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Tin Whiskers Behaviour on Immersion Tin Surface Finish at High Temperature and Humidity Condition

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Abstract – *Towards green technology application in electronic industry, pure tin as a Pb-free component lead finish become a popular choice, yet facing the problem with tin whisker formation. Tin whisker has turned into a serious concern in the electronic industry where it may cause short circuit to electronic components. It has been long believed that residual stress generated by external stresses, internal stresses, and temperature is the root cause of whisker formation. In this study, the effect of temperature and humidity on the formation and growth of tin whiskers was studied focusses on immersion tin surface finish only. The samples were prepared by a chloride-based bath and held at 30°C/ 60% RH (normal condition) and 55°C/ 85%RH (severe condition) for several time intervals. The morphology of tin whiskers on an immersion tin finish on copper substrate was observed by FESEM. The whiskers length was measured using JEDEC-Standard JESD22-A121A. The results showed that, tin whiskers formed in various types including nodules, straight, twist, bent, and spiral shape with striations along its circumference for normal condition, but only nodule- and spiral-type of whiskers only can be observed for severe conditions. Surprisingly, normal condition produce longer whiskers length as compared to the whiskers formed when the surface exposed to the severe condition.*

Keywords: Immersion Tin Surface Finish, Tin Whiskers, Copper Substrate, JEDEC Standard JESD22-A121A, JEDEC Standard JESD20-1A

1.0 INTRODUCTION

The strategies of using pure tin or nearly pure tin for lead-free materials has created a new challenging issue with the formation of tin whiskers on tin plating surface [1]. Tin whiskers can be defined as spontaneous single crystals that grow out from the surface finish which is caused by the formation and growth of intermetallic compounds (IMCs) that would generate the compressive stress in tin surface finishes [2-6]. The tin whiskers phenomenon has been documented since 1940s by the National Aeronautics and Space Administration (NASA) [7]. Many studies have been conducted to ensure better understanding regarding the fundamentals of tin whiskers formation thus can establish the mitigation method for whiskers to growth.

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Many researchers agree that tin whiskers can cause a short circuit, hence breaking off the components, resulting in the degradation of electrical or mechanical parts performance [3, 4,8].

The formation of tin whiskers was influenced by several factors, including IMCs formation [9-11], external stress [12], plating thickness [9,11], temperature and humidity [13], and the oxide layer [11]. Chason et al.[10] proposed that many kinetic processes need to be considered in order to study the whiskers growth such as microstructural evolution, inter-diffusion of Sn-Cu, IMCs growth, stress generated in Sn, and whiskers nucleation and growth because the study involved complex multilayer structure with multiple phases; Sn, Cu, IMC, oxide.

Based on the published literatures [15-19], high humidity condition promotes whiskers growth due to the oxidation on tin plating which is related to the formation of oxide layer on tin coating. According to the oxide theory, oxide layer on tin coating inhibit the stress relaxation from internal stress thus enhance the stress to tin films and leads to formation and rapid growth of tin whiskers. The internal stress was generated by the growth of IMCs.

Crandall et al.[15] has study the effect of high relative humidity on whiskers growth in tin coating with Si and brass substrate respectively in the 65 - 90% RH range. According to his study, he found that whiskers density increase dramatically with 85% RH produced highest whiskers densities for tin on Si substrate. Su et al.[16] manipulated the relative humidity at constant temperature (60°C/85%RH and 60°C/93%RH) in her study. She found that higher relative humidity has higher whiskers population and longer maximum whiskers length compare to lower humidity. Higher relative humidity condition has higher oxidation rate thus oxidation has been thought to accelerate the whiskers growth.

Most of the previous studies focused on whisker growth on electroplated tin. However, electroplating method itself contains residual stress. The residual stresses arising during the electroplating process appear as a strong handicap for the fabrication of reliable microstructures or microsystems due to the current induced to the system [20]. Immersion plating on the other hand is a current-free deposition of metal on the substrate and relatively lower residual stress as compare to electroplating. The main purpose of this research is to investigate and understand the effects of high temperature and humidity on tin whisker formation and growth on immersion tin surface finish.

