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An assessment of Sn whiskers and depleted area formation in thin Sn films using quantitative image analysis

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Abstract An experimental technique was developed to determine the extent of Sn whisker growth and depleted area formation on evaporated 1 μm tin (Sn) films. Deformation of the Si substrate placed a controlled magnitude of compressive or tensile stress across the films. Quantitative image analysis was used to monitor whisker growth and size of the depleted areas. The test conditions were: stresses 10–40 MPa; temperature, 180 °C; and time durations, 1–8 weeks. The whisker length increased with compressive stress. The whiskers appeared within the first week, but then did not grow significantly with additional time. Some whiskers were located in the centers of depleted areas. The depleted areas size was not sensitive to the applied stress, but did increase with annealing time. Both Sn whiskers and depleted areas were the result of potentially similar rapid, long-range diffusion processes. However, differing trends suggested that separate driving forces and/or rate kinetics controlled the two phenomena.

Introduction

The topic of tin (Sn) whiskers has become an important issue in electronic packaging due to their potential to create short-circuits between neighboring conductors. Also, Sn whiskers can become debris that contaminates microelectronic devices, especially, mirco-electrical mechanical systems (MEMS) [1]. Early-on, the risks caused by Sn whiskers were mitigated by replacing pure Sn coating with electroplating tin–lead (Sn–Pb) finishes having 3–10% Pb by weight [2, 3]. However, the electronics industry has returned to a preference for the pure Sn finish in order to remove Pb from electronics [4–10]. A consequence has also been the reappearance of the risks associated with Sn whiskers. Detailed reviews of Sn whiskers have been compiled by Galyon and Osenbach et al. [11, 12].

Numerous mechanisms have been proposed for Sn whisker growth. Those mechanisms include dislocation loops [13, 14], helical dislocation motion [15], recrystallization [16–21], and grain boundary diffusion [22, 23]. A grain boundary fluid flow mechanism was more recently proposed by Li in [23]. However, none of these mechanisms has been confirmed, experimentally.

Compressive stresses are generally regarded as the driving force for Sn whisker growth. The compressive stress can be generated by intrinsic stresses within the Sn layer, in particular, those films deposited by an electroplating process. There are also extrinsic sources of compressive stresses such as the bending deformation of the underlying substrate as well as by the formation of copper/tin (Cu/Sn) intermetallic compounds (IMC) at the interface between the Sn layer and the Cu-based substrate [14]. In many studies, several or all of these stress contributors are active, which makes it difficult to identify the expressed effect of stress or other variables on whisker growth.

A test program was developed to examine the fundamental behavior of Sn whiskers. First, evaporated thin films of Sn were used in order to eliminate the high, and often variable, intrinsic stresses of electroplated coatings. Second, the effects of stresses caused by IMC growth were eliminated by depositing the Sn films on a coated silicon wafer. Using this test geometry, external stresses, either compressive or tensile, could be applied to the films at

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well-defined levels. The findings of the study reported upon here include both the behavior of Sn whiskers on thin Sn films as well as the second phenomenon of *depleted zones.*

Experiment

One micron thick pure Sn films were evaporated on 1 inch diameter silicon (Si) wafers after depositing an adhesion layer of 20 nm chromium (Cr). The use of silicon wafer avoided the formation of copper tin intermetallic compounds that create extrinsic compressive stresses. Biaxial stress was applied to the Si disk by the fixture shown in Fig. 1. Each wafer was supported underneath around its edge. The Sn film was subjected to a compressive stress distribution resulting from the load applied to the center of the wafer when the film was oriented upwards (Fig. 2a). Reversing the wafer orientation applied a tensile stress to the Sn film (Fig. 2b). The stress distributions were based on the calculation described in [24].

The test conditions are as followed. The entire setup was exposed to 180 °C in a vacuum of 10^{-4}Torr. The time periods were 1–8 weeks. After each 1 week interval, the sample was taken out of the furnace, unloaded, and cooled down to room temperature. Next, quantitative whisker and depleted zone data were taken at the same locations on the Sn coating using scanning electronic microscope (SEM). After completion of the observations, the sample was re-loaded and returned to the vacuum furnace for the next 1 week exposure duration. The observations were made over a total annealing time of 8 weeks. An image analysis method was developed using MATLAB to measure whisker numbers and lengths as well as the depleted zone areas from SEM photographs.

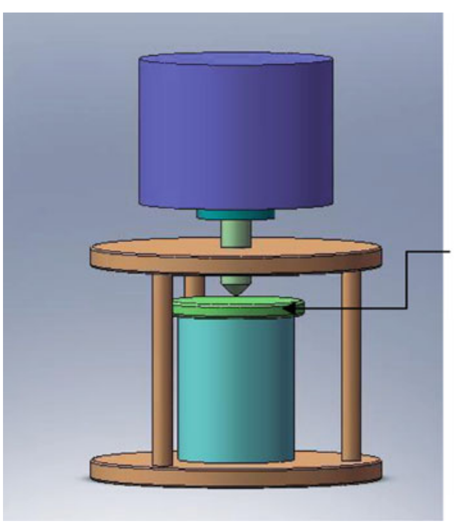

Fig. 1 The 1″ disk of n-type Si wafer, orientation [100], 350 μm thick with a Sn layer coated over the surface is bent by a dead weight (500 g) applied at the center and supported by a hollow cylinder below

Results

Qualitative analysis

Compressive stress

Shown in Fig. 3 are images that exemplify the whiskers and depleted zones at the location that is 4 mm (6.5 MPa per Fig. 2a) away from the center of the Sn film. The time duration of 8 weeks. Both Sn whiskers as well as depleted areas appear in the images. Although not clearly visible in these low magnification images, most of the depleted zones had a whisker grown inside. It is the presence of the whiskers, as well as their morphologies that are discussed below, demonstrated the depleted zones were not the result of delamination and peeling. Microstructural details about the whiskers and depleted zones can be found in [25]. Hillocks are also present in the Sn films. The hillock dimensions are 1–2 μm in diameter and 1–2 μm high. Because hillocks do not pose a strong reliability concern, they were not considered explicitly in the current analysis.

After a total of 8 weeks, the whiskers had not changed appearance from that after the first week. The whiskers did not increase or decrease over the time. Also, no new whiskers appeared following those documented after the first week.

