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Effect of Environmental Conditions on Tin (Sn) Whisker Growth

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Abstract

This research focuses on tin whisker growth under two different environmental conditions namely hygrothermal and a salt solution. Tin coated brass coupons were used for this study to analyze the growth of tin whiskers over time. Their growth rates were examined periodically using optical and scanning electron microscopy. The physical characteristics of the tin whiskers were identified for each environmental condition. It was discovered that submersion of tin coated brass substrates in 5% salt solution considerably increased the density (number of whiskers per unit area), and the length of the whiskers. In addition, it was found that the geometry and aspect ratio of tin whiskers were different for each environment.

Keywords

Tin Whisker, Environmental Conditions, Tin Coated Brass Coupons, Whisker Growth, Corrosion

1. Introduction

Tin whisker growth is a challenging problem facing the Aerospace, Defense and High Performance Electronics industry. Tin whiskers are microscale, needle-like filaments made of purely tin that grow from tin plated surfaces. Their shapes can vary from “odd-shaped eruptions” to “nodules”, as well as straight, kinked, hooked, forked, or pyramidal structures [1]. They can grow spontaneously at room temperature from electroplated [2] and vapor-deposited tin films [3]. Whiskers can grow to reach a few millimeters in length and are hollow with a striated surface [2]. Three main parameters, density, length, and growth rate, influence the risks created by tin whiskers [4]. The length of a whisker is measured from the plating surface [5] and can be measured in two ways according to the Joint Electron Device Engineering Council (JEDEC) standard. The first method is to take the summation of all the straight divisions of the whiskers [6]. The second method is to take the length from the base of the

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whisker to the most distant point (not including any bend at the tip) and draw a circle. The radius of that circle is taken as the length of the whisker [4]. Growth rate portrays the end-to-end span of a whisker changing over a certain time. Tin whiskers are reported to be single crystalline [7] [8] pure tin protrusions, a few micron diameters [9]. Tin whiskers have been found to grow on electroplated matte tin on a brass-alloy substrate when exposed to a condensation induced corrosive environment [10].

The growth mechanism of tin whiskers is not well understood; they can grow spontaneously at room temperature or take many years. Over the last fifty years, many studies have been undertaken to understand the mechanism(s) of the whisker growth. There exist many reconciled as well as contradictory explanations. Several driving forces seem to influence whisker formation. The primary ones are oxidation [6], recrystallization [11] and stress [12] [13]. Some factors behind these driving forces have been considered. These are based on internal residual stresses, externally applied stresses [14], stored energy, surface energy effects [15], formation of intermetallics [8], thermal expansion mismatch**,** corrosion, and electromigration [16]. There are also other processing variables that can affect whisker growth such as film thickness and plating conditions [3], grain size [7], microstructure and composition [17].

These driving forces led researchers assent to stress being the main force behind whisker growth, particularly the relief of stress [14]. Tensile stress is thought to slow the growth, whereas compressive stress to expedite whisker formation [7], although tin whisker protrusion on tensile regions of a tin finish was observed [9]. It is rationalized that bent tin coated substrates tend to form more whiskers at a faster rate, on the inner-curved area rather than on the flat area [18]. The rate of whisker buildup was found to be proportional to the applied stress. Moreover, the higher the stress evolution is, the longer are the whiskers [14]. However, stress itself is not sufficient to provide the impetus behind whisker growth.

Some researchers rationalize that oxidation is the impetus behind whiskering. It has been claimed that an oxide layer formed on the surface is required for whisker growth [8]. It was also proposed that whiskers can be prevented by the sole removal of oxygen from the tin film [15]. The formation of a tin oxide layer within the grain boundaries also increases internal compressive stress to the tin grain boundaries. However, when a weak spot or a fissure in this tin-oxide film exists, the compressed tin in the tin grains tries to escape through it. However, the necessity of oxygen in tin whisker growth was countered by a study [17] that eliminated any native oxygen or tin oxide formed and still found whiskers growing from the surface. The author explained that although the surface was removed from the oxygen, the grain boundaries might still have contributed to whisker growth through oxidation occurring due to previously seeped oxygen.

Crystallographic dislocation was the prime focus of whiskering back in 1950's [17]. This idea proposes that tin atoms migrate along crystallographic dislocations to where the whisker is eventually formed. Screw dislocation was proposed by one study with tin making atomic migration into the center of the whisker through the core and the atoms eventually depositing at the whisker's base. Other studies indicated that atoms keep moving due to the screw dislocations until they reach the whisker base. Also, edge dislocations are caused due to slip or glide of the grain boundaries that cause atoms to migrate to the nucleation point from the bulk tin surface. According to Lee and Lee [8], whisker growth is a form of stress relief, where an individual grain has a different planar orientation (e.g., if the grain is (420), the preferred direction is (220)) from that of the surrounding grains. Due to this different planar orientation, an increase in the out-of-plane strain occurs forcing the oxide layer to form a crack, which aids in the excess internal release through the bulk tin by forming whiskers. Lee and Lee also noted that Bardeen-Herring dislocation transports the tin atoms to the crack until the stress is relieved.

