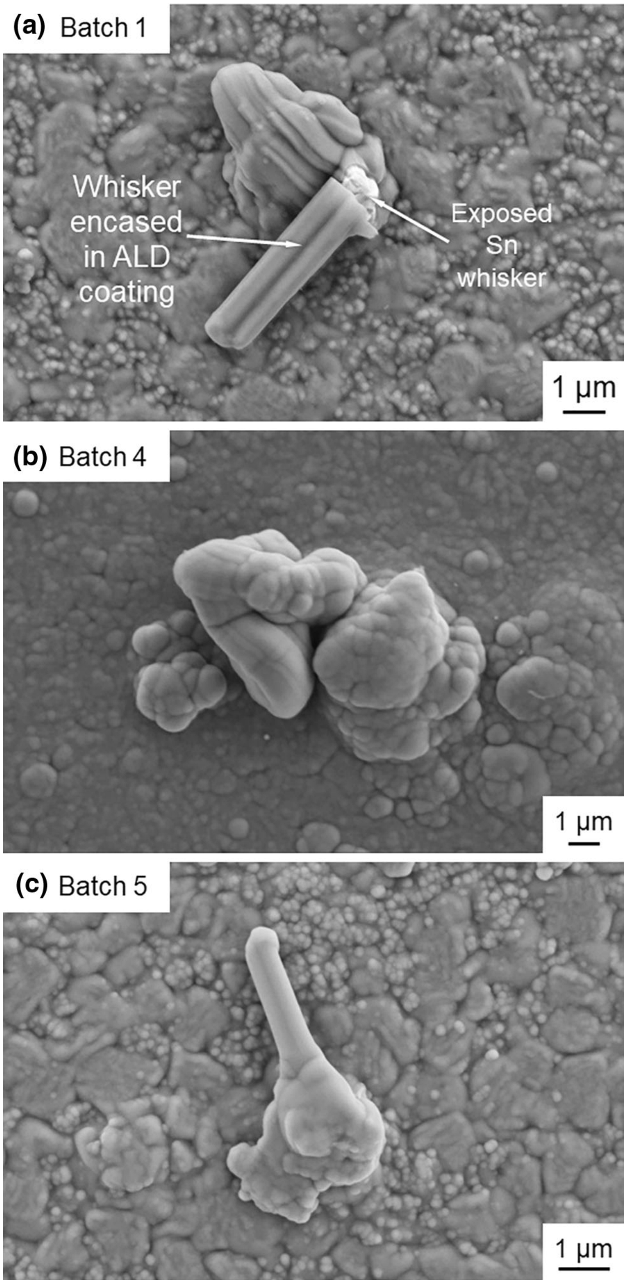

Fig. 7. Secondary electron images showing examples of whiskers encased within an oxide shell after ALD processing: (a) sample from batch 1 showing a whisker covered with a partially cracked oxide coating, (b) an encased whisker from a batch 4 sample and (c) an encased whisker from a batch 5 sample.

control samples increased significantly from 3000 whiskers cm^{-2} after 1 year to > 5000 whiskers cm^{-2} after 3 years.

For ALD processed batch 1 samples there was no increase in whisker density compared with that measured after 1-year storage. Indeed, the measured whisker density was slightly lower than that previously recorded as a result of sample to sample variations in whisker density and from differences in the areas randomly selected for analysis. SEM analysis of an ALD coated sample from this batch showed no evidence of new whisker growth penetrating through the coating; all the observed whiskers were seen to be coated with ALD. Encouragingly, batch 3 samples, processed using ALD 1 (87 nm coating thickness) still showed no evidence of whisker growth 3 years after processing.

Effect of ALD Coating Thickness (Batch 16)

Optical microscope analysis, after 36 months of storage at room temperature (Fig. 10), showed that, compared with uncoated control samples, which had an average whisker density of 6721 ± 15 cm^{-2}, the ALD processed samples all demonstrated a significant reduction in whisker growth. The application of 1.5 nm and 2.4 nm coatings reduced the whisker density to 1177 cm^{-2} and 1086 cm^{-2}, respectively. In general, a progressive reduction in the number of filaments and large eruptions was observed as the thickness of the ALD coating increased. The whisker densities measured for samples with 0.9 nm and 4.0 nm thick coatings were somewhat lower than the other data points; this was most likely due to these samples having a shorter delay between electroplating and ALD coating, i.e. less whisker growth was able to occur prior to coating.

