Automatikus karakterizáló módszer ón-whiskerek növekedésének statisztikus kiértékeléséhez - Kutatási jelentés

1. INTRODUCTION

Tin whiskers are 1-10 μm thick and 10-500 μm long crystal eruptions which can form on surface coatings and solder joints with high Sn content [1–3]. They can cause reliability issues in microelectronics via short circuit formation between the leads of the components. Tin whisker growth is induced by compressive mechanical stress in the Sn layer which can originate from: residual stress in the Sn deposition; a direct external mechanical load; volumetric expansion by material transformation (intermetallic and oxide growth) and thermomechanical effects [4]. Whisker growth is a stress release response to compression. The main properties of the Sn layer which have an effect on whisker formation are the thickness and the grain parameters (size, structure and orientation) [5,6]. Tin whisker growth can be reduced by increasing the Sn layer thickness [7]. A smaller grain size can hasten the growth of the intermetallic layer and oxides, therefore more stress might arise in this way [8], however, a smaller grain size on the other side ensures better stress relaxation capacity of the layer. The grain orientation also has some minor effects on the tin whiskering behaviour of the layer [7,9].

Three technologies are applied in the electronics industry to prepare Sn layers: electroplating, chemical (immersion tin plating) and vacuum evaporation. Tin layers produced by electroplating or by immersion technology are usually used for surface coatings in soldering technologies [10]. Vacuum evaporated Sn and Cu-Sn thin films are applied as a bonding layer to Cu conductors in electronic devices [11] and for anode material in lithium-ion batteries [12]. Tin whisker growth from vacuum evaporated Sn layers is a less researched area (compared to the electroplated or immersion tin layers). Kehrer and Kadeit observed whisker growth from a 700 nm thick evaporated Sn layer [13]. The phenomenon even occurs at room temperature as was proven by Rodekohr et al. [14] and Chen and Shih [15]. Illés et al. showed that evaporated tin layers on copper substrate can develop very large amount and various types of tin whiskers at room temperatures even after only few days of the layer deposition [16].

Measurement of whisker length and density, and calculation of their statistics are critical aspects of tin whisker investigations. The tin whiskers are usually observed by Scanning Electron Microscope due to their micron scale thickness [17], however in some cases they were studied by optical microscopes as well [18,19]. The axial length of a whisker is usually measured according to the JESD201 standard (the distance between the tip of the whisker and the surface) [20]. D. Susan et al. developed a method to measure whisker lengths and angles with a time-lapse in situ SEM study [21]. In the case of rare whisker appearance but with high lengths, usually the authors measure only the average whisker lengths [22]. The maximum whisker length is an important parameter, since this may indicate the risk of short circuit formation [23]. In the case of dense but short whiskers, the whisker density on a given surface is a more informative parameter [17], and it is usually calculated from 10–25 different SEM micrographs [5,20]. Sometimes, whiskers under a given length (e.g. 10 μm) were omitted during the calculations as non-dangerous objects [24]. In statistical studies about tin whisker phenomenon, the authors usually apply lognormal distribution to describe whisker density or length [24], since this distribution represents that tin whisker populations tend to have many shorter whiskers and fewer longer ones [25]. Other statistical methods (e.g. Duncan’s test) can also be found in, in the work by Skwarek et al [26]. Nonetheless, the authors usually do not pay enough attention to perform the measurements reproducibly, do not provide enough information about the applied statistical evaluation methods [27], and even nowadays, most whisker researchers continue to rely on manual whisker counting in SEM images. There is still no automated method to detect, count, and to measure whiskers [28], which makes the results of different studies difficult to compare. According to our literature review it can be concluded that there is a need for an automatic and standardisable evaluation method for whisker parameters which would provide comparability of the results from different researches.

2. MATERIALS AND METHODS

2.1. Experimental

A method based on the image processing was developed to automatically characterise whiskers. The key step of the image processing method in this case is the image segmentation using binarisation, i.e. separating the objects (whiskers) from the background (substrate). A new thresholding method was developed for the binarisation. The whisker parameters obtained by the method reported here were compared to manual counting as a reference, and to four widely used automatic methods for general image processing purposes as well: to the method by Otsu [29] – a clustering analysis method based on image variance; to the method by Weszka [30] – based on the determination of the rate of change of grey-level around each pixel in the image; and to two methods, which are based on determination of the Entropy of the image derived from its histogram, described by Pun [31] and Kapur [32] respectively. The latter three methods are described in the Appendix in details.

It was proven in previous studies that the evaporated tin layers on copper substrates can produce numerous tin whisker in various shapes and lengths in a short time, this layer deposition technology was chosen therefore for the comparison of the thresholding methods. During the sample preparation, 99.99% pure Sn was vacuum evaporated onto 1.5 mm thick copper substrates. The evaporation was carried out with a Balzers BA 510 evaporator utilising the Electron Beam – Physical Vapour Deposition (EB-PVD) method. Before the evaporation, the samples were cleaned in isopropyl alcohol and were neutralized by ion bombardment (directly in the evaporator). The applied cathode heating current was 100 mA with 7 kV acceleration voltage. A high vacuum (10^{-3} Pa) was used, and the evaporation duration was 25 minutes. The deposition resulted in an average of 400 nm thick Sn layer with 1–1.5 μm sized globular grains. The samples were then stored at room temperature for up to 150 days to have whiskers in many different lengths. Whisker growth was monitored every 15 days by a FEI Inspect S50 Scanning Electron Microscope (acc. voltage 20 kV, magnification 1000x).

