WHISFUSE Project

Current capacity and effects of vacuum of tin whiskers

European Space Agency

ESR. Executive Summary Report

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1. Contextualization of the WHISFUSE project

The **WHISFUSE** project responds to the **ESA tender** (ESA AO/1-11458/22/NL/FE) for the study on the **"Current capacity and effects of vacuum of the tin whiskers"**.

Lead free component terminations and solder finishes are widely available in the industry and are used in almost all COTS EEE parts (COTS: Commercial Off The Shelf / EEE: Electrical, Electronic and Electro-Mechanical). Due to the increasing demand for these materials and finishes in space flight equipment and instruments, this investigation has been required to assess the potential risks associated with pure or high percentage tin (Sn), which is known to have the potential to grow tin whiskers. However, this activity has not focused on understanding the growth mechanisms of tin whiskers. Rather, **the main aim has been to determinate if the vacuum environment has any effect on tin whisker growth, and to identify if there is an increased risk of short circuits in space flight electronics during a mission**. Vacuum testing, simulating the space environment, has been the baseline to evaluate if the vacuum environment can slow or prevent the fusing of tin whiskers. Thus, the WHISFUSE project has had **two main technical objectives**:

- **To determine if vacuum influences the likelihood and speed of tin whiskers growing.**
- **To determine the characteristics and current carrying capacity of tin whiskers in vacuum compared to normal, ambient atmosphere.**

For both objectives, comparisons with ambient conditions have been used to establish a baseline, from which any differences have been declared and assessed. To accomplish these objectives, an intensive work plan has been required, involving a multidisciplinary technical team composed of three different organizations: CIDETEC (Spain), BME (Budapest) and Łukasiewicz-IMiF (Poland).

2. Experimental procedure

2.1 Samples preparation

__Substrates.__ Based on the literature data and the extensive experience of the partners of WHISFUSE, pure copper (DIN classification E-Cu57 with Cu > 99.9%) substrates were selected as an appropriate support material to promote the formation of Sn whiskers. The Cu samples were mirror polished and had square geometry (30x30x2 mm), with a small flange (15x5 mm) to facilitate manipulation during Sn deposition. These Cu substrates were annealed at 200 °C for 3 h to reduce eventual internal stress.

__Sn-deposition methods__. Sn samples were fabricated applying 3 different methods on Cu substrates:

- Electrodeposition: Sn-matt /Sn-bright (thickness)
- Physical vapor deposition (PVD):
- Soldering:

Whisker growth / Aging conditions. The specimens were aged in vacuum and ambient conditions to assess the key factors affecting the growth and electrical properties of the whiskers.

Table 1. Performed aging test during WP2.

Aging test	Ambient atmosphere (CIDETEC)	Vacuum conditions (BME)
Isothermal	**A1** (T = 90 °C, RH = 50 %)	**V1** (T = 50°C, P = 8.3x10^{-6} mbar)
Cyclic	**A2** (T = -30 °C to 90 °C, RH = 50 %)	**V2** (T = 25 °C to 125 °C, P=8.3x10^{-6} mbar)

2.2 Structural and chemical analysis

The developed whiskers were analyzed by scanning electron microscope (SEM, Thermo scientific Quattro S), transmission electron microscope (TEM, JEOL JEM-2100), and X-ray diffractometer (XRD, Bruker D8). During the aging test, samples were transported from each chamber to SEM microscope. In the case of ambient tests, samples were extracted from the chamber one by one (in the shortest time possible) to minimize the overall time spent in an uncontrolled environment. In the case of samples aged at vacuum, after a sample was taken out from the vacuum condition for investigation, it could not return to the test since the oxidation of the Sn is relatively fast. So, the test was always such a “non-return” type, which means that the investigated samples were not returned to the vacuum chamber after the SEM investigation (so at each checkpoint, different samples were investigated). The samples were taken for investigation directly after being removed from the vacuum chamber. N_2 flushing was applied during the sample removal from the chamber in order to avoid the oxidation of the samples, which stayed further in the test.

2.3 Electrical testing

Measurements in vacuum have been made by using Kleindiek micromanipulators, SEM/FIB system HELIOS NanoLab 600 Dual Beam and KEYSIGHT Semiconductor Device Analyzer B1500 (the assembly scheme is shown in Figure 1). A tin whiskers plate is placed together with micromanipulators inside the vacuum chamber of SEM/FIB system.