2.0 METHODOLOGY

The substrates material used in experiment was a commercial oxygen-free pure copper with 1 mm thickness, 35 mm width and 50 mm length. Plating process by immersion was conducted by following the reference [12]. Prior to plate with tin, the substrate was prepared by the following procedures; ground with Silicon carbide paper (800 grit) to remove the oxide layer; immersed in 2-Butoxyethanol solution at 70 - 90 °C for 5 minutes to remove dirt; cleaned in NaOH solution at 60 °C for 3 minutes for surface degrease; etching in 10% H_2SO_4 solution for 1 minute to deoxidize the surface; immersed in palladium chloride solution for 3 minutes to activate the surface. The plating process was conducted in the stainless steel plating bath set up at a constant temperature. The plating bath was prepared by dissolving the 20 g/L of stanneous chloride as a tin precursor, 16 g/L of sodium hypophosphite as a reducing agent, 37 ml/L of hydrochloric acid (37% ml), 50 ml/L of Sulfuric acid (50% ml), 200 g/L of thiourea, and 5 ml/L of phenolsufonic acid in 1L distilled water. The immersion tin process was conducted at temperature of 75 °C and the deposition time was 8, 12, and 20 minutes in order to obtain 1.2, 1.5, and 2.3 µm of tin thickness respectively.

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The samples were then stored in the humidity chamber for certain time interval which is 1, 4, 8 and 12 weeks under the environment condition of 30°C/60%.RH (normal condition) and 55°C/ 85% RH (severe condition). The observation of tin whiskers behaviour at every time interval was conducted exactly at the same samples in order to achieved consistent parameters and minimizes unwanted factors that varied the results.

The cross sectional samples were observed by using optical microscope with 500x magnification equipped with image analyser (Nikon-Eclipse LV150). The coating thickness was measured by using i-Solution Lite software attached with image analyser. The surface morphology analysis of tin whiskers and IMCs was conducted by using field emission scanning electron microscopy, FESEM (Supra-35VP, Carl Zeiss) to investigate the tin whiskers types and IMCs formation within tin layer and copper substrate and energy dispersive x-ray (EDX) to measure the chemical composition of tin whiskers and IMCs. All whiskers length was measured from the top view of FESEM micrograph using i-Solution/ Lite software. The measurement of whiskers length complied with the JEDEC standard No. 22-A121A [3] where the whiskers length was determined by taking an average value of five longest whiskers length per sample.

IMC was analysed from the top and view of FESEM micrograph to obtain clearer results. IMCs analysis was conducted on Cu-Sn and Cu-Ni-Sn samples to verify the existence of IMCs between the copper substrates and tin or Ni layer due to the reaction occurs during plating process. The sample was etched for 5 minutes in the etchant solution of nitric acid (40 gm), sulfamic acid (2 ml), fluoroboric acid (1.5ml), and distilled water (60 gm). FESEM was used to observe IMCs from the top and EDX was used to determine the chemical composition of IMCs.

3.0 RESULTS AND DISCUSSION

The whiskers behaviour was analysed by surface observation of the plated samples exposed under the condition of 30°C/60%.RH (normal condition) and 55°C/85% RH (severe condition) by using FESEM. The exact samples were analysed for four different time interval which is 1, 4, 8, and 12 weeks. Fig. 1 shows the FESEM micrographs of tin whiskers (some of them are pointed by solid arrows) and CuO flowers (hollow arrows) observed on the immersion tin surface finish for 1.2 µm coating thickness after (a) 1 week, (b) 4 weeks, (c) 8 weeks, and (d) 12 weeks under the normal environment condition (30°C/60% RH). It can be clearly seen that the tin whiskers of sample exposed for 4 weeks (Fig. 1b) are longer than the sample exposed for 1 week (Fig. 1a). The whiskers kept growing at week 8 (Fig. 1c) and longest for week 12 (Fig. 1d).