For the samples with thicker coatings it could be clearly observed that the whiskers were encased within an ALD coating and their growth must, therefore, have occurred prior to processing (e.g. Fig. 11a and b).

Thermally Treated Control Samples (Batch 13)

Analysis of the thermally treated (but not ALD coated) samples from batch 13, after 12 months of storage at room temperature, showed that whisker growth (21 ± 18 cm^{-2}) was greatly reduced compared with unprocessed samples from the same batch (Fig. 4), and comparable to that of ALD coated samples from batches 4, 5 and 6. Maximum whisker lengths were also comparable. These results indicate that the thermal treatment alone brings about a reduction in whisker growth similar to that afforded by ALD processing. Although the whisker density on these samples had slightly increased after 40 months of storage to 39 ± 15 cm^{-2} (Fig. 9), the magnitude of the increase was not significant.

To elucidate the mechanism by which whisker growth is reduced for the thermally processed samples, focussed ion beam (FIB) techniques were used to prepare cross sections from selected Sn-Cu deposits. Figure 12 compares the deposit cross-sections of a thermally processed and an unprocessed control sample from batch 13, together with an ALD coated sample from batch 4 (500 nm coating), which was processed at approximately the same time. Comparison of the thermally processed sample (Fig. 12a) with the unprocessed control sample from the same batch (Fig. 12b) shows a

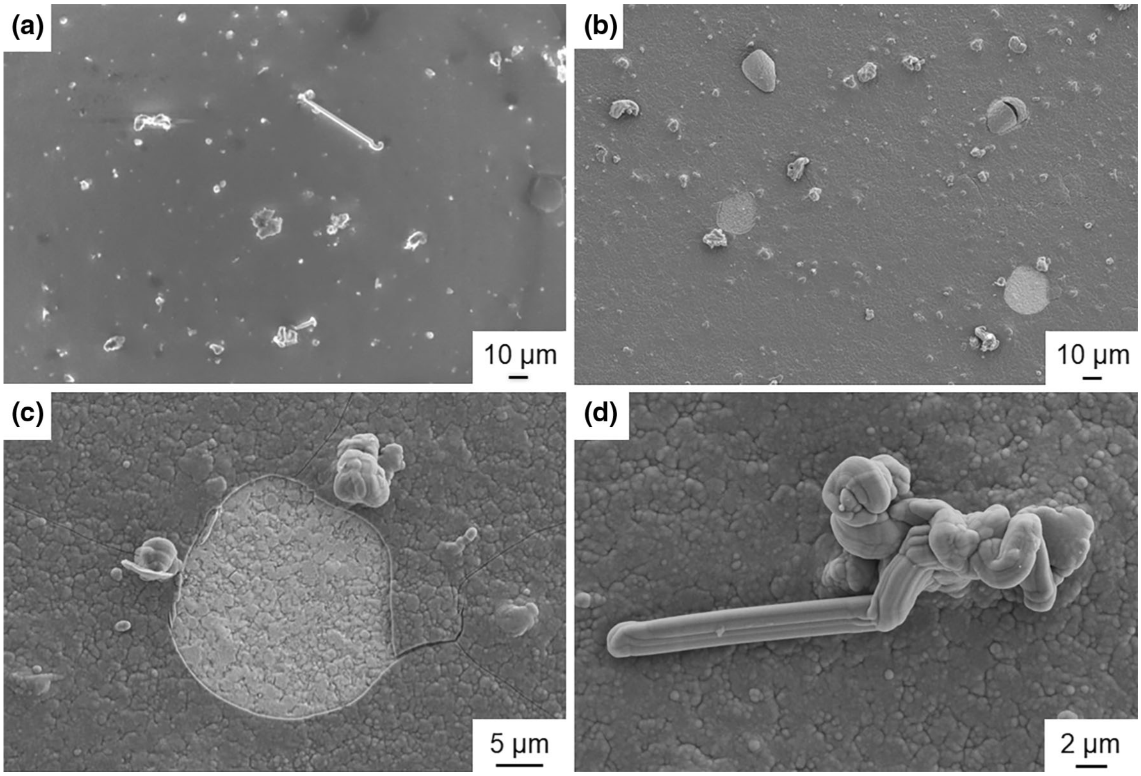

Fig. 8. Secondary electron images showing the surface of a sample from batch 8: (a) long filament whisker present on the ALD coated surface, (b) low magnification image showing failed regions of the ALD coating, (c) image showing the exposed Sn-Cu deposit surface in the failed region of the coating and (d) filament whisker encased in ALD.