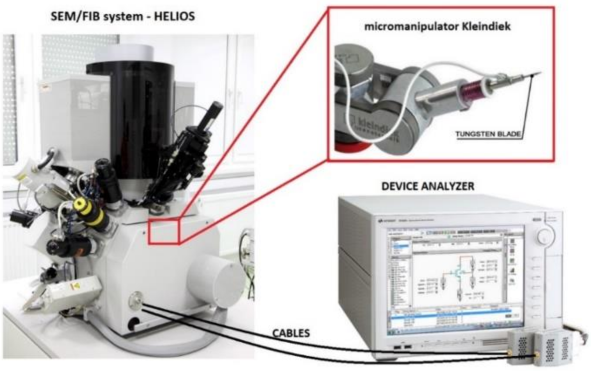


Figure 1. Measurement system in vacuum.

Two tungsten blades (needles) are used to establish electrical connection. Blade_1 touches the tin plate in whiskers vicinity, and the Blade_2 touches the lifted end of the whisker (see Figure 2). If there is an oxide at the whisker's surface, it behaves like a capacitor which has to be broken by sufficiently high voltage for current to flow. Between the blade_1 and the tin plate there is no oxide, since a surface has been scratched with the tip. The tin plate has been grounded, so the blade touching plate has to be in contact with ground also.

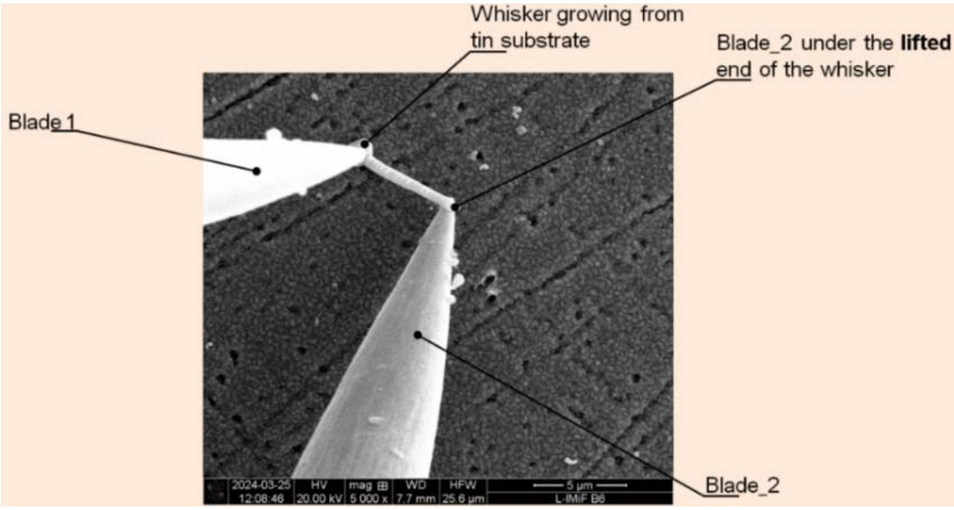


Figure 2. Tungsten blades of Kleindiek micromanipulators for electric connection of a whisker in vacuum.

In a vacuum chamber, heat can be radiated by electromagnetic emission or transferred into the substrate at one end of the whisker. No convection is possible. Current density of the whiskers can be lower.

3. Results and discussion

3.1 Comparison of whiskers growth in ambient atmosphere vw vacuum condition

3.1.1 Electrodeposited samples

The bright and matt electroplated Sn layer showed different whisker susceptibility during the ambient atmosphere and vacuum aging tests. Matt coatings produced higher density of whiskers at vacuum conditions compared with ambient atmosphere aging test. The maximum whiskers length is achieved during vacuum isothermal test, where reported values are between 15-65 µm. The type of whiskers observed is different in each case, at ambient isothermal exposure, "column and needle" whiskers are observed, while at vacuum isothermal test, mostly "hillocks and filaments" are observed.

In the case of bright coatings, whiskers were detected only during ambient aging tests, and no whiskers have been observed during vacuum aging tests.