Fig. 2 shows the FESEM micrographs of tin whiskers (solid arrows) and CuO flowers (hollow arrows) observed on the immersion tin surface finish for 1.2 µm coating thickness after (a) 1 week, (b) 4 weeks, (c) 8 weeks, and (d) 12 weeks under the severe environment condition (55°C/85% RH). The similar growth behaviour of tin whiskers is observed on the samples exposed under severe condition as compared to normal condition where the tin whiskers were grown longer with exposure time. Fig. 3 shows the behaviour of tin whiskers formed on the samples with different coating thickness after 12 weeks of exposure under normal and severe conditions. The results showed that thicker tin coating thickness produced longer whiskers length for both exposure conditions.

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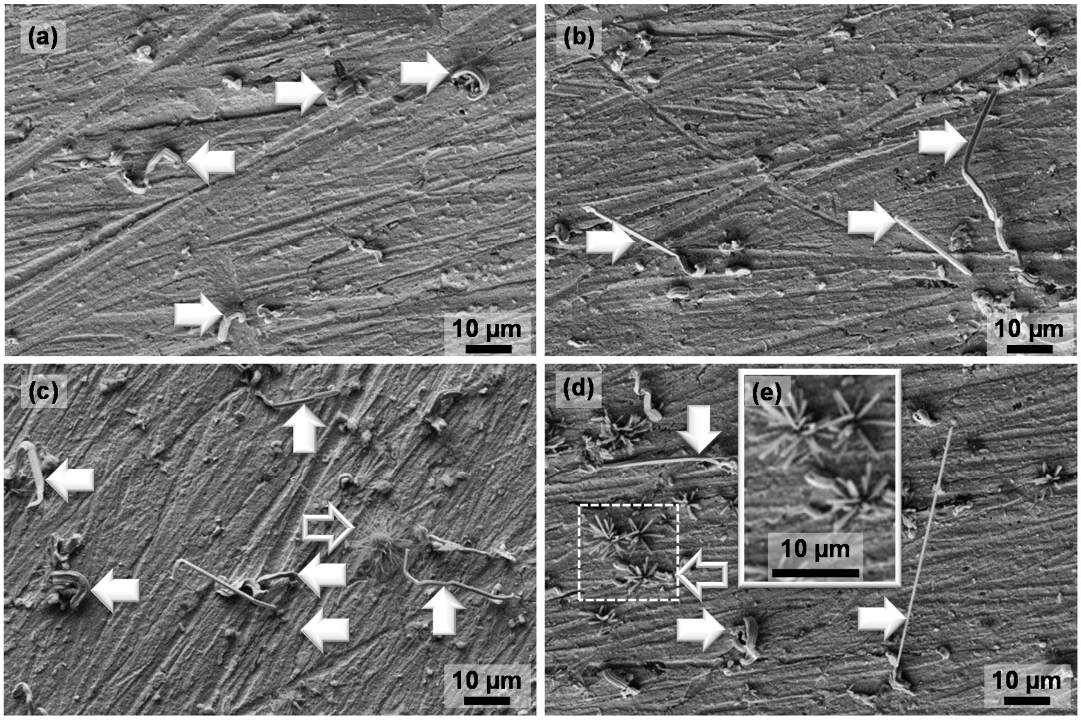

Figure 1: FESEM micrographs of tin whiskers (solid arrow) and CuO flowers (hollow arrow) observed on the immersion tin surface of 1.2 µm coating thickness after (a) 1 week, (b) 4 weeks, (c) 8 weeks, and (d) 12 weeks under the environment condition of 30°C/60%.RH. Image (e) is high magnification image of CuO flowers in the marked area in image (d).