On the other hand, the depleted areas did change over the course of the annealing period. Some of the depleted areas changed both size and shape. Also, new areas appeared while others were annihilated within the interval. Shown in Fig. 4 are high magnification images of a local field of view that illustrates the change to the depleted zone geometry. Note that the whiskers and hillocks in the image did not change length through the photograph sequence. The depleted zone changed significantly around the large whisker. Elsewhere, some depleted areas became covered with a Sn film and at other areas, Sn films developed the depleted zones. For example, to the upper left-hand location of the tall, thin whisker (i.e., at approximately the “11 o’clock” position) a small depleted area appeared through the sequence of photographs of #4, #5, and #6.

Another interesting phenomenon is observed between the photographs #1 and #3 in Fig. 4. At the upper left-hand position to the thick whisker in #1 is a small hillock. Note in picture #2 that the expanded depleted zone consumed that small hillock. However, further annealing represented by #3 image caused the depleted area to retreat and the reappearance of that small hillock. So, it appears that there is a memory to the Sn film surface morphology during the annealing sequence.

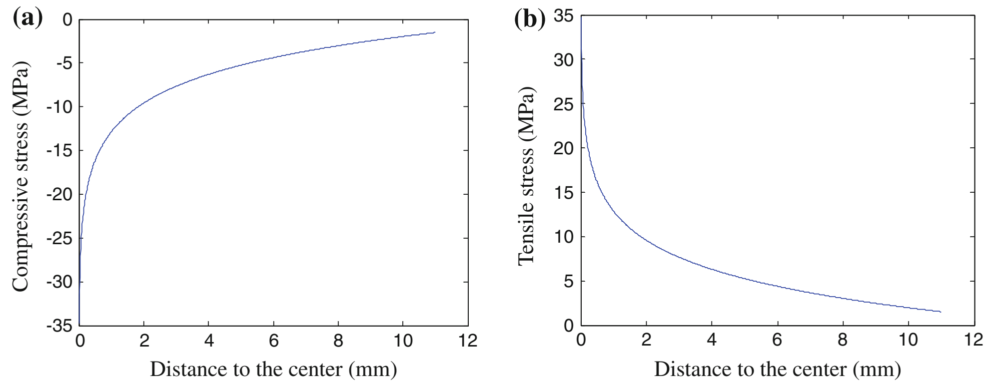

Fig. 2 **a** Compressive stress, **b** tensile stress distributed on the 1 inch diameter Sn layer when loaded by 500 g at the center

Tensile stress

Whiskers and depleted zones also appeared when the Sn film was exposed to a tensile stress. The number of both entities increased from the center of the wafer towards the edge, which coincides with the direction of decreasing tensile stress. As was similarly performed with the compressive stress, a location 4 mm from the center (6.5 MPa) was monitored to measure whisker and depleted zone growth over the eight 1 week annealing periods. The corresponding images are shown in Fig. 5. The yellow circles identify regions of change over the sequence of photographs. For example, the circle in the lower left-hand corner identifies the development of a hillock through images 3–8. The yellow circles near the center of the photographs identify a depleted zone that cycled through appearance and disappearance as a function of the annealing time (progression through the photographs).

The whisker growth was also recorded under the tensile stress. As a qualitative assessment, shown in Fig. 6 are SEM photographs of the Sn film surface at distances of 2, 4, 6, 8, 10, and 12 mm from the center. At those locations, the stress values are 18.24, 11.58, 7.69, 4.93, 2.78, and 1.03 MPa, respectively. The annealing time was 1 week. After the first week's annealing, there appeared only a few Sn whiskers under the tensile stress 18.24 through 2.87 MPa (Fig. 6(1–5)). However, in Fig. 6(6), which represents the *lowest* tensile stress, their number increased significantly. The number and length of the whiskers remained largely unchanged over the remaining annealing cycles. On the other hand, the depleted areas generally increased in both size and number with annealing time. The fact that whiskers developed under the macro applied tensile stress implies that there was an intrinsic compressive stresses superposed on the Sn film. The net effect was a compressive stress responsible for whiskers. The fact that the depleted zones did not exhibit the same sensitivity to annealing time as did the whiskers is an indication of a potentially different driving force.

Quantitative analysis

A quantitative analysis routine was used to describe the change of whiskers and depleted areas, based upon SEM images and a MATLAB program. A synopsis of that technique is provided below, using compressive stress data as the example.

Quantitative analysis methodology of whiskers

Figure 7a shows an SEM image of an area that has several whiskers. The MATLAB program first changed the original gray-tone image to a black-white image which is shown in Fig. 7b. All the image pixels in this area were stored into a 2-dimensional data array. Using the image processing module of MATLAB, the number and the length of each whisker were obtained; as were the maximum length, total length, and average length of all whiskers in the image calculated by the MATLAB program. The angle of growth was taken into account. For example, whiskers that were imaged by the SEM at an angle of 45° to the surface, had their lengths multiplied by 1.414 to obtain the actual lengths. The adjusted lengths are listed in Table 1.

At each of eleven 1 mm steps from the center to the edge of the Si wafer, four SEM images were taken with the same magnification of 250×. All 44 images were processed by MATLAB to obtain the distribution of tin whiskers over the Sn film. The whisker data were represented as a mean and standard deviation of the measurements.

Quantitative analysis methodology of depleted areas

Shown in Fig. 8a is an original gray-tone SEM image of a number of depleted areas or zones. The MATLAB program changed the gray-tone image to a black-white image that is presented in Fig. 8b. The white regions indicate