3.1.2 Soldering samples

Similar behavior to electroplated bright coatings has been observed in the case of soldering coatings. During ambient aging tests whiskers are detected on soldering samples surface (similar behavior between isothermal and cyclic exposure), whereas during vacuum aging test no whiskers have been found on the samples surface.

3.1.3 PVD samples

In the case of PVD coatings, during aging tests at high temperatures, no whiskers have been found as the interdiffusion between Sn and Cu was fast enough to degrade the coatings before the whiskers formation. However, during V1 aging test (at 50 °C), no samples degradation was observed and PVD samples produced 2-3 orders of magnitude more whiskers than the rest of the samples in any test.

3.1 Structural characterization of whiskers

3.1.1 TEM investigation of the vacuum-grown whiskers

The morphology of the whisker grown in vacuum conditions was unique compared to the structure of the ambient grown whiskers. Figure 3 presents a PVD samples which spent 250 h in vacuum and were investigated ~3 weeks later. An interesting phenomenon was revealed that the end part of the whiskers had an unusual block-like structure with a totally smooth surface, while at the root of the whiskers, they had a twisted body with grooves, which is the more usual whisker morphology.

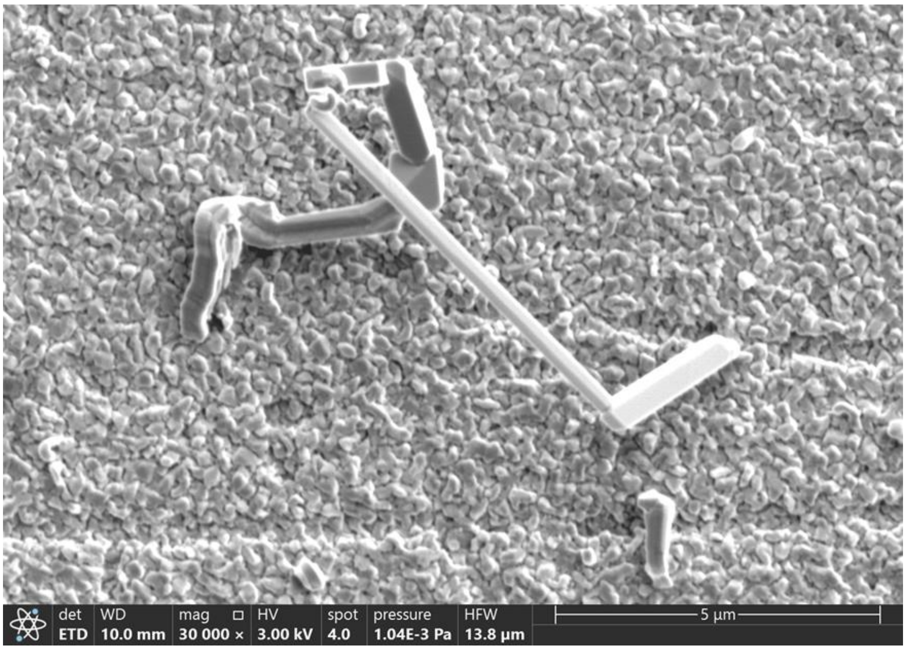


Figure 3. Sn whisker morphologies on a PVD sample in vacuum and ambient atmosphere.

Comparing the results of the twisted and block-like whiskers, it can be concluded that the twisted whiskers contained more Cu_6Sn_5 inclusions than the block-like one. The concentration was ~4-5 at. % in the block-type whiskers and 8-12 at. % in the twisted whiskers (measured on the full TEM samples). The presence of Cu_6Sn_5 in the whisker body is not uncommon for very thin PVD layers and can be explained by the interface flow mechanism. The extremely high stress caused by the growth of the Cu_6Sn_5 intermetallic compound (IMC) layer within the thin Sn layer could initiate the interface flow mechanism between the Sn layer and the IMC layer. This suggests that Sn and Cu (or Cu_6Sn_5) atoms/molecules flow along a fluid-like viscous layer at the interface of the Sn film and the IMC layer. They constantly migrate within this viscous layer toward regions of lower stress, which are the roots of the whiskers, since whisker growth is a stress relaxation mechanism [1]. The Cu_6Sn_5 inclusions in the body of the whiskers could have a major role in the formation of twisted whiskers [2].