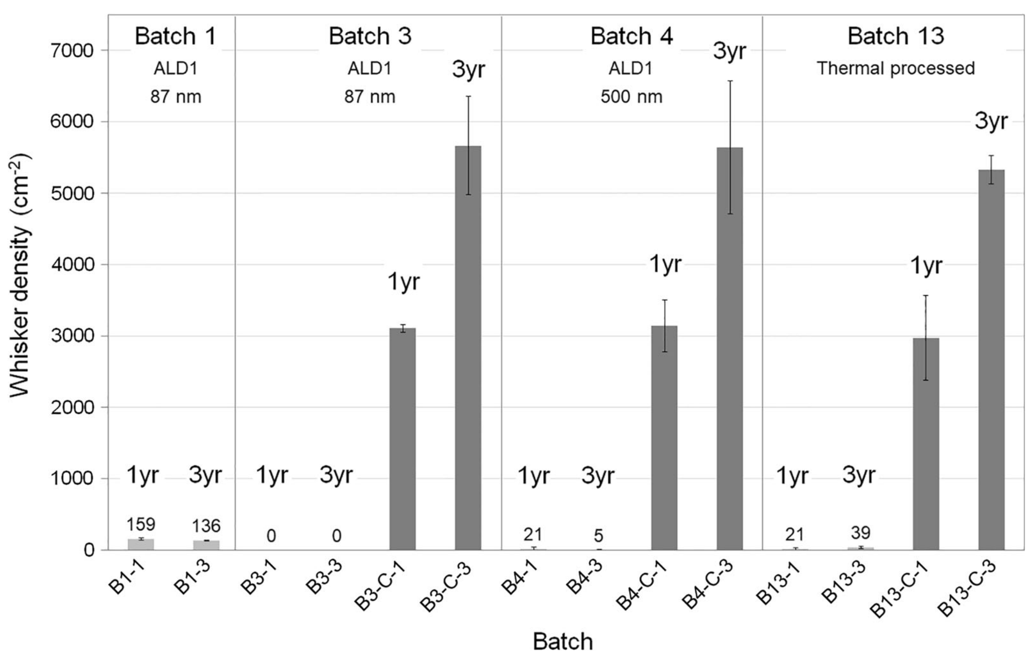

Fig. 9. Graph comparing whisker growth data after 3 years storage at room temperature for ALD processed and control samples.

distinct difference in the morphology of the Sn-Cu intermetallic compound (IMC). For the thermally treated sample, the intermetallic compound was relatively uniform in thickness, whilst that present in the untreated control sample was much less regular and varied greatly in thickness, which is consistent with previous analyses of Sn-Cu deposits stored at room temperature.[30,31] The morphology of the IMC present within the ALD processed Sn-Cu coating from batch 4 (Fig. 12c) was similar to that of

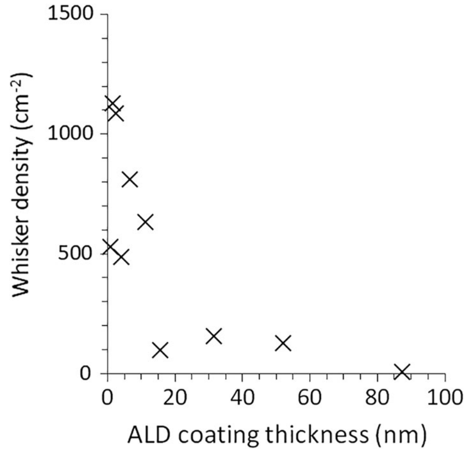

Fig. 10. Graph showing the effect of increased ALD coating thickness on the number of filament whiskers and odd shaped eruptions present on the deposit surface.