It has recently reported [19] that the mysterious driving force behind tin whisker is the strain gradient. It was found through digital image correlation (DIC) that the strain or stress gradient is the main responsible factor for the development of whiskers or hillock formation instead of the compressive stress field. At first, the stress or strain concentration occurs within the grain boundaries through several causes (oxidation, intermetallic compound (IMC) formation, CTE mismatch, etc.). Strain energy can be calculated at any grain boundary, where (h_1, k_1, l_1), (h_2, k_2, l_2) and (h_3, k_3, l_3) may represent the grains [20]. The grains, which have the higher strain gradient, cause damage to the neighboring weaker grains as strain energy reaches a critical value. At these damaged grain sites, re-nucleation occurs, which ultimately grow new whisker grains. At this time, tin atoms set off on their journey to the base of the whisker grain along the surface and continue the whisker elongation, since newly formed tin grains have higher surface diffusivity.

It is also believed that whiskering is an extrusion phenomenon, which might be caused by an IMC layer and

grain boundary cracks. The rate of formation of IMC changes with different substrates under the tin film since different materials have different reaction with tin or tin alloys. IMC generation leads to molar volume difference from either the tin or the substrate material. For example, brass substrate forms a common IMC-Cu_6Sn_5 with tin at room temperature [7]. The growth of the IMC becomes even faster at elevated temperatures forming another IMC Cu_3Sn from Cu_6Sn_5 above 100˚C [6]. The IMCs are formed because of the inter-diffusion of tin and brass atoms. Brass diffuses more into tin than tin does into brass, which can be examined from the brass-tin phase diagram [21]. The densities of brass, tin and Cu_6Sn_5 are 8.96, 7.28 and 8.27 g/cm^3, respectively, indicating that the density of the IMC falls between that of brass and tin. These molar volume differences create compressive stress within the tin grains, which is tangential to the surface. Moreover, the IMC plays a crucial role in creating the compressive stress at the interface of the tin coating and the substrate. The theory of contribution from IMC stress has some deficiencies as well. Most of the work was conducted on tin plated brass, which formed IMC-Cu_6Sn_5 with tin. Moreover, many studies on whiskers did not find any IMC formation for certain material substrate such as the tin coated aluminum [17]. Another theory proposed by Galyon and Palmer [22], named as "Integrated Theory on Whisker Formation and Growth" described that a vacant zone is created within the brass substrate, named the Kirkendall zone, close to the tin film and brass substrate interface due to the dominant diffusion of brass into tin [22]. This Kirkendall zone forms shrinkage because of the vacancy, and the shrinkage effects to a state of tensile stress in that zone. That tensile stress acts on the tin film as compressive stress via the IMC. The IMC forms nearly to surface, while the tin is transported to the lower stress regions. During the penetration of the film, any remaining bulk tin or surface tin oxide between the IMC and free space is pushed aside by the penetrating whisker.

In the current research tin coated brass coupons were used to study to formation of tin whiskers over time. Two different environmental conditions, namely hygrothermal and submersion in salt solution, were used in an attempt to accelerate tin whiskers growth. The growth of tin whiskers was examined periodically using optical and scanning electron microscopy. The physical characteristics of tin whiskers, density and length were identified for each environmental condition.

2. Materials and Experimental

All the coupons (tin coated brass) were provided by Boeing Corp. These coupons were 102 mm long, 25.4 mm wide and 0.76 mm thick. Two sets of samples, including one scratched and one unscratched, were exposed to two different environmental conditions: hygrothermal and 5 wt% NaCl solution environments. The coupons were hung by nylon string tied to each coupon using metal hooks.

The hygrothermal or thermal cycling experiment was performed in a Thermotron chamber (Thermotron, Holland, Michigan, USA). All the coupons kept in this chamber were hung on a metal hook by a nylon string (**Figure 1**). The dwell time used was based on the JESD22-A104 B standard [23], which states that a 5 to 10 minute dwell time is appropriate for tin whiskers. Dwell time is the duration the sample is held at the minimum and maximum temperature and relative humidity. The ramp rate was 2˚C/min. The chamber cycled between 15˚ to 85˚C, giving a rise and fall of 70˚C for 35 minutes each. The temperature and relative humidity profile is shown in **Figure 2**. The relative humidity was 85 wt% ± 15 wt%, while the temperature varied ±0.1˚C.

In the 5 wt% NaCl solution submersion experiment, the coupons were divided into scratched and unscratched groups. Both groups had one uncoated control coupon and four coupons coated with four different coatings. The five scratched and five unscratched coupons were hung by a nylon string and submerged in separate tanks containing the aqueous 5 wt% NaCl solution (**Figure 3**). It should be mentioned here that this article is examining tin whisker growth on the uncoated samples only. Examination of the effectiveness of various conformal coatings on whisker mitigation is in progress and will be reported later.