No correlation was found between the orientations of the Sn and Cu_6Sn_5 crystals and the shape of the whiskers. However, it must be noted that the crystal structure of the block-like whiskers exhibited a slightly tighter grid structure (with Sn atoms being closer to each other) than that of the twisted ones. The development of block-like Sn whiskers in vacuum was probably influenced by the lower amount of Cu_6Sn_5 present in their body.

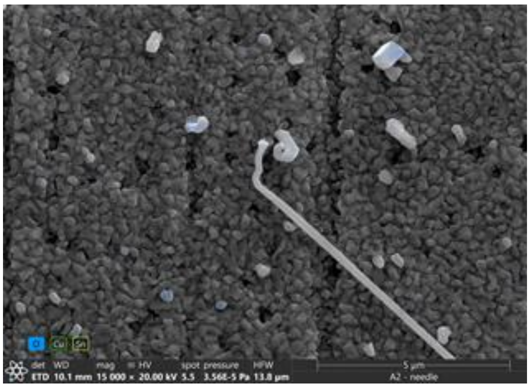
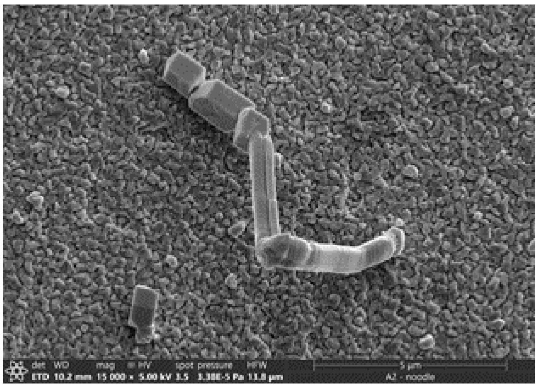
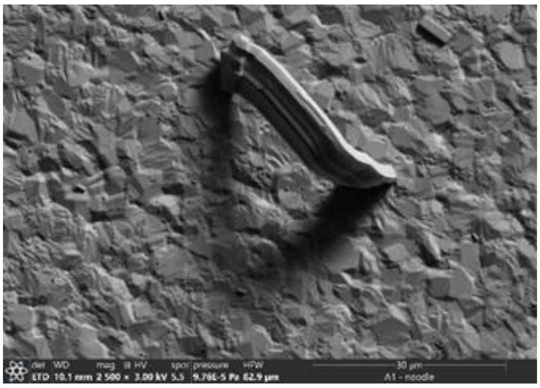
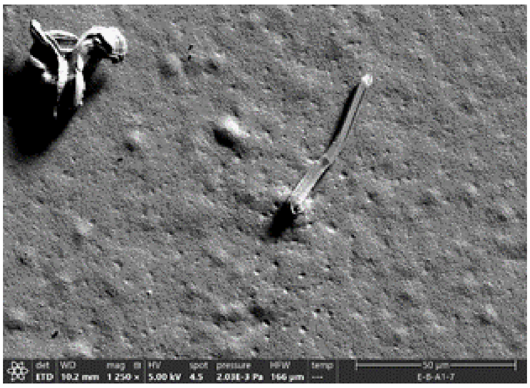
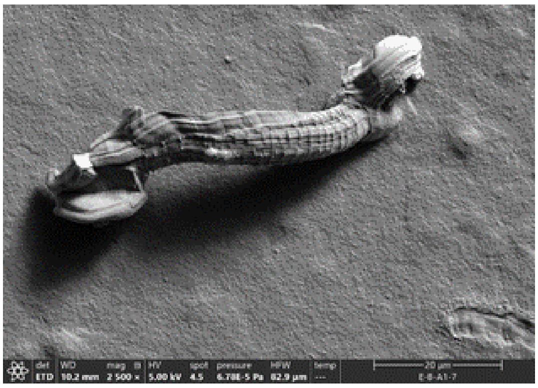
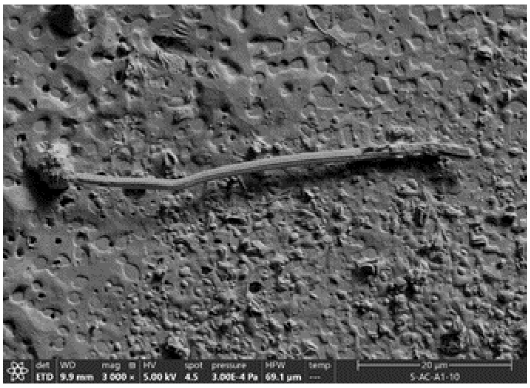
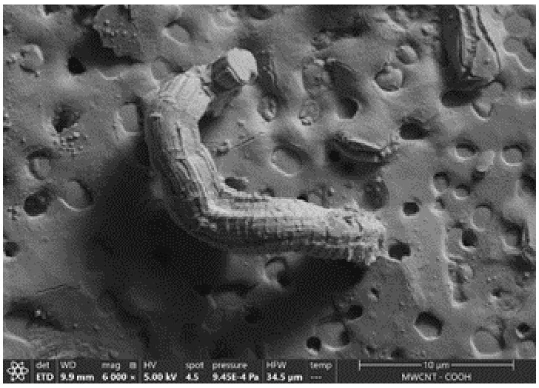
In the literature, the formation of the grooves on the whisker body was explained by the grain boundary cracking phenomenon at the root of the whiskers [3, 4], which we did not observe in our study. So, it can be concluded that the grain boundary cracking at the whisker roots can be the cause of groove formation, but it is not the only one. We believe that it could be related more to the surface oxidation of the whisker itself, but we could not prove it during the study**.**