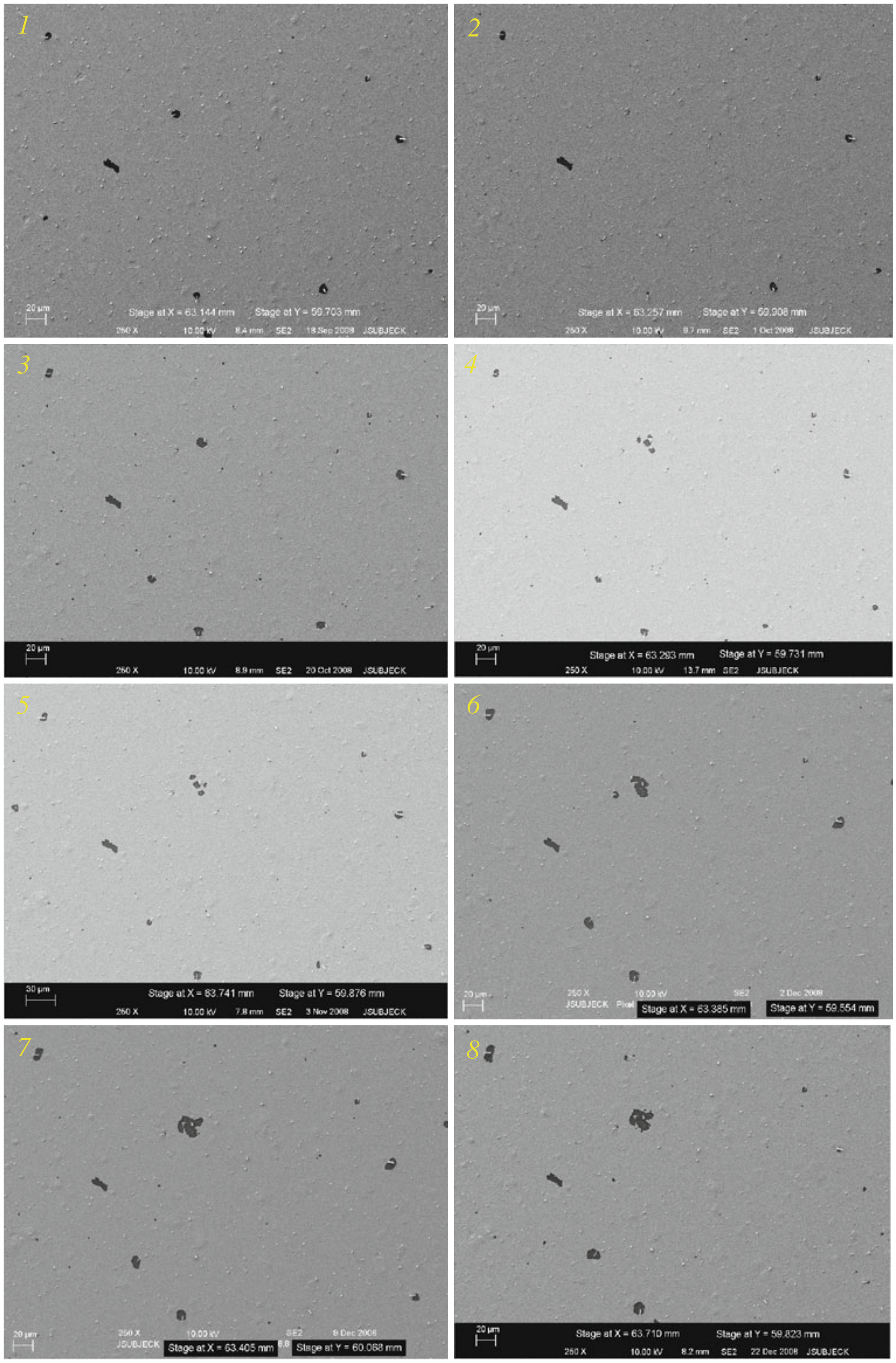

Fig. 3 SEM images that show the same area after each of the eight annealing cycles (weeks at a distance of 4 mm from the center point, stress = 6.5 MPa). The sample was exposed to 180 °C in vacuum at 10^{-4} Torr

those depleted areas. The MATLAB program calculated the number and the area of each depleted zone as well as the maximum area, total area, average area, and standard deviation. Areas smaller than 1 μm^2 were neglected. In the analysis, the depleted area results were listed in Table 2.

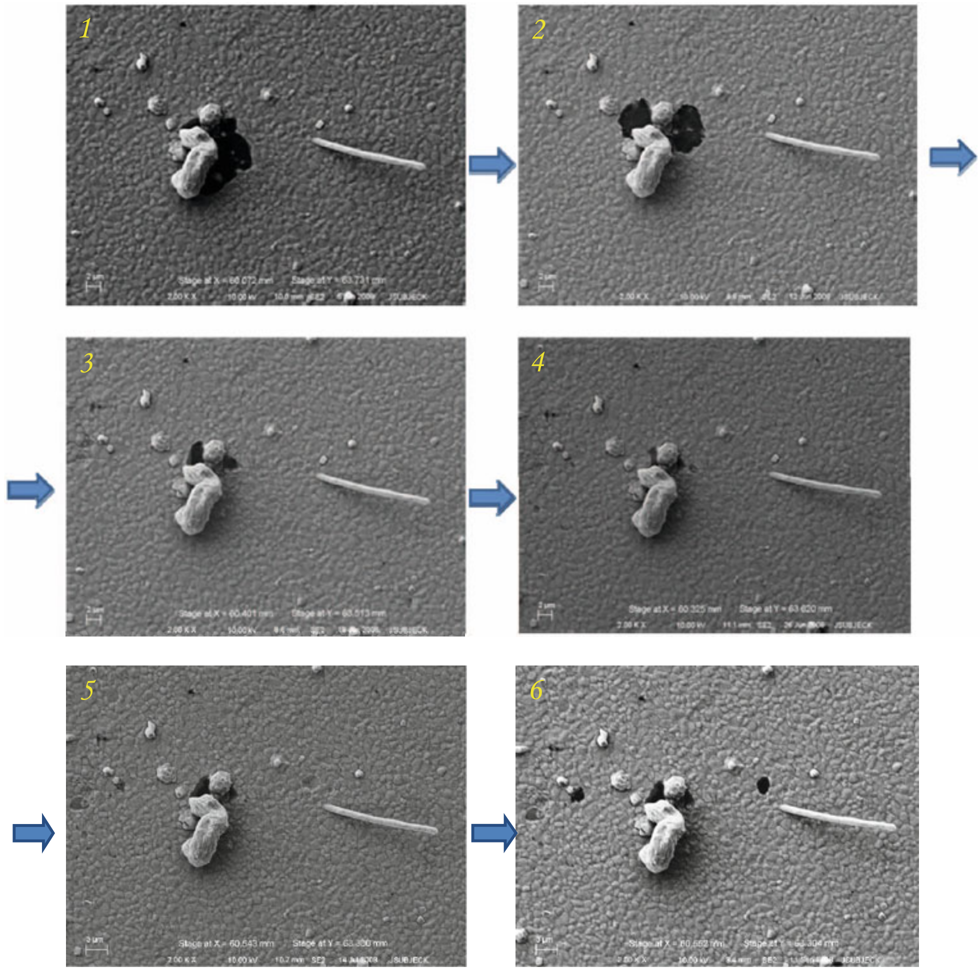

Fig. 4 Tracking whiskers and depleted zones through a 6 weeks annealing procedure. The sample was annealed at 180 °C, in vacuum at 10^{-4} Torr and under an applied compressive 500 g load