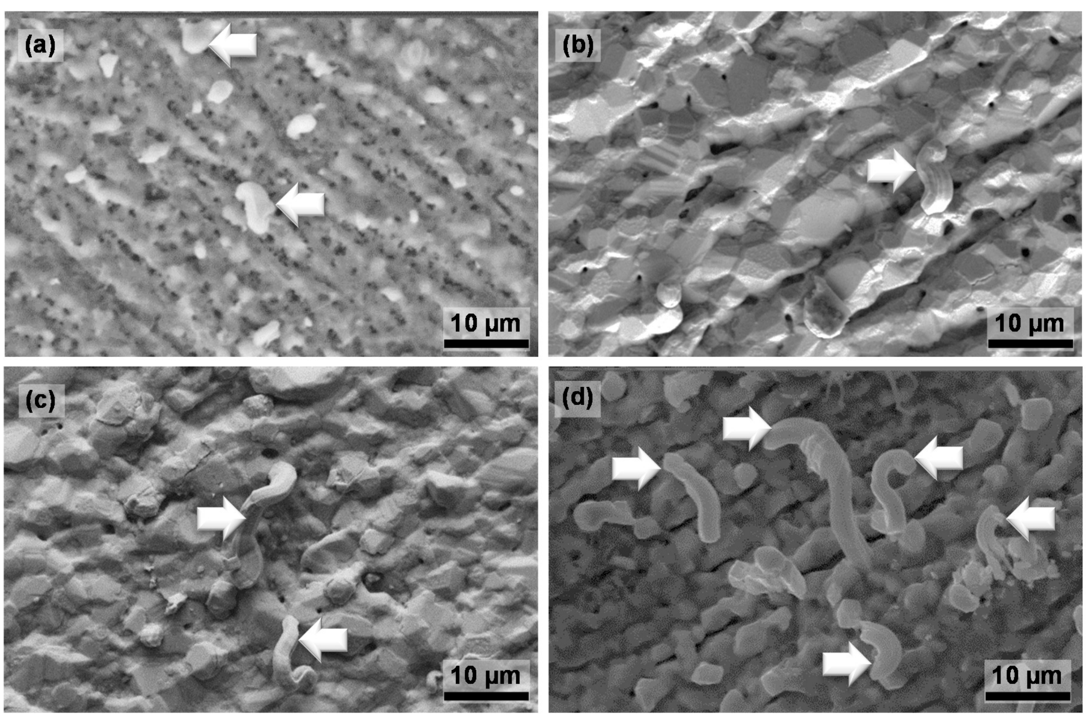

Fig. 2 FESEM micrographs of tin whiskers (solid arrow) and CuO flowers (hollow arrow) observed on the immersion tin surface of 1.2 µm coating thickness after (a) 1 week, (b) 4 weeks, (c) 8 weeks, and (d) 12 weeks under the environment condition of 55°C/85%.RH.