Samples from each experiment were taken out after several weeks for light optical microscopy (LOM) and scanning electron microscopy (SEM) examination. During the initial phase, after about 5700 hrs (~238 days), the control samples from both hygrothermal and salt solution environments were chosen to determine the whisker growth. Once the coupons were analyzed, they were taken back to their environments, respectively, and the exposure/immersion continued. In the second phase, after about 7000 hrs (~292 days), tin coated brass coupons were taken out from their respective environments and microscopic analysis was performed. The third phase SEM microscopy study was performed after 12,500 (~500 days) hrs.

Figure 1. Coupon arrangement in the thermotron environmental test chamber for hygrothermal exposure.

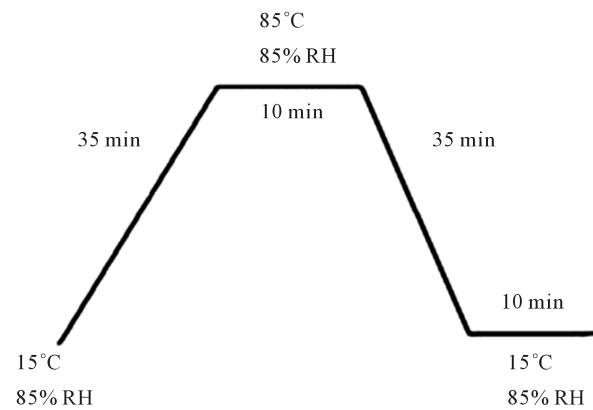


Figure 2. Temperature and relative humidity profile of the hygrothermal cycle that the coupons were exposed to in the thermotron chamber.

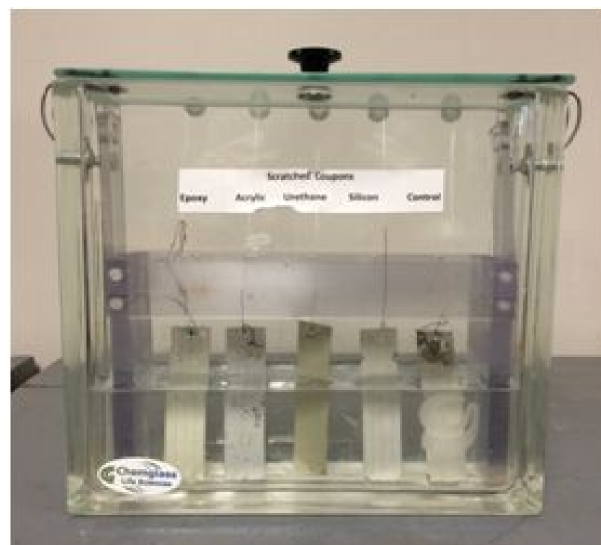


Figure 3. Scratched tin plated uncoated (control) and coated brass coupons submerged in aqueous 5 wt% NaCl solution.

3. Results and Discussion

3.1. Optical Microscope Observations

Figure 4 shows the optical micrographs of an unscratched control sample. The micrographs reveal surfaces of the coupon that are not exposed to either corrosive or hygrothermal environments. Reflective tin grains on the surface are seen in brighter areas of the micrographs. The darker spots on the surface might be the grain boundaries. The surface shows an overall uniform appearance in all the regions.

Optical observations on the scratched flat control sample from the hygrothermal environment after 5700 hours of exposure revealed deformations in the inspected regions due to the scratches and small crater formation (**Figure 5**). The surface in the figure also revealed brighter (marked in black circles) and darker regions (marked by red circles). The brighter regions could be the upward protruding of a reflective tin surface, which could be the indication of hillock formation. The darker region could be an indication of grooves forming around hillocks.

Optical micrographs of the scratched coupon from the salt solution after 5700 hours exposure were taken at different locations on the coupon. Some irregularities in the pattern from the surroundings raised suspicion. The overall surface showed non-uniform and contrasting colors. A few locations (**Figure 6**) appeared to be jagged on the right side at a very shallow level where compressive forces may have led to craters along the regions inspected. Some brighter spots (marked in black circles) in the middle of the micrographs were considered possible tin whisker locations. Large sized craters (marked in red circles) are also seen in the micrographs, indicating possible locations for greater compressive stresses.

3.2. Scanning Electronic Microscope (SEM) Observations

After careful observation of tin coated brass coupons through an optical microscope, the regions determined to be suspicious from the optical microscopy examination were analyzed by SEM at definite intervals of 5700, 7000 and 12,500 hours, respectively. Additionally, energy dispersive X-ray spectroscopy (EDS) was used to determine the composition of the protrusions to substantiate whether the protrusion was a tin whisker.