Discussion

Distribution of whiskers across the Sn film within the first annealing cycle

Compressive stress

It was noted previously that the Sn whiskers appeared after the first 1 week period of annealing. Afterwards, the number and length of the whiskers did not change over the remaining seven 1 week intervals. The compressive stress had a distribution across the sample radius that is shown in Fig. 2. Thus, whisker length was then correlated with the local stress and plotted in two manners in Fig. 9. The total length of whiskers served as a surrogate parameter for the total number of whiskers (when divided by the average length). That parameter is plotted in Fig. 9a, which shows a gradual decrease with decreasing compressive stress. The trend was hardly monotonic per the mean values; however, those fluctuations were just outside the statistical significance of the data. Also, at locations of 9 and 11 mm from the center, the data exhibited particularly large error bars. The average whisker length exhibited a weaker trend as a function of location from the wafer center (Fig. 9b). The mean values also exhibited a slow decrease with distance from the center.

The whisker length data were plotted as a function of stress; the plots are shown in Fig. 10. (The direction of the *x*-axis is from low to high stresses, which is from the outer edge to the center of the wafer.) The means of the total whisker lengths increased with the magnitude of the compressive stress from 0 to 15 MPa and then leveled-off at stresses greater than 15 MPa (Fig. 10a). A similar trend was observed for the average whisker length parameter in Fig. 10b, except these data suggest that the plateau had began at a lower stress of 5 MPa.

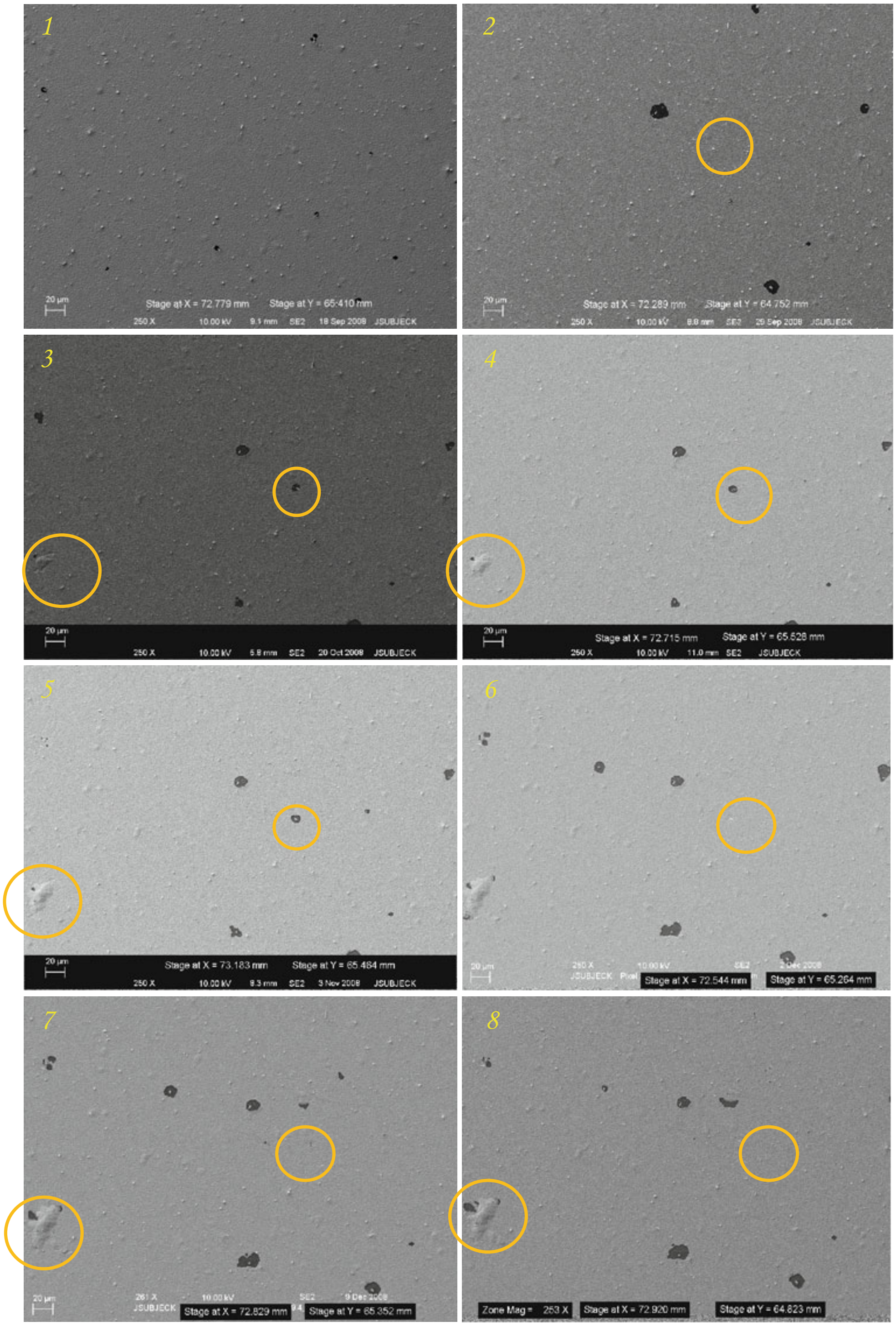

Fig. 5 SEM images that show the same area after each of the eight annealing cycles at a distance of 4 mm from the center point under the tensile stress of 6.5 MPa). The sample was exposed to 180 °C in vacuum at 10^{-4} Torr during application of the tensile 500 g load