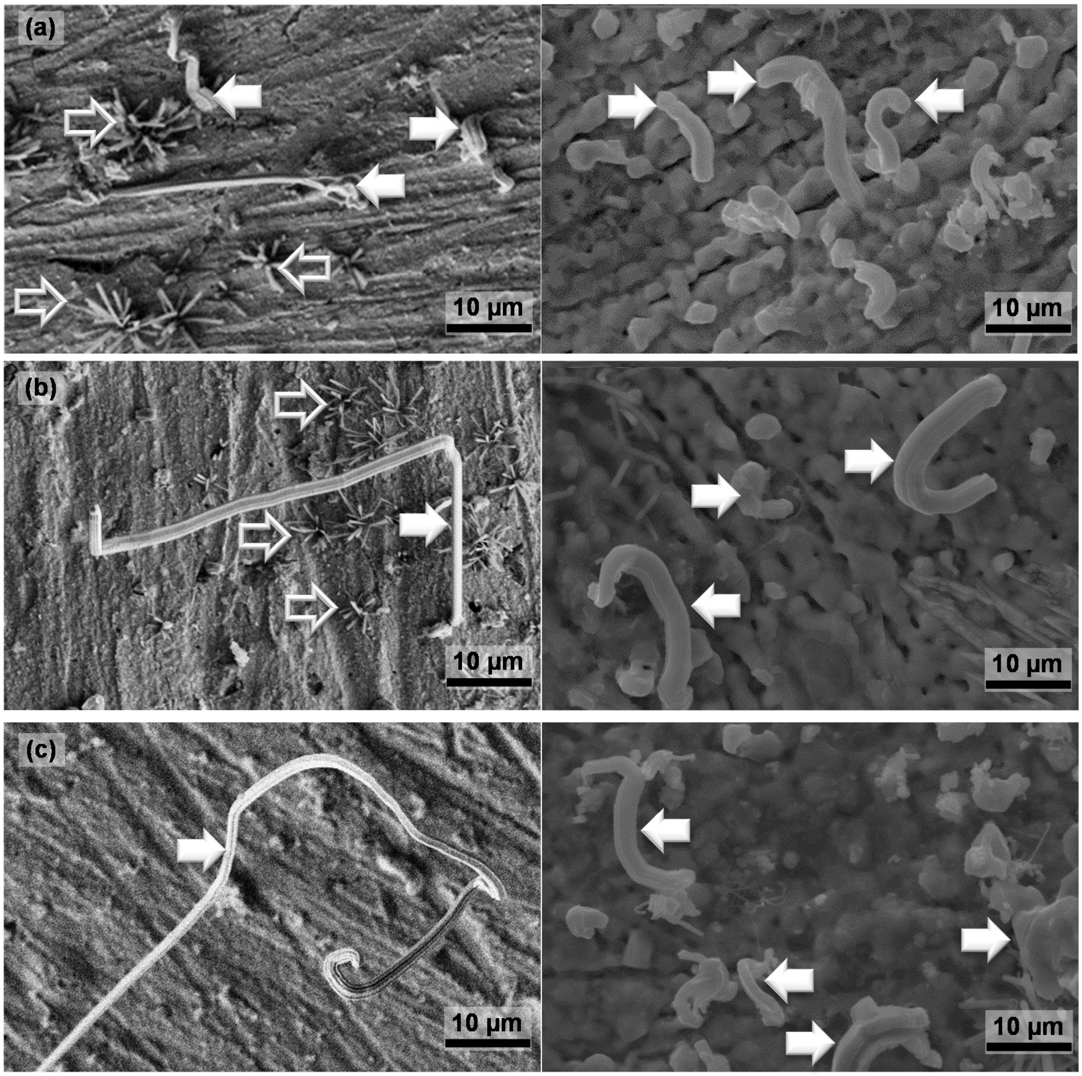

Fig. 3 FESEM micrographs of tin whiskers (solid arrow) and CuO flowers (hollow arrow) observed on the immersion tin surface of 1.2 µm (a), 1.5 µm (b), and 2.3 µm (c) of coating thickness after 12 weeks of exposure under the environment condition of 30°C/60%.RH (left) and 55°C/85%.RH (right).

The quantitative analysis was conducted in order to demonstrate the growth behaviour of tin whiskers on different tin coating thickness and exposure conditions. Fig. 4 (a-c) shows the graph of whiskers length over exposure times after 12 weeks exposed under normal and severe conditions for 1.2, 1.5, and 2.3 µm tin coating thickness respectively. Based on the Fig. 4 (a-c), the tin whiskers length is directly proportional to the exposure times for both normal and severe exposure conditions. From the graph, the whiskers length in all thicknesses exposed under normal condition is relatively longer as compared to the tin whiskers length exposed under severe condition. According to the Fig. 4a, in 1.2µm tin coating thickness, the longest whiskers is 48.96 µm after 12 weeks exposed under normal condition while only 13.32 µm of whiskers length was recorded after 12 weeks exposed under severe condition. In Fig. 4b, the longest whiskers for 1.5 µm tin coating thickness is 54.24 µm for normal condition, while

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severe condition showed only 15.94 µm of whiskers length after 12 weeks of exposure time. In Fig. 4b,3 (c), for 2.3 µm tin coating thickness, the longest whiskers is 58.58 µm after 12 weeks exposed under normal condition as compared to 17.5 µm of tin whiskers length after 12 weeks exposed under severe condition.