Figure 7 and **Figure 8** show the SEM micrographs of the tin coated brass coupon specimen after 12,500 hrs (500 days) of exposure in 5% NaCl corrosive environment. Tin whiskers of different sizes and shapes were found, as seen in the SEM micrographs. These whiskers are mainly populated at the center of the sample where one would expect a greater chance for residual and compressive stresses. These areas are key locations for the tin whisker hillock formation and growth. The sizes ranged from 30 to 50 µm in length and 15 - 20 µm in width (**Figure 7**) for one set of tin whiskers and from 100 - 250 µm in length and 5 - 10 µm in width (**Figure 8**) for another group of whiskers at a different location. In **Figure 7** and **Figure 8**, away from the tin whiskers, due to the corrosion from the salt solution, severe pitting is visible. This pitting may become severe over time and might be detrimental to the whisker growth and formation. Also, diamond shaped dark regions are clearly visible next to the tin whiskers, representative of salt crystals in the corrosive environment.

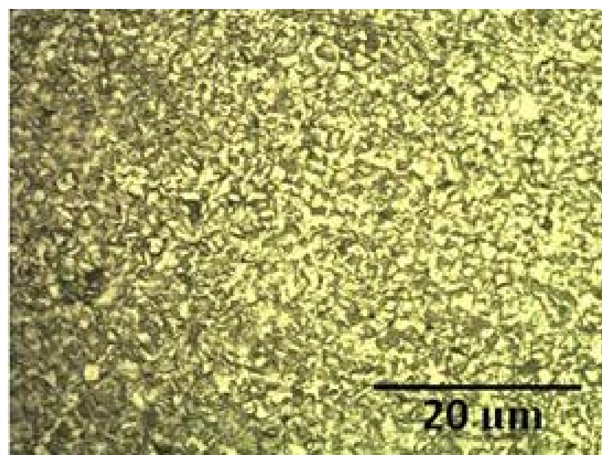


Figure 4. Optical micrograph of unscratched (control) tin coated brass coupon (50×).

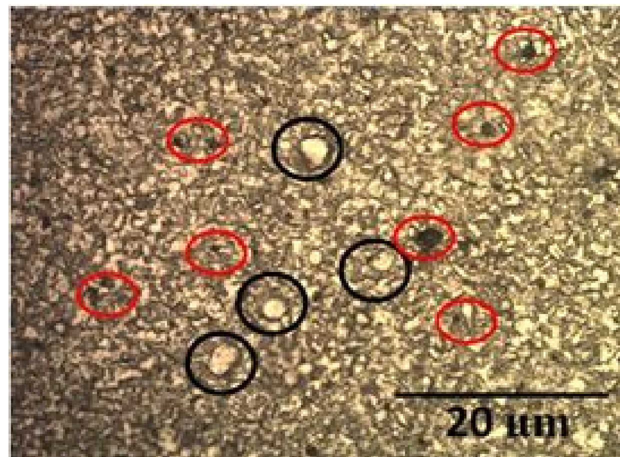


Figure 5. Optical micrograph of tin coated brass coupon in a hygrothermal environment (50×) after 5700 hours exposure.