The data presented in Fig. 10 indicated several characteristics of whisker growth with respect to the applied compressive stress. First of all, according to Fig. 10b, creating additional compressive stress does not automatically lead to an increased length of the whiskers. There appeared to be an upper limit of the average length—about 15 µm—reach at 5 MPa. Second, combining the results in Fig. 10a and b, increasing the stress further, from 5 MPa to approximately 15 MPa, increased the *number* of whiskers (total whisker length) rather than

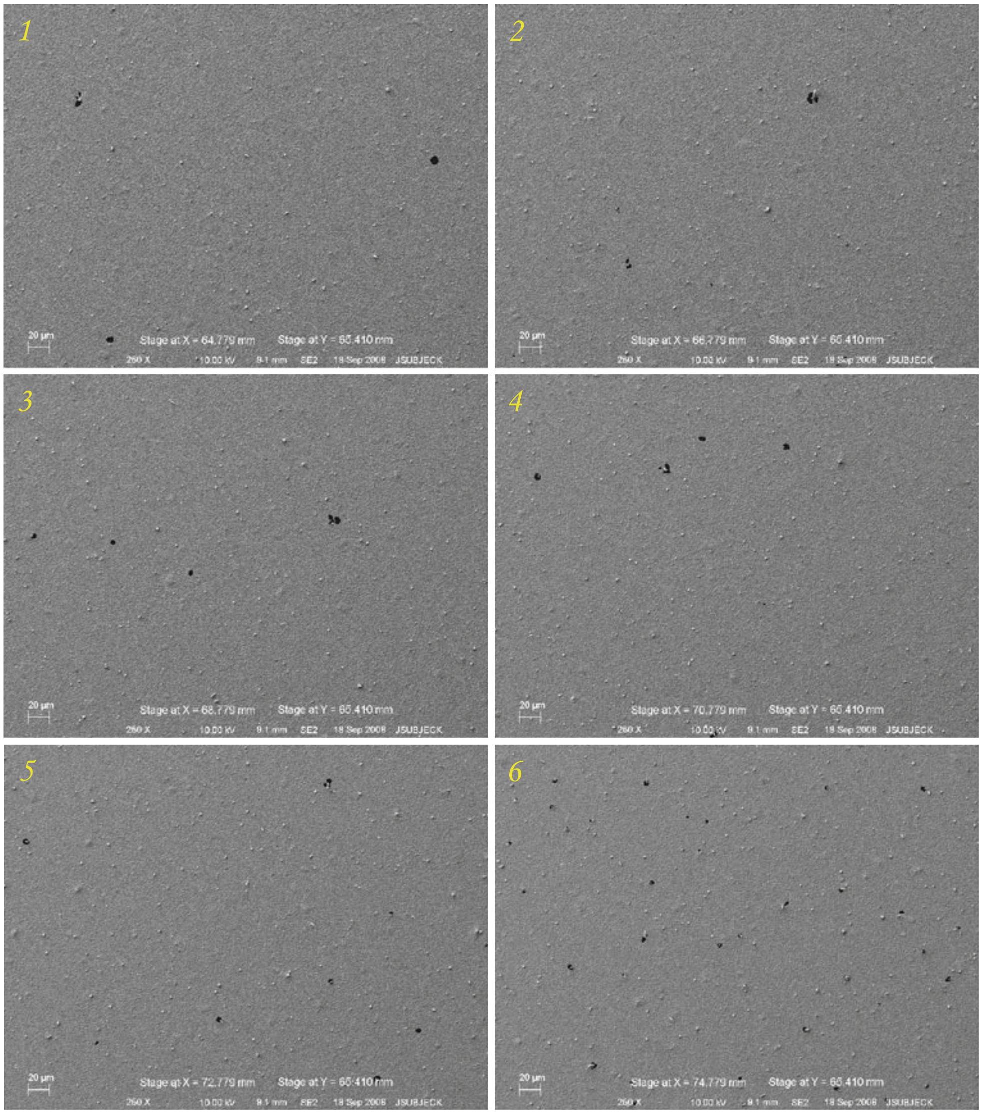

Fig. 6 SEM photographs showing whiskers and depleted areas after the first week of the annealing treatment. The sample was exposed to 180 °C in vacuum at 10^{-4} Torr. The observation were taken at (*1*) 2 mm, (*2*) 4 mm, (*3*) 6 mm, (*4*) 8 mm, (*5*) 10 mm, and (*6*) 12 mm from the center of tin film, respectively

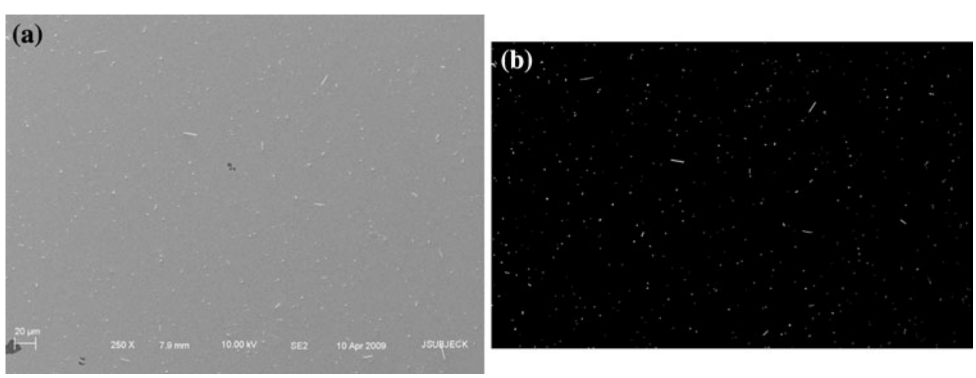

Fig. 7 **a** The original *gray*-tone SEM image. **b** *Black-white* image after MATLAB processed the image for whiskers

extending the lengths of those whiskers that have already been formed. Then, at stresses above 15 MPa, there is no further development of whiskers—either length or number. This trend implied that these higher stresses led to an alternative deformation mechanism to alleviate the compressive stress.

Table 1 The number and length of whiskers calculated by MATLAB program

Serial no. of whisker	Length of whiskers (μm)
1	21.02602
2	16.29517
3	9.46171
4	29.43643
5	9.987361
6	14.19257
7	21.55167
8	10.51301
9	7.884758
Maximum length	29.43643
Total length	140.3487
Average length	15.5943

The lengths represent the original length values multiplied by 1.414 to reflect their 45 degree orientation

Tensile stress

The quantitative analysis was performed on total whisker length and average whisker length as a function of tensile stress across the wafer. The stress values (wafer locations) are 1.03, 2.78, 4.93, 7.69, 11.58, and 18.24 MPa, respectively. The corresponding plots are shown in Fig. 11.