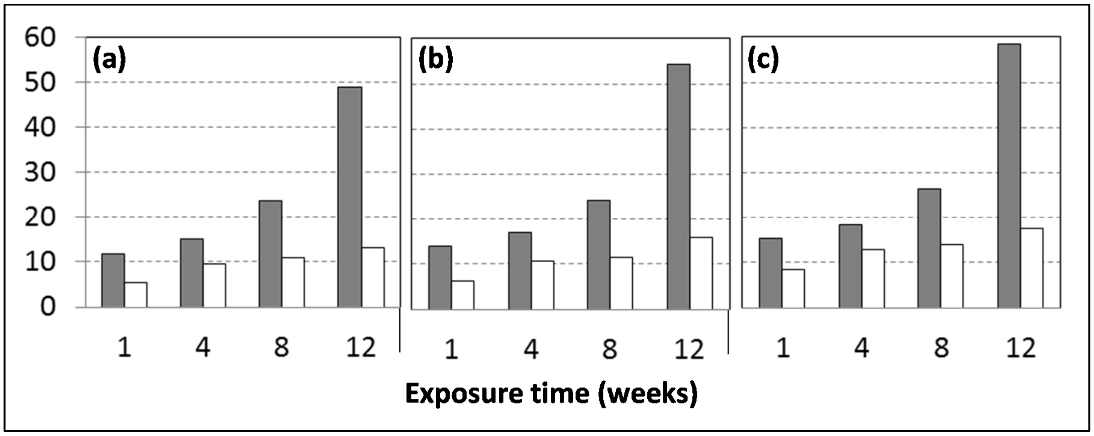

Fig. 4 Graph of whiskers lengths over exposure times after 12 weeks exposed under 30°C/ 60%RH and 55°C/ 85% RH for (a) 1.2 µm, (b) 1.5 µm, and (c) 2.3 µm tin coating thickness.

In general, thicker tin coating thickness produced longer tin whiskers length. This is due to the thickness of tin coating has influence the whiskers formation and growth. It can be explained by the excessive amount of Sn atoms for thicker tin coating has caused greater Sn supplies for longer whiskers growth as compared to thinner tin coating that has less Sn atoms supply. However, the tin whiskers growth for normal condition was more aggressive as compare to severe condition. This fact is contrast to the research reported in the literature where high temperature and humidity condition has contributed to the higher tin whiskers growth electroplated tin surface finish [13]. This can be explained by the fact that the aggressive tin whiskers growth on the electroplating surface finish was influenced by the residual stresses inherited from the electroplating process. Immersion tin however is a stress free plating process in which, at high temperature and humidity condition, creep behaviour is dominated.

The slow whiskers growth under severe condition is related to the creep process occurred in tin surface and formation of copper oxide, CuO. Creep process in tin plating cause relaxation of external stress under exposure temperature thus cause discontinues growth of whiskers in indented surface. Since the melting point, T_m of pure tin is 263°C (536 K) and the creep temperature is $0.4T_m$ (214.4 K), therefore at exposure temperature of 55°C (358 K), creep has dominated because it is above creep temperature. This can be simplified as the stresses generated IMC growth were gradually releases during the creep process. This result is consistent to the studied done by Liang et al. [22]. Unlike electroplated surface finish, the tin whiskers growth has influenced by residual stresses.