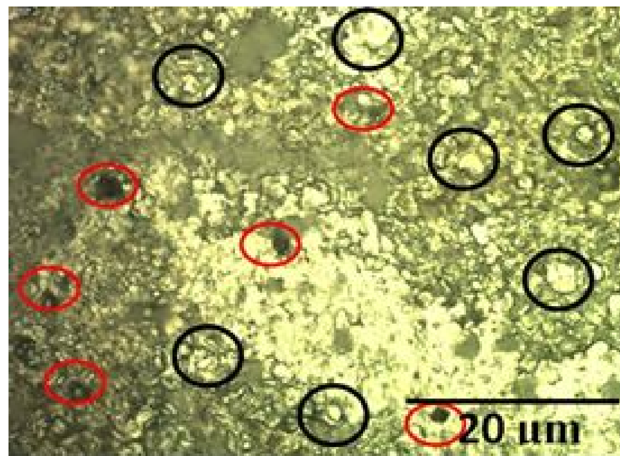
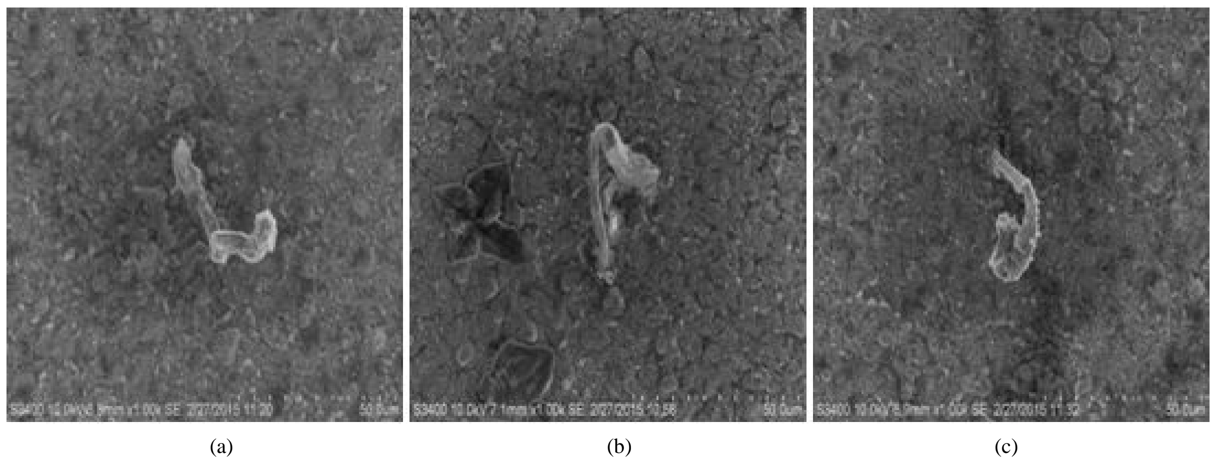
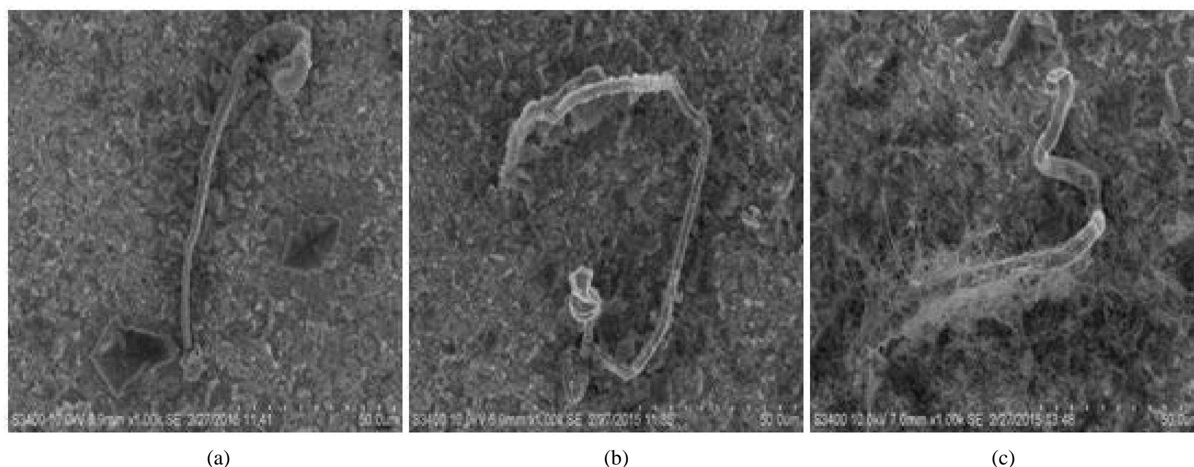


Figure 6. Optical micrograph of scratched tin coated brass coupon exposed to a corrosive (aqueous NaCl) environment for 5700 hours (50×).



(a) (b) (c)

Figure 7. SEM micrographs of tin coated brass coupon in corrosive environment (5% NaCl solution) showing fully grown whiskers (30 - 50 μm) after 12,500 hrs (~500 days).



(a) (b) (c)

Figure 8. SEM micrographs showing extra long grown (100 - 250 μm) tin whiskers from the tin coated brass coupon from the corrosive environment (5% NaCl solution) for 12,500 hrs of immersion time.

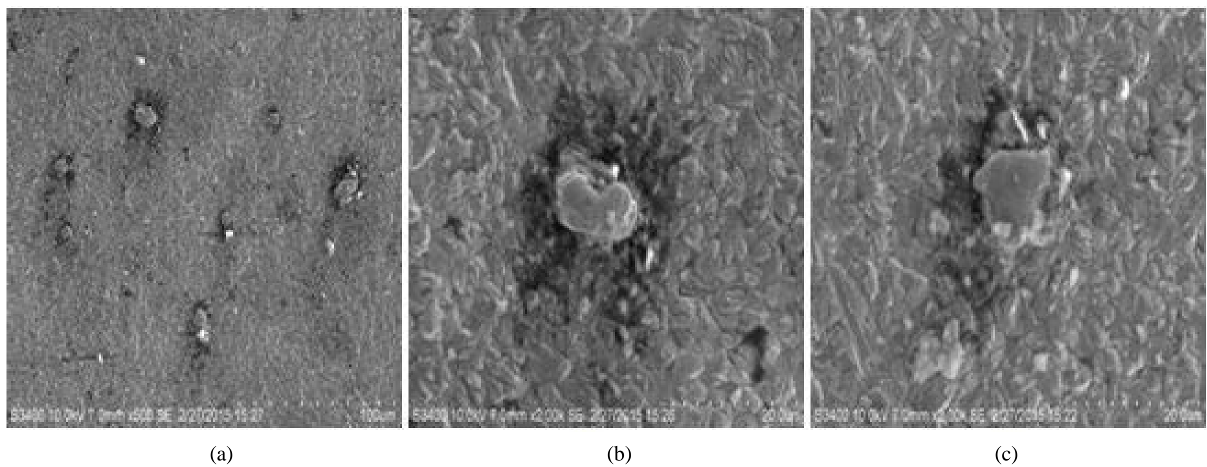
Similar SEM characterization was performed on the tin coated brass coupon from the hygrothermal environment after 12,500 hrs of exposure. There were not many fully grown whiskers found on the hygrothermal sample in comparison with the sample from the corrosive environment. The whiskers found on the hygrothermal coupon are not as long, growing only up to 20 μm in length. Locations with hillocks and the grooves around them were found on the hygrothermal coupon (**Figure 9**). Hence it appears that the time for growth of tin whiskers is relatively slower in a hygrothermal than in a corrosive environment.