3.1.2 Elemental composition of whiskers (oxygen content)

- Tin coatings prepared by the PVD method and aged under vacuum conditions exhibit low oxygen content (4-7 at. %).
- In the electrodeposited coatings, oxygen content is slightly lower on the whisker body of the sample from Sn-matt coatings aged in a vacuum compared to non-whisker area. Conversely, in the sample from Sn-bright aged at ambient conditions, there is slightly more oxygen on the whisker body from than in non-whisker area, with oxygen content fluctuating between 16 and 24 at. %.
- It is also noteworthy that oxygen concentration is considerably higher – up to 60 at.% - in samples fabricated via the soldering process and aged under ambient conditions. If an SnO_2 layer is formed, it tends to appear on pit-hole tin surfaces, suggesting that specific factors related to the tin-deposition method (electrochemical process duration, specific deposition temperature, etc.) play a crucial role.

Therefore, it is evident that sample origin and aging conditions significantly influence tin coating oxidation. However, it should be noted that data statistics are currently insufficient to fully confirm the above hypothesis.

Table 2. Whiskers on surface of Sn coatings.

	Needle type whisker	Nodule type whisker
PVD		
Electrodeposited matt		
Electrodeposited bright		
Soldering		

3.2 Electrical characterization

As a summary of the results, comparison between electric parameters of whiskers is presented, considering the manufacturing method of Sn coating. Figure 4 shows a diagram of breakdown voltage (U_{br}) vs whisker diameter (D).

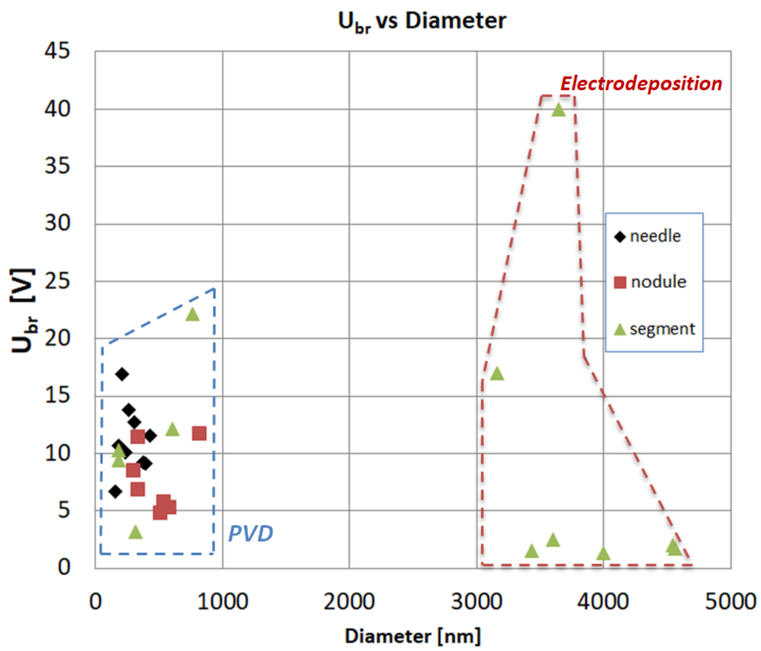


Figure 4. Breakdown voltage vs whisker's diameter diagram.