Even the whiskers growth behaviour for samples exposed under normal condition and severe condition are similar, but the types of whiskers are different between the two conditions. In general, more thin and long whisker can be observed on the samples exposed under normal condition (Fig.1) while severe condition showed thick and short whiskers (Fig.2). The twist-, nodule-, spiral-, straight-, and bent-type tin whiskers can be observed on the samples exposed under normal condition (Fig.1). The high magnification image of various tin whiskers types formed on the samples exposed under normal condition are shown in Fig. 4. Most of the tin whiskers formed in the high expect ratio such as straight- (Fig. 5b), twist- (Fig. 4d), and bent-type whiskers and they have grown much thinner and longer than spiral (Fig. 5a) and nodule-type (Fig.5c) whiskers. However, nodule- and spiral-type whiskers are thicker and have striations along the length of their column while other types have smooth surface. Meanwhile, the whiskers formed in the surface exposed under severe conditions showed only spiral- and nodule-types of tin whiskers without striations along the length of their column (Fig.2).

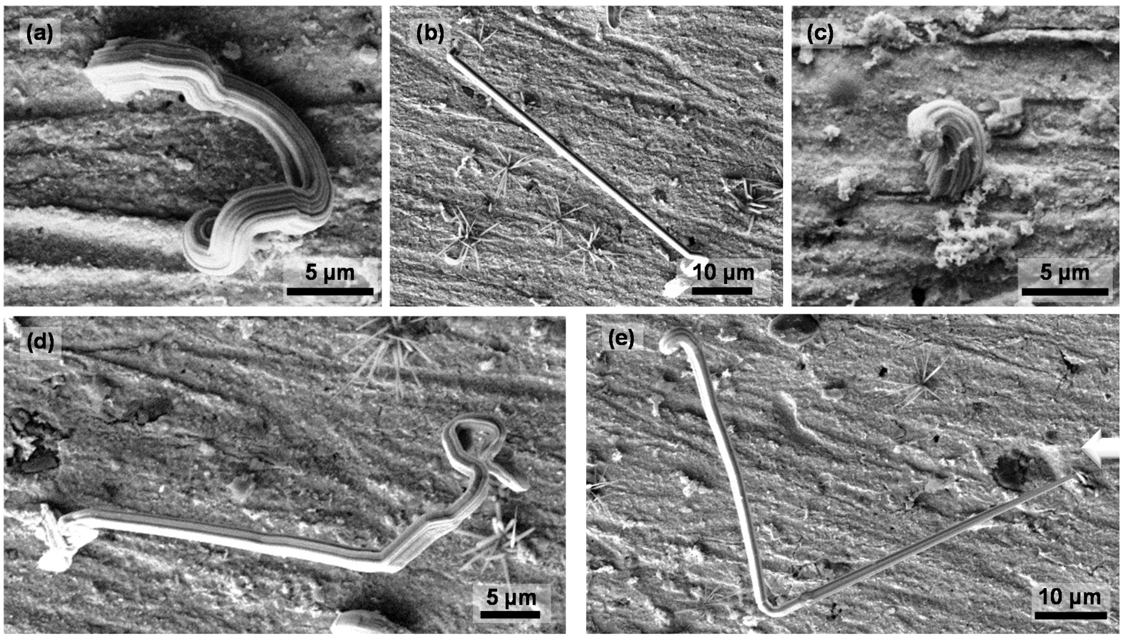

Fig.5 FESEM micrograph of various types of tin whiskers formed on the immersion tin surface finish exposed under 30°C/60%.RH (normal condition): (a) spiral, (b) straight, (c) nodule, (d) twist, and (e) bent.

4.0 CONCLUSSION

The study on tin whiskers behaviour was successfully conducted on the immersion tin surface finish coated at various coating thickness and tested under two different conditions whish are under 30°C/60%.RH (normal condition) and under 55°C/85%.RH (severe condition). As for the effect of coating thickness, thicker coating produced longer tin whiskers length for both environment conditions due to the greater Sn atoms supply provided by thicker tin surface finish as compare to thin coating thickness. Severe condition however produced shorter tin whiskers length as compare to the normal environment condition due to the domination of creep behaviour at high temperature has resulting in the relaxation of the stresses that supposed to force the IMCs growth.

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