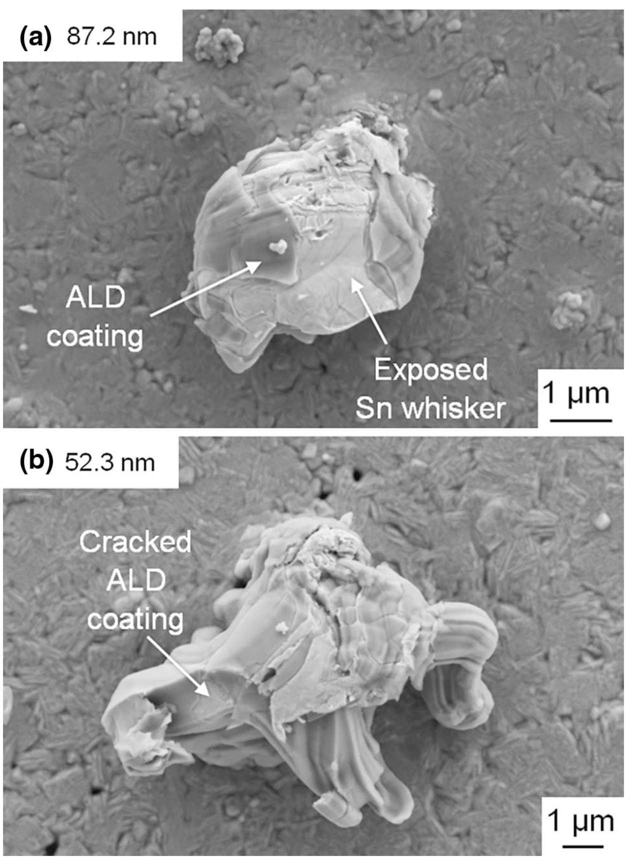

Fig. 11. Secondary electron micrographs showing the morphology of whiskers for batch 16 samples with different thicknesses of ALD coating applied: (a) whisker with a fractured ALD shell, 87.2 nm ALD coating and (b) whisker encased within a cracked ALD coating, 52.3 nm.

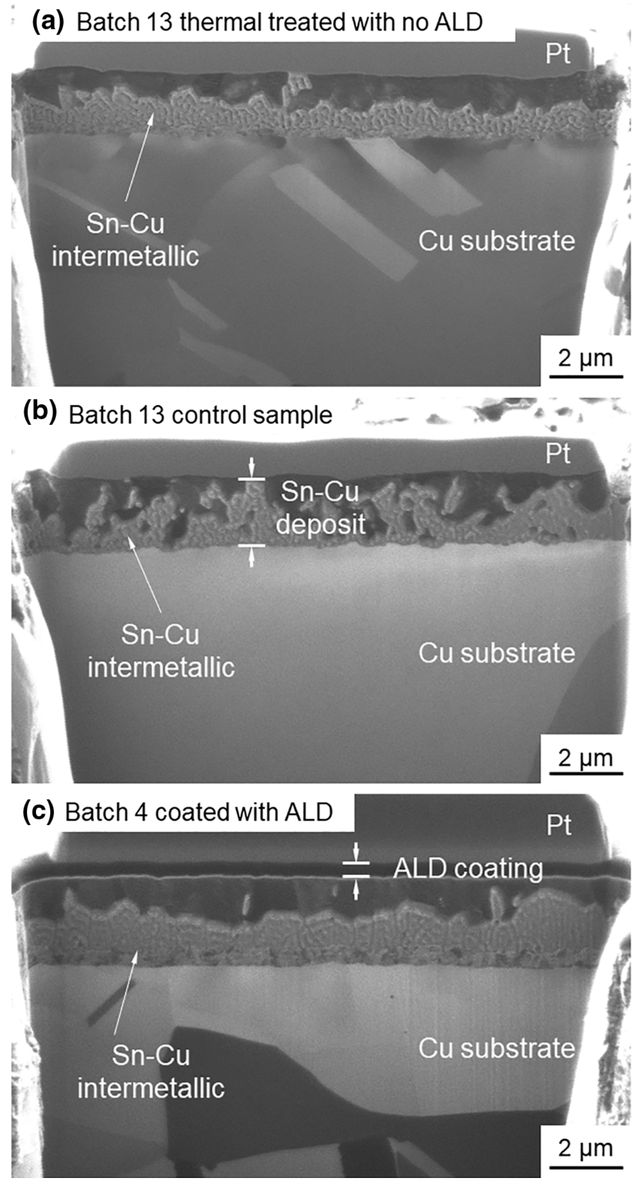

Fig. 12. FIB cross-sections showing the distribution of the Cu-Sn intermetallic: (a) thermal treated sample from batch 13 showing a relatively planar intermetallic phase, (b) unprocessed control sample from batch 13 and (c) an ALD coated sample from batch 4 also showing a relatively planar intermetallic phase.

the thermally processed sample. Generally, IMC having a planar morphology is considered less likely to promote whisker growth.[32] Furthermore, given that the thermal treatment (and ALD processing) was performed at 125°C, annealing of residual stresses within the electroplated Sn-Cu coating will serve to retard the onset of whisker growth; a post-electroplating thermal treatment at 150°C for 1 h is generally considered to be an effective whisker mitigation strategy.[33] Chason and co-workers proposed that whisker growth was suppressed both by the generation of tensile stresses in the coating upon cooling after thermal treatment, due to thermal expansion coefficient mismatches, and by the rate at which IMC growth occurs being reduced by the presence of a uniform thick IMC layer formed during initial processing.[6]