Some segment whiskers show significantly lower breakdown voltage, mainly for whiskers obtained in PVD coatings and in electrodeposited bright samples. The differences in breakdown voltage are induced by presence or lack of oxide at whisker's surface. When the oxide layer is thinner (or neglectable), the breakdown voltage is low compared to the breakdown voltage on oxidized samples. In addition, some whiskers from electrodeposited matt samples (aged at vacuum conditions) present values of breakdown voltage. It is probably due to oxide layer formation (after vacuum ageing), when samples were transporting and waiting for measurements in air. Additionally, segment whiskers coming from the electrodeposited samples had bigger diameter (red mark in Figure 4) but the breakdown voltage was not significantly higher.

Figure 5 shows a diagram of breakdown current (I_{br}) vs whisker diameter (D). Some segment whiskers show significantly higher breakdown current, and these whiskers correspond to those that have been generated on electrodeposition coatings (red mark in Figure 5). The difference in current capacity seems to be induced by whisker's diameter. The higher diameter gives the higher current capacity. Current capacity of segment type Sn whisker could be higher than 100 mA. Segment whiskers usually do not fuse. Their electric connection is broken due to change in shape induced by thermal deformation or melting and solidification. Whiskers that were grown on electrodeposited samples are mainly thick segment type. In consequence their mean current capacity is higher than PVD whiskers.

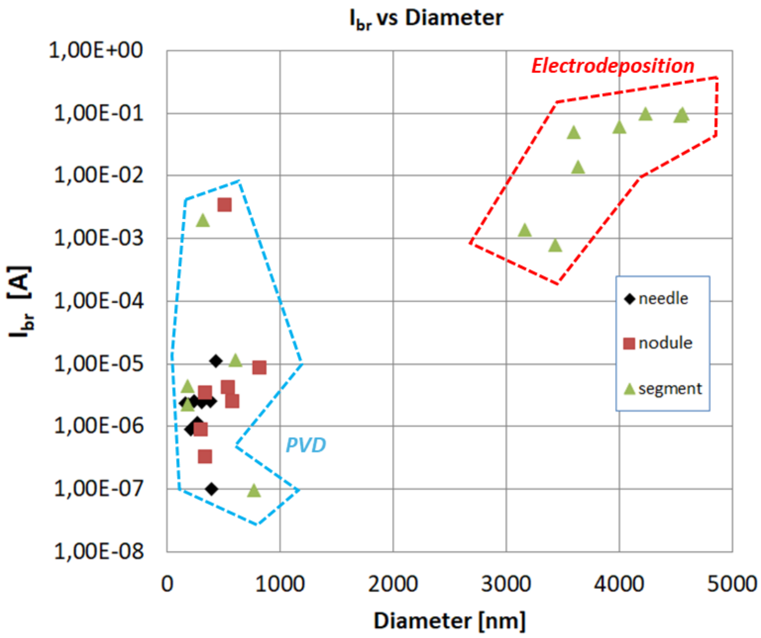


Figure 5. Breakdown current vs whisker's diameter diagram.

4. Conclusions

The main conclusions obtained during project execution are listed attending to each activity performed:

Selection of materials, manufacturing methods and samples preparation

- Three different manufacturing methods for applying Sn coatings on Cu-rich substrates were employed: electrodeposition (producing both Sn-bright and Sn-matt deposits), soldering (using Sn99Cu1 and SAC307 alloys) and PVD (Physical Vapour Deposition with 99.99% Sn). Each method was optimized, considering Sn coating microstructure, thickness and surface finish.

- Each manufacturing method produced Sn coatings with varying thicknesses, each differing in order of magnitude due to method limitations or inherent characteristics. The thinnest coating was obtained by PVD (approximately 0.3-0.5 µm), in the electrodeposition samples (both bright and matt conditions) approximately 5 µm thickness was achieved, and in the soldering samples the thickest coating was obtained, around 10 - 50 µm (more irregular than the previous one).