SEM analysis was performed on the scratched tin coated brass control coupons from both the hygrothermal and corrosive environments after 7000 hours (~292 days). The tin whiskers were seen starting to protrude out form the hillocks of the tin coated brass coupon from the hygrothermal environment (**Figure 10(a)** and **Figure 10(b)**). Conjoined whiskers having the same root are seen in **Figure 10(a)**. Two protruding whiskers and one nascent whisker are seen in this figure. The whisker on the left (marked by an arrow) is twisted. Strong striations along the surface are also conspicuous on these whiskers. The protruding whiskers are 10 μm long. The whisker shown in **Figure 10(b)** appears to bend in a shape almost like a shaft; this bend starts nearly at the root. The length is about 8 μm long. EDS analysis was performed on these whiskers to confirm the tin composition.

The tin whiskers observed after 7000 hours of exposure in the hygrothermal environment looked similar in length to those observed in the corrosive environment (5% NaCl solution) at 5000 hours. Again, this can be attributed to the slower whisker growth in the hygrothermal environment when compared to the corrosive environment. There are several reasons for the faster growth of tin whiskers in the corrosive environment. Corrosive environments provide greater amounts of stresses from the salt media and also the periodic degradation of the tin coatings on the tin coated brass samples. This repetitive stress plus appropriate temperature and humid conditions accelerate the tin whisker formation.

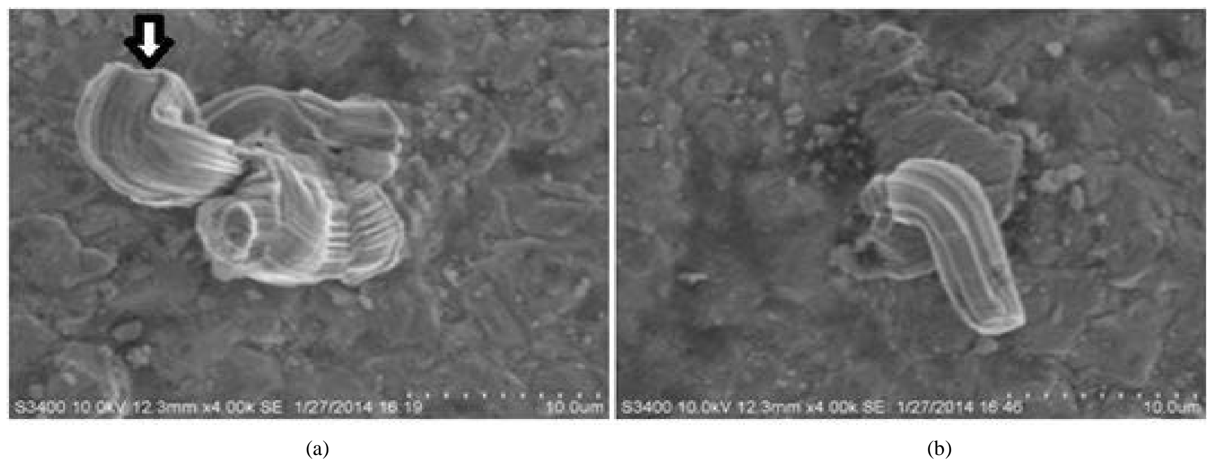
On the other hand, SEM analysis was performed on the scratched tin coated brass control coupon from the corrosive environment (5% NaCl) after 7000 hours. SEM micrographs showed that some whiskers had longer lengths of about 20 and 25 μm (**Figure 11(a)** and **Figure 11(b)**, respectively), with diameters of 7 and 5 μm, respectively. After passing a certain length of growth, the whisker in **Figure 11(a)** curves towards the surface of the coupon. Around the base or the root, areas are clearly visible with salt crystals around them. The whisker shown in **Figure 11(b)** formed a hook shape at the top by bending towards its own body. The whisker is almost laying flat on the coupon surface. The whiskers found on the scratched coupon from the salt solution have clear striations along the whisker length, which can be an intriguing feature. Suganuma *et al.* [16] observed striations on the whiskers grown in thermal cycling. The striations start from the base of the whisker and run to the tip.EDS analysis confirmed the presence of tin from the protrusions.