These results suggest that the elevated temperatures experienced during ALD processing contributed significantly to the levels of whisker mitigation observed for the ALD coated samples. It should be noted, however, that low levels of Al (6 at.%) were detected on the surface of the thermally treated test coupons using x-ray photoelectron spectroscopy (XPS) (Fig. 13). The binding energy of the Al 2p peak was measured to be 74.5 eV, which is consistent with previously reported values for Al_2O_3.[34] This suggests that a layer of Al_2O_3, comparable with those deposited during ALD processing, or an oxide film incorporating Al_2O_3, was present on the surface of the Sn-Cu deposit, possibly resulting from a slight leak of the reactive gases into the process chamber. Thus, the whisker mitigation observed for these samples may not simply be due to the influence of the thermal cycle.

Summary of Whisker Growth Trials

These investigations demonstrate that the ALD *process* can bring about very significant reductions in both whisker density and whisker length. ALD 1 (batches 1, 3 and 4) and ALD 2 (batches 5 and 6) appear equally effective at mitigating whisker growth. Results have shown that ALD mitigates against the growth of new whiskers (batch 3) and prevents further growth of whiskers that may have been developed prior to coating (e.g. batch 1). Even very thin ALD coatings (< 10 nm) were observed to bring about significant reductions in whisker density. However, it should be noted that the true influence of the ALD coating thickness on whisker growth may be masked by the influence of the thermal exposure and that thicker ALD coatings may indeed provide enhanced long-term resistance to whisker growth. Samples processed using ALD 3 (batches 7 and 8) showed greatly increased whisker densities, although there was no evidence of whisker growth after ALD coating nor did whisker growth seem responsible for the observed coating failures.

The slight increase in whisker density observed after 40 months of storage for samples from batch 13, which were subjected to a thermal treatment only, may be in response to compressive stresses built up over time within the Sn-Cu coating. In comparison, ALD coated samples from batches 1, 3 and 4 showed no increase in whisker densities compared with the original analysis. These results suggest that the ALD coating itself may act as a physical barrier to impede whisker growth, as shown by previous studies that have investigated the influence of applied surface oxides on whisker growth.[31,35,36] ALD processing may, therefore, provide greater long-term whisker mitigation than thermal treatment alone, which may simply retard the onset of whisker growth. Longer-term

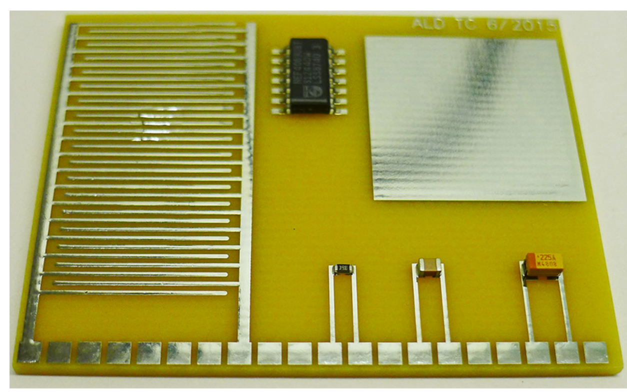

Fig. 14. Photograph showing the test board used to measure surface insulation resistance (SIR).