- The observed microstructure of each Sn coating is as follows:
    - PVD coatings exhibit a granular structure with tightly adjacent crystallites; and there are areas where the Sn coating is not continuous.
    - Electrodeposited Sn coatings offer two distinct microstructures: Sn-matt with coarse microstructure (a "rocky" microstructure") and Sn-bright with refined microstructure (a stone-like mosaic or a sandy patch)

    - Soldering coatings show pit-hole surfaces.

Whisker growth/aging tests

- Results from the aging tests, considering the Sn coating manufacturing method, are as follows:
    - Electrodeposited bright and soldering samples: No whiskers were detected under any vacuum condition (whether isothermal or cyclic). However, when these Sn coatings were exposed to ambient atmosphere, long whiskers were observed, reaching a maximum length of 60 µm.
    - Electrodeposited matt samples: Whisker growth was observed under all conditions, at vacuum or ambient atmosphere, and isothermal or cyclic condition.
    - PVD samples: Whisker growth could only be studied under vacuum isothermal aging test. At high temperatures, the Sn coating suffered degradation. However, where whisker growth occurred, whisker density was 2–3 orders of magnitude higher.
- There appears to be a strong correlation between Sn layer thickness and whisker formation: the thinner the Sn layer, the easier and faster whisker growth occurs, a trend observed exclusively in vacuum conditions. It can be said that the Sn-layer thickness and/or Sn coating characteristics influence the formation of whiskers. However, under ambient conditions, this relationship could not be studied due to, as previously noted, the PVD Sn-layer undergoes degradation at high temperatures.

Structural characterization of whiskers

- The morphology of the whiskers was different depending on whether they grew under vacuum or ambient conditions. In vacuum, block-like whiskers with flat surfaces were grown, and the development changed to twisted whisker bodies with grooves after they were removed from the vacuum chamber. This morphology was typical for the PVD and electrodeposited matt Sn layers (only these samples produced whisker in both the V1 and V2 tests).
- Comparing the results of the twisted and block-like whiskers, it can be concluded that the twisted whiskers contained more Cu_6Sn_5 inclusions than the block-like one. The concentration was ~4-5 at. % in block-type whiskers and 8-12 at. % in twisted whiskers (measured on the full TEM samples). The presence of Cu_6Sn_5 in the whisker body is common for very thin PVD layers and can be explained by the interface flow mechanism. No correlation was found between the orientations of Sn and Cu_6Sn_5 crystals and the shape of the whiskers. However, it must be noted that the crystal structure of the block-like whiskers exhibited a slightly tighter grid structure (with Sn atoms being closer to each other) than that of the twisted ones. The development of block-like Sn whiskers in vacuum was probably influenced by the lower amount of Cu_6Sn_5 present in their body.
- The results of EDS measurements show:

- Whiskers from soldering samples aged at ambient conditions, had the highest oxygen content values of all analyzed whiskers.

- Whiskers from electrodeposition process, there was slightly less oxygen in vacuum aged whiskers or somewhat more oxygen on the whisker body of ambient aged whiskers compared to the out-of-whisker areas. Therefore, it is clear that the sample origin and its aging conditions significantly affect the Sn coating oxidation. However, it should be noted that the data statistics are insufficient to confirm the above hypothesis fully.

Electrical testing of whiskers

- The differences in breakdown voltage are induced by presence or lack of oxide at whisker's surface. When the oxide layer is thinner (or neglectable), the voltage of breakdown is low in comparison to situation when oxide is present. In addition, two whiskers from electrodeposited matt samples, aged at vacuum conditions, present the values of higher breakdown voltage. In this case the probable explanation is that oxide was formed after vacuum ageing, when samples were transporting and waiting for measurements in air. Segment whiskers coming from the electrodeposited samples had bigger diameter

- The difference in current capacity seems to be induced by whisker's diameter. The higher diameter gives the higher current capacity.

- In vacuum whiskers were touched by tungsten needle. If there was an oxide at whisker surface, a capacitor was formed and charged by current until insulator breakdown appeared. In consequence I-V characteristics started at low current and at certain voltage they showed abrupt breakdown of oxide and the whole whisker (breakdown was immediately after a kink).

- The breakdown voltage fluctuates from 2 to 17 V depending on oxide thickness at whisker Surface. The current capacity fluctuates from µA up to above 100 mA.

5. References

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