The hygrothermal scratched flat control tin coated brass coupon was analyzed under SEM after 5700 hours of exposure (**Figure 12**). **Figure 12** shows the initiation or the starting points of whisker growth as a root and hillocks, represented in red circles. **Figure 12(a)** shows the hillock formation in a deep groove as in a scratch location (magnification 1000×). This root or hillock is about 20 μm in size and has a tail-like elongation below the



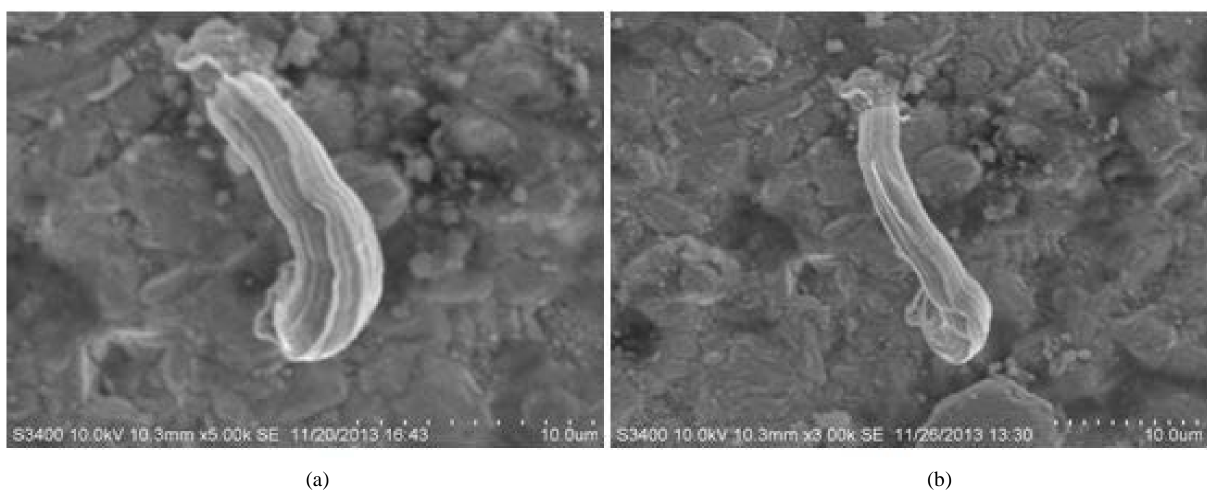
(a) (b) (c)

Figure 9. SEM micrographs of the tin coated brass coupon from hygrothermal environments showing hillock regions concentrated at a single location (**Figure 9(a)**) and zoomed in images of the hillocks (**Figure 9(b)** and **Figure 9(c)**).



(a) (b)

Figure 10. SEM micrograph showing conjoined whiskers from scratched flat coupon after 7000 hours of exposure to hygrothermal environment; magnification is 4k×.



(a) (b)

Figure 11. SEM micrographs showing long and bent whiskers from scratched flat coupon after 7000 hour exposure to corrosive environment (5% NaCl aqueous solution) at different locations; magnifications are 5k× (a) and 3k× (b).

root, about 30 μm sizes. Suganuma *et al.* [16] have observed that thermal cycling whiskers grow thick and winding and deep grooves tend to form around the region of the whisker growth as compared to room-temperature whiskers, which grow straight, though sometimes thin. These locations were hit by EDS to analyze the elemental composition at that location. **Figure 12(b)** shows the SEM micrograph and its EDS, respectively, at a different location. A tin peak was observed on the target location represented by spectrum 3 in **Figure 12(a)**, which conforms the whisker formation at the regions with hillocks.

A scratched flat tin coated brass coupon submerged in 5% NaCl solution (corrosive environment) after 5700 hours was taken for SEM analysis on the suspected locations. **Figure 13(a)** shows the tin whiskers starting to grow from the hillocks or the roots. Their EDS spectrum confirms the tin peaks at respective locations (**Figure 13(b)**). Unlike the hillocks observed on the coupons from the hygrothermal environments, the whisker locations in 5% NaCl solution showed hillocks formed with deep grooves around them. From the SEM micrograph of **Figure 13(a)**, it can be noticed that the whiskers are formed from the edge of the hillock and are bent along the direction of the hillock edge. It is also seen that the surrounding location around the hillock region is pitted and corroded; salt crystals can also be seen on the surface due to long term exposure to salt solution. **Figure 13(b)** gives the EDS results on these whiskers. It is clearly evident that the whisker protrusion is from the tin coating on the brass coupon. The EDS confirms 100% tin presence on the whisker.