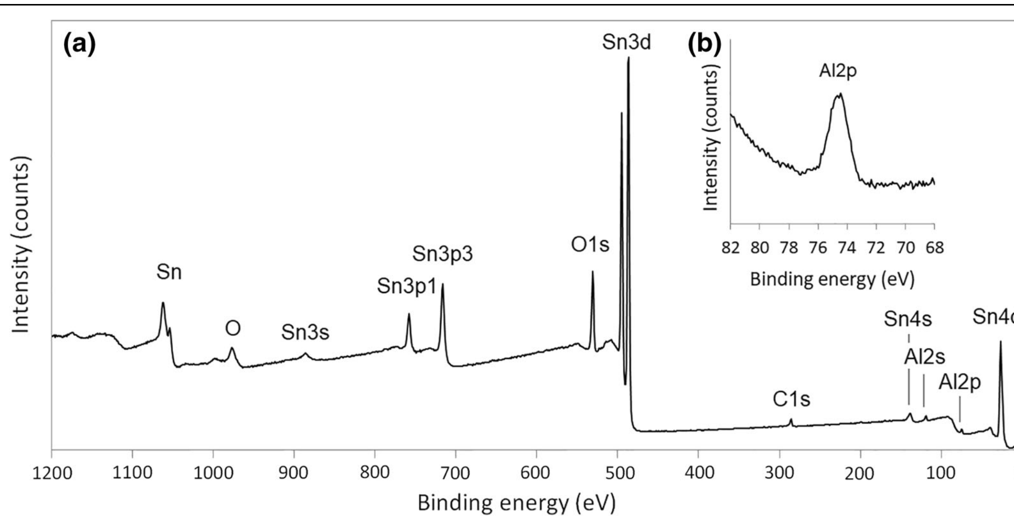

Fig. 13. XPS analysis showing the presence of Al on the surface of a thermally processed sample from batch 13: (a) survey scan and (b) high resolution scan of the Al2p peak.

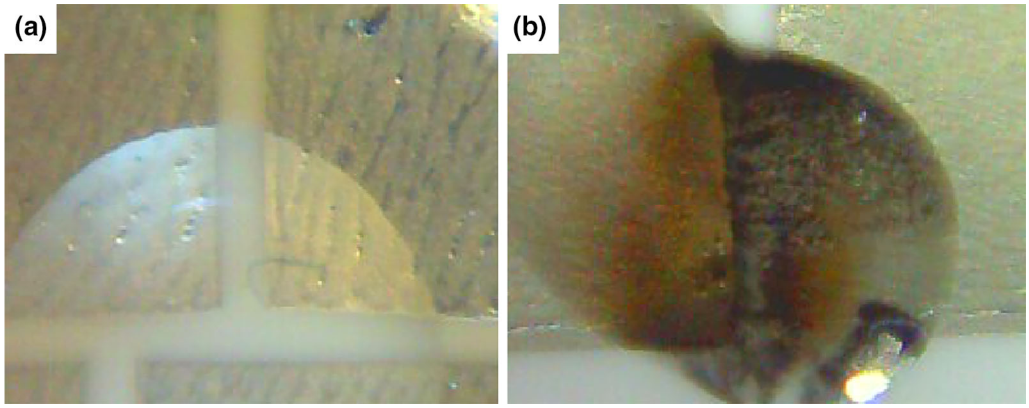

Fig. 15. Photographs showing the appearance of test coupons after the bias voltage test: (a) ALD coated sample and (b) non-coated sample.

monitoring of whisker growth will more clearly elucidate the benefits of the ALD coating.

Corrosion Protection

Although the present work has concentrated mostly on the tin whiskers issue and the development of ALD recipes, the corrosion protection properties of the ALD thin films were also evaluated. Surface insulation resistance (SIR) testing was undertaken using test boards manufactured with a finger pattern structure in accordance with IPC-TM-650, as shown in Fig. 14.

Conventional environmental tests have not yet been performed, but initial basic tests have been performed on selected samples to observe the influence of the ALD coating. These were carried out using a pure silver conductor on a ceramic substrate with a bias voltage applied to readily evaluate the influence of the ALD coating. The purpose of this was to obtain an initial indication of the properties of the new ALD recipes. Figure 15 shows two similar boards with electrodes under a tap-water droplet with a 2.5 V bias voltage over a ca. 0.5 mm gap after 15 min. The uncoated samples started to form dendrites immediately and caused a short circuit in a few minutes while the coated sample maintained its original appearance.

CONCLUSIONS

This study shows that ALD processing has the potential to function as a highly effective whisker mitigation strategy. For samples processed using ALD 1 and ALD 2, very significant reductions in whisker growth were demonstrated compared with unprocessed control samples. Furthermore, no whisker growth was indicated to have occurred from batch 3 samples after 3 years storage at room temperature. Potential factors affecting the effectiveness of the whisker mitigation are indicated to be the pre-treatment and the thickness of the applied coating, although for samples processed using recipes ALD 1 and 2 there does not, to date, appear to be any benefit in increasing the coating thickness from 87 nm to 500 nm. Further studies are required, both to optimise the properties of the applied ALD coating and to clarify whether the coating itself acts as a physical barrier to prevent the growth of whiskers.

Our investigations suggest that the ALD process is a potential option to provide both environmental protection and whisker mitigation for electronic assemblies and components for space applications (e.g. satellites), which require that IC packages do not out-gas as conventional polymer packages typically do. The ALD coating process provides both degassing and encapsulation for the components and substrate because the deposition is made under vacuum and at elevated temperatures.

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