The total whisker length decreased with increasing tensile stress. The data confirms the fundamental premise that applied tensile stresses reduce the propensity for whiskers to grow from the Sn film. However, it does not prevent such growth, altogether, because of internal stresses, which when exceeding locally the applied tensile stress, can generate whiskers. In the present samples, that maximum total length was 20 μm, which occurred at the maximum tensile stress.

The second plot is that of average whisker length. The change in tensile stress did not significantly affect the average whisker length, which remained at approximately 5 μm. This trend differed from that of the average whisker length as a function of the compressive stress. Based on the superposition of applied stresses and intrinsic stresses, the fact that average whisker length converged on 5 μm for both tensile stresses and minimum compressive stress confirms the concept of localized intrinsic stresses, in even the evaporated Sn films, that are capable of generating whiskers.

Table 2 Quantitative analysis results for depleted areas

Serial no. of depleted area	Depleted area (μm^2)
1	7.384615
2	19.61538
3	19.84615
4	17.92308
5	7.230769
6	3.615385
7	24.07692
8	8.076923
9	21.76923
10	33
11	13.30769
12	8.461538
13	28.15385
Maximum area	33
Total area	212.4615
Average area	16.3432
Standard deviation	9.113281

Evolution of depleted areas over the eight annealing cycles

Compressive stress

It was observed in Figs. 3 and 4 that the depleted area changed with the accumulation of annealing cycles. The quantitative analysis of this phenomenon is presented in Fig. 12, which is a plot of the total depleted area as a

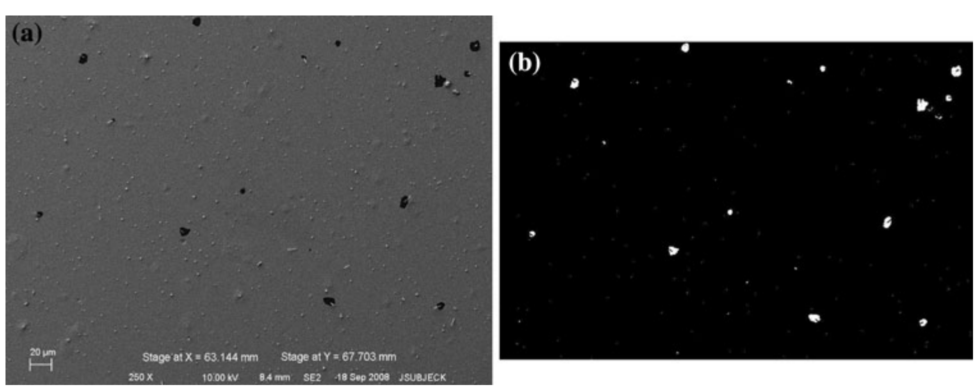

Fig. 8 **a** An original SEM *gray*-degree image. **b** *Black-white* image after MATLAB processing including more depleted area

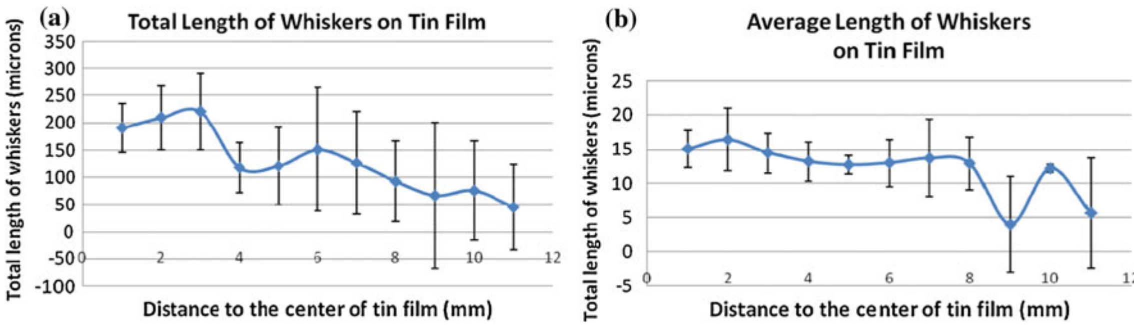

Fig. 9 Whisker distribution on Sn film surface from the center to the edge by analyzing the total length and average length of the whiskers. The total annealing period was 1 week

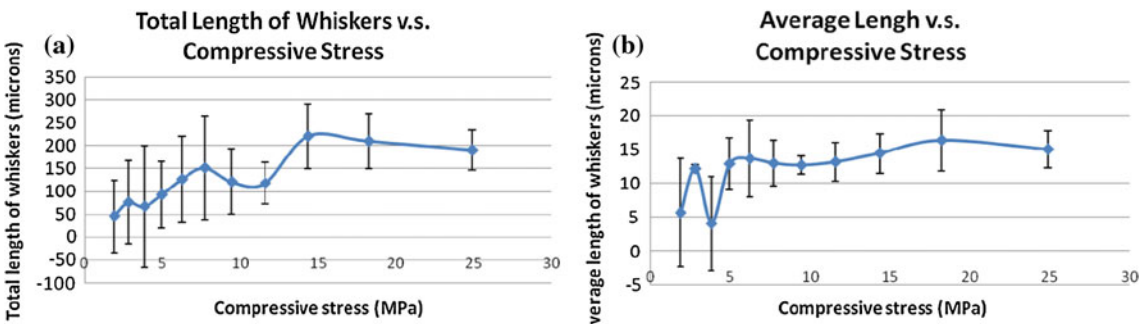

Fig. 10 Whisker development versus compressive stress by analyzing **a** total length and **b** average length. The total annealing period was 1 week [28]

function of compressive stress across the Si disk radius. The different colors reflect the annealing intervals 2–8 weeks. The week one data were omitted because the images were not sufficiently clear for the quantitative image analysis routine. However, it can be inferred from Fig. 12 that the total area should be less than 100 μm^2.