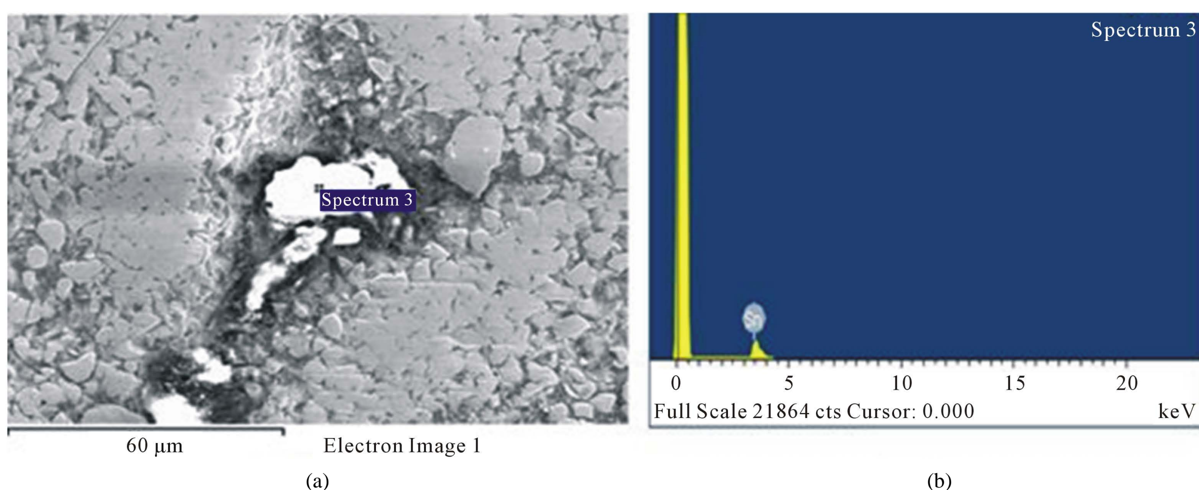


Figure 12. SEM micrograph of control flat tin coated brass coupon after 5700 hours in hygrothermal environment showing the (a) whisker hillock location and (b) EDS spectrum at that location.

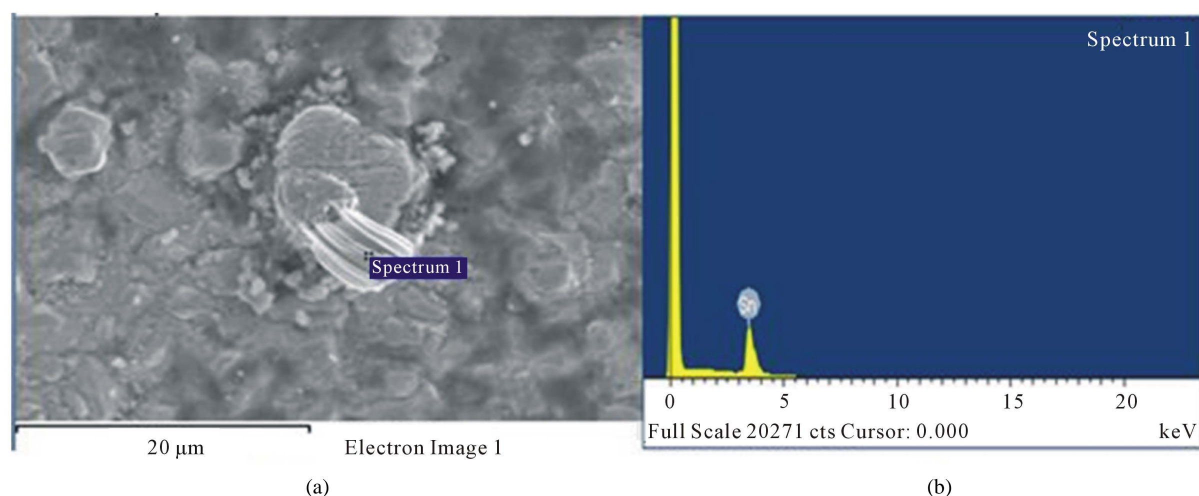


Figure 13. SEM micrograph of scratched flat tin coated brass coupon after 5700 hour exposure to corrosive environment (5% NaCl aqueous solution), magnification 1k× (a) and EDS results at that location (b).

4. Conclusion

The effect of environmental conditions namely: hygrothermal cycling and immersion in NaCl (5 wt%) aqueous solution on tin whisker growth was investigated using tin coated brass substrates. Optical and scanning electron microscopy was used to examine the initiation and propagation of these whiskers as a function of time. The scratched flat coupon from the hygrothermal environment had numerous hillocks made of pure tin after 5700 hours of exposure. The hillocks had deep grooves around them. The scratched flat coupon from the corrosive environment after 5700 hours showed fully grown whiskers up to 20 µm. These whiskers had striations along the lengths. After 12,500 hours of exposure, the coupons taken from the corrosive environment exhibited very long whisker up to 250 µm. These whiskers were thinner and have more bends than those grown on coupons exposed to hygrothermal cycling. Our finding indicated that whiskers were growing faster in the corrosive environment than in the hygrothermal environment. This is believed to be due to the formation of non-uniformed surface corrosion which results in differential stress states that enhances and accelerates tin whisker growth.

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