The depleted areas did not exhibit a monotonic trend as a function of compressive stress across the Sn film surface for each annealing period. It was interesting that the fluctuations and, in particular, a peak at the stress value of 4.93 MPa, were reproduced at each annealing interval. The total depleted area increased as a function of accumulated number of annealing cycles. The trend was just within the measurement error bars. It is important to recognize that the overall increase of depleted area reflects a net effect because, locally, area sizes increased or decreased with cumulative annealing time as was shown in the SEM photographs, earlier.

Recall that the Sn whisker growth caused by a compressive stress (Fig. 10), exhibited a dependency on stress. However, the whisker growth data did not exhibit a significant sensitivity to the annealing treatment. On the other hand, the depleted zones showed a strong dependence on annealing time. Therefore, there was not a direct correlation between the total whisker length and the total depleted zone area. This comparison suggests that the respective mass transport mechanisms may not be the same for both phenomena. This observation implies that the whiskers do not necessarily serve as a repository for the Sn atoms that are lost from the depleted regions. In fact, there is an absence of local regions of Sn accumulation—whiskers or hillocks—near every growing depleted zone. Rather, the Sn film as a whole acts as a sink or source of Sn to be used as such by the development of either a depleted zone or whisker, respectively. Clearly, the mass transport process that is responsible for the depleted areas is also long-range in nature.

Zero stress

The changes to the depleted zone footprints were further explored by annealing a sample *without* an applied stress. The SEM images are shown in Fig. 13, which were obtained at locations of 2, 4,…, 12 mm from the center of the wafer after the 1 week of annealing. There were depleted areas observed in the Sn film. The distribution of the depleted areas did not exhibit a dependence on the location across the Si wafer, as expected in the absence of a stress. There are two possible causes for zones to continue in the absence of a stress: (a) The depleted areas are created by *local* stress fields. (b) The depleted areas are not stress driven; rather, they reflect the same Sn mass transport as

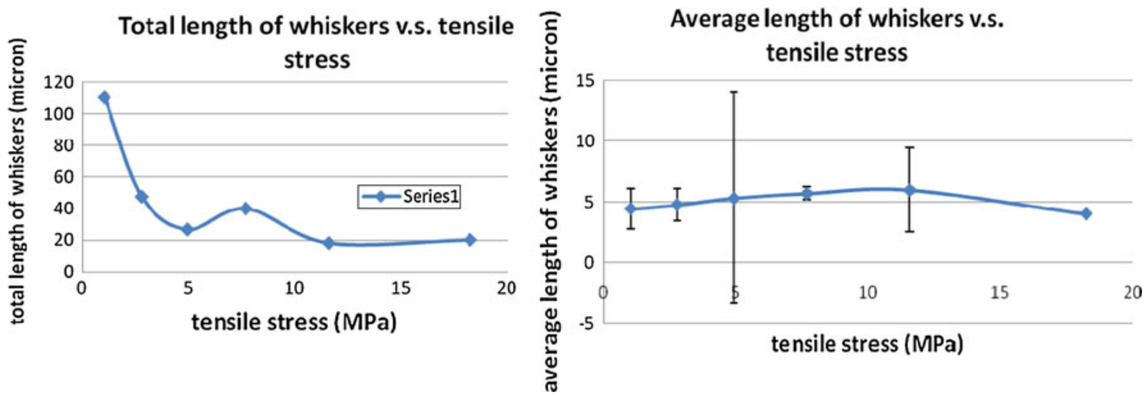

Fig. 11 **a** Total whisker length and **b** average whisker length after 1 week annealing period when exposed to a tensile stress

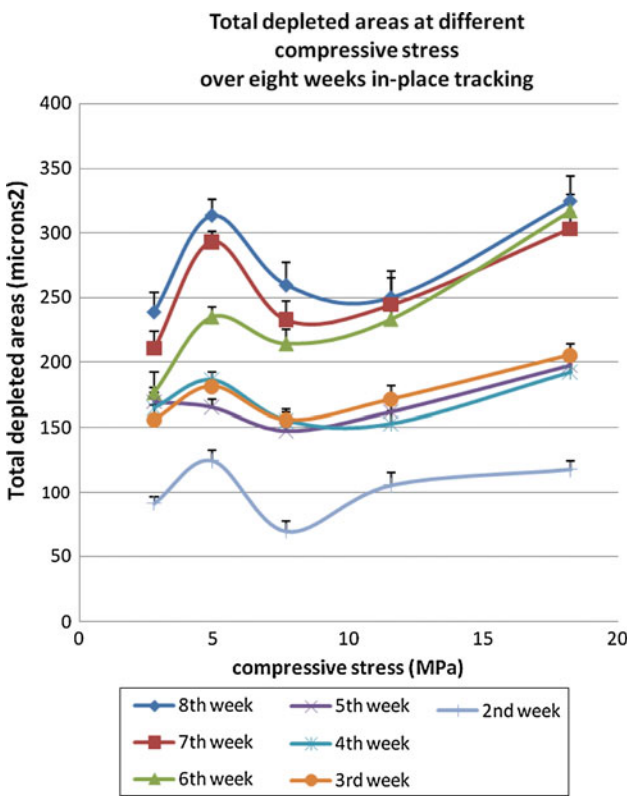

Fig. 12 Total depleted area as a function of compressive stress for the accumulated annealing periods 2–8 under the compressive stress. The stress value along the *x*-axis is 2.78, 4.93, 7.69, 11.58, and 18.24 MPa, respectively

support whisker growth, but under the effect of a different driving force.

Mechanism of Sn mass transport

As described above, both Sn whisker growth and the depleted area formation require a rapid, long-range mass transport of Sn atoms through the thin film. The differences in the details of their respective development histories indicate that the driving forces and/or rate kinetics for the two phenomena are not be similar.

There are four possible pathways for the Sn transport:

(1) the interface between top surface oxide layer and the Sn film;
(2) deformation structures within the Sn grains [26];
(3) grain boundaries in the film; or
(4) the interface between the Sn film and substrate.

The first scenario does not appear to be likely given that whisker growth has been observed for elemental metals that grow a very limited oxide—e.g., gold (Au). So, the oxide layer is not a necessary condition for whisker development. Woodrow [27] used isotope tracer to track the Sn atoms movement, and found a possible pathway to be the interface of Sn film and substrate [24]. The second scenario is diffusion through the grains of the Sn film. Certainly, deformation structures can develop within the grains due to the compressive stress. Those structures may provide a faster diffusion path (e.g., dislocation core diffusion). But there is no direct evidence to support it so far.

The third scenario was mass transport along grain boundaries. These 1 μm thick films are a single grain thick. Therefore, there are no boundaries that are oriented horizontally in the plane of the film to provide a path for Sn atoms.

The fourth path has evidence to support it. The formation of depleted zones has the Sn atoms removed from the bottom of the area, which implies that there is transport along the Sn/substrate interface. If it is assumed that the same long-range mass transport mechanism governs whisker growth, then a likely transport path is also the Sn/substrate interface. The Sn transport would take place along the Sn/Cr interface. In the case of depleted areas, the Sn atoms were "emptied" from the grain by diffusion along the through-thickness grain boundaries to the Sn/Cr interface. The atoms then diffuse along the latter interface.

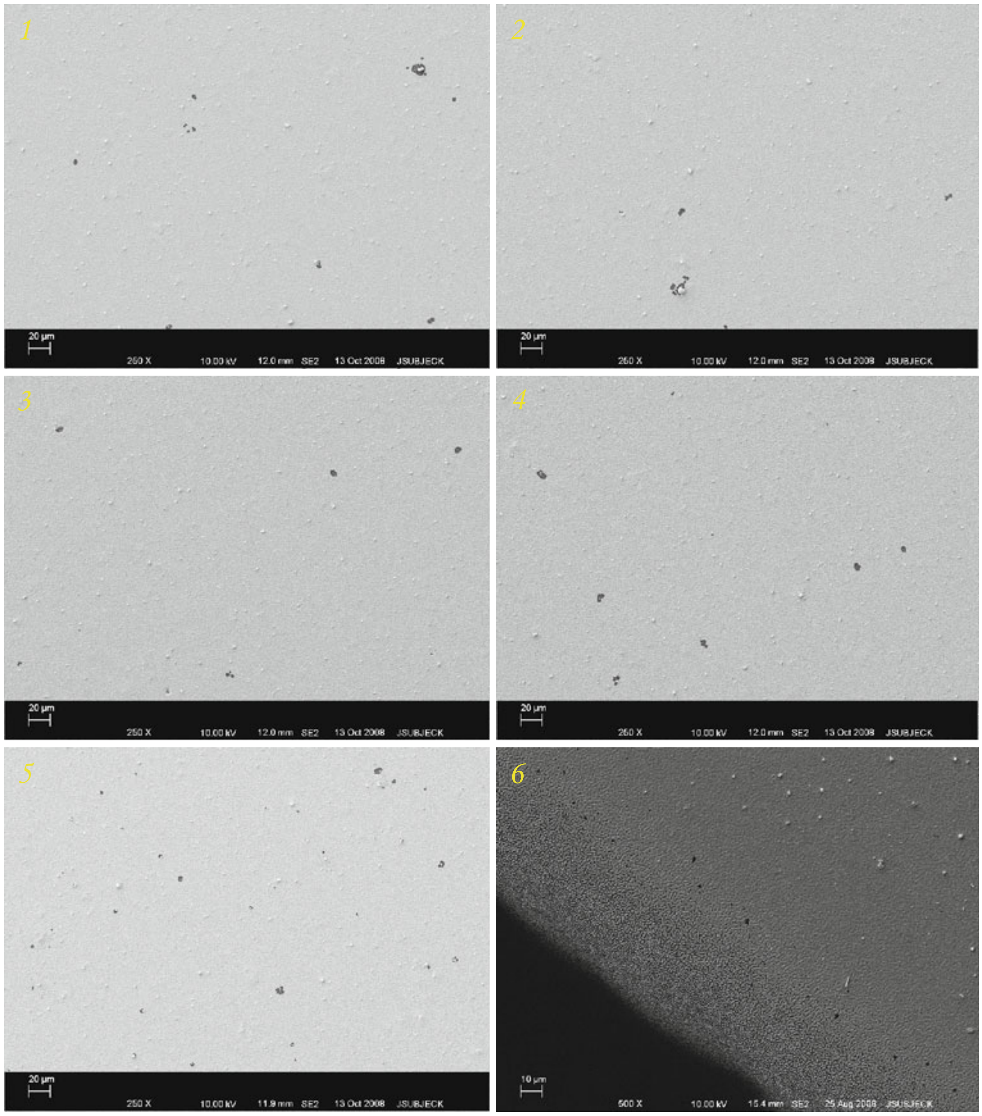

Fig. 13 The sample annealed at 180 °C in vacuum at 10^{-4} Torr without an applied stress. From (*1*) to (*5*), the distances from the center to the edge are 2, 4, 6, 8, 10, and 12 mm, respectively. Note that (*6*) has higher magnification than the others

In the case of whisker development, mass transport along the Sn/Cr interface would supply Sn atoms to the base of the whisker [18].

Conclusion

An experimental technique was developed to determine the extent of Sn whisker growth and depleted zone formation on evaporated 1 μm Sn films. Deformation of the Si substrate placed a controlled magnitude of compressive/tensile stress on the films. Quantitative image analysis was used to monitor whisker length and depleted area. The conditions used to generate these phenomena were: compressive or tensile stresses of 2–20 MPa; temperature of 180 °C; and time durations between 1 and 8 weeks.

The total and average whisker length increased with compressive stress levels to levels of the latter of 15 and 5 MPa, respectively. The whiskers appeared within the first 1 week annealing interval and did not grow significantly with time. Some whiskers were located in the centers of depleted areas. Under applied tensile stress, the appearance of whiskers confirmed the role of local, compressive stresses within the Sn films.

The depleted areas exhibited fluctuations as a function of applied stress, both compressive or tensile. There was no discernable, overall trend as a function of the magnitude of the compressive stress. In the absence of an applied compressive stress, the depleted areas were still observed. The area increased with annealing time.

It was concluded that Sn whiskers and depleted areas were the result of the same rapid, long-range diffusion processes. The specific mechanisms of mass transport were narrowed to two scenarios based upon this study: (a) the combination of Sn/Cr interface fluid transport and vertical grain boundary diffusion or (b) an intra-granular, deformation structure mechanism. Nevertheless, the driving forces and/or rate kinetics appeared to differ between the whisker growth and depleted zone formation.

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