2.2. Automatic method based on calculation of the Mean Intercept Length of objects

The self-developed automatic method, which is developed in Matlab software, includes a new thresholding method to separate the objects (whiskers) from the background (substrate). A global thresholding method was developed, since no illumination inhomogeneity appears in SEM images.

Our thresholding method is based on the calculation of the mean intercept length (1) of the separated objects, and averaging it both in vertical and horizontal direction. The calculation of the mean intercept length is described in the ASM Handbook [33]; and in the ASTM E112-12 standard [34] as the Heyn Lineal Intercept Procedure:

$$L = \frac{L_L}{N_L} \tag{1}$$

where L_L is the sum of linear intercept lengths per a unit length of test lines (L_T), and N_L is the number of interceptions of objects per a unit length of test lines (Fig. 1). An intercept ($L_{intercept}$) is a segment of test line overlaying one object [34]. The length of the test lines (L_T) are the width (horizontal direction) and the height (vertical direction) of the SEM image.

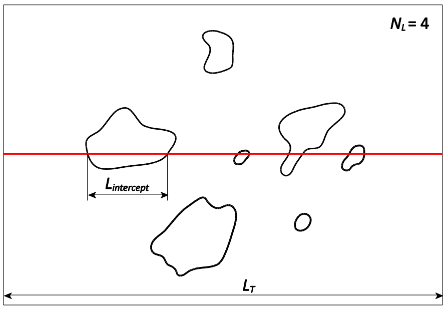

Fig. 1 Determining the mean intercept length (one test line is indicated by the red line; $L_{intercept}$ is indicated for one object; N_L is 4 in the example) [33]

The mean intercept length strongly correlates with the size of objects for disoriented structures, and with the width of objects for highly anisotropic structures respectively. Whiskers are highly anisotropic in size and their thickness is quite even. Besides, their average thickness (1.75 µm) differs from the average grain size of the substrate (1.25 µm). So, it has been found that if the mean intercept length (*MIL*) is calculated for every possible threshold level: (t = 1 … `maximum_intensity`), a definite value on the *MIL* function can be found, which represents the whiskers; and its argument is the optimum threshold level. The parts of the *MIL* function (illustrated in Fig. 2) can be described as follows:

1. At first, the image is completely white after binarisation, and the value of *MIL* equals to the average of the height and of the width of the image (277 µm in our case).
2. The *MIL* function then decreases, as the general image noise is more and more eliminated, indicated by mark I in Fig. 2.
3. The function reaches a local minimum, which represents the value of *MIL* regarding the noise induced by surface roughness of the substrate.
4. Next, the *MIL* function increases, as the noise induced by the substrate surface is eliminated gradually (indicated by mark II. in the Fig. 2).
5. The function reaches a local maximum, which represents the *MIL* for the whiskers. The threshold value belonging to this point is the optimal one.
6. The *MIL* function then decreases again, because more and more details are excluded from the edges of the whiskers (III. in Fig. 2).
7. In the end, at the threshold value of 255, the image is completely black after binarisation, and the *MIL* equals to zero.

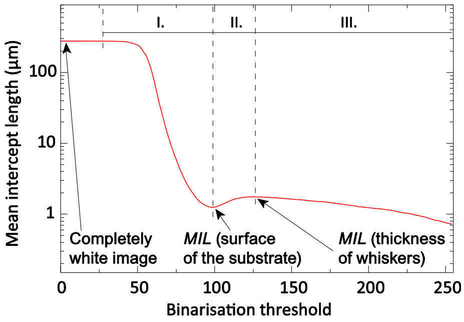

Fig. 2 MIL function for the optimal binarisation threshold level determination (value is 125 in this case)

To find the optimal threshold level, the local maximum of the *MIL* function is sought after the first local minimum of that function (2). The threshold value belonging to this local maximum is the optimal one (t_o). It should be noted that special attention was paid to acquire the SEM images with as identical contrast settings as possible, so that the threshold level belonging to the first local minimum of the *MIL* function is lower than the value of 110. The threshold level at the next local maximum is expected to be lower than 160. Besides, general noise reduction was applied before calculating the *MIL* function utilising MATLAB's `bwareaopen` and `imclose` functions.

$$\begin{aligned} [\sim, t_i] &= \min\left(MIL(t)\right) \ \{t \mid t \in \mathrm{N} \wedge 0 < t < 110\}, \\ [\sim, t_o] &= \max\left(MIL(t)\right) \ \{t \mid t \in \mathrm{N} \wedge t_i < t < 160\} \end{aligned} \tag{2}$$

where $MIL(t)$ is the mean intercept length as a function of threshold level, t_i is a temporary variable, and t_o is the optimal value of the threshold.

After the segmentation, the number of whiskers was calculated by labelling all the connected components with `bwlabel` function of Matlab software. The adjacency for the area opening was set to value 8 (p and q are adjacent if $q \in N_8(p)$). The number of whiskers equals to the maximum element ID in the label matrix. The length of the whiskers was determined by overlaying ellipses and rectangles onto the connected components (`regionprops`) and by measuring their major axis and diagonal lengths respectively. This method is perfect for indicating the risk of short-circuit formation, if the length of whiskers exceeds a certain limit, e.g. the distance between two leads of a fine-pitch component (~200 μm). Concerning the two length parameters obtained from overlaying ellipses or rectangles, the shorter should be selected. For very straight whiskers, the major axis of the ellipse measures over the length of the whisker, while the diagonal of the rectangle provides accurate projected length (Fig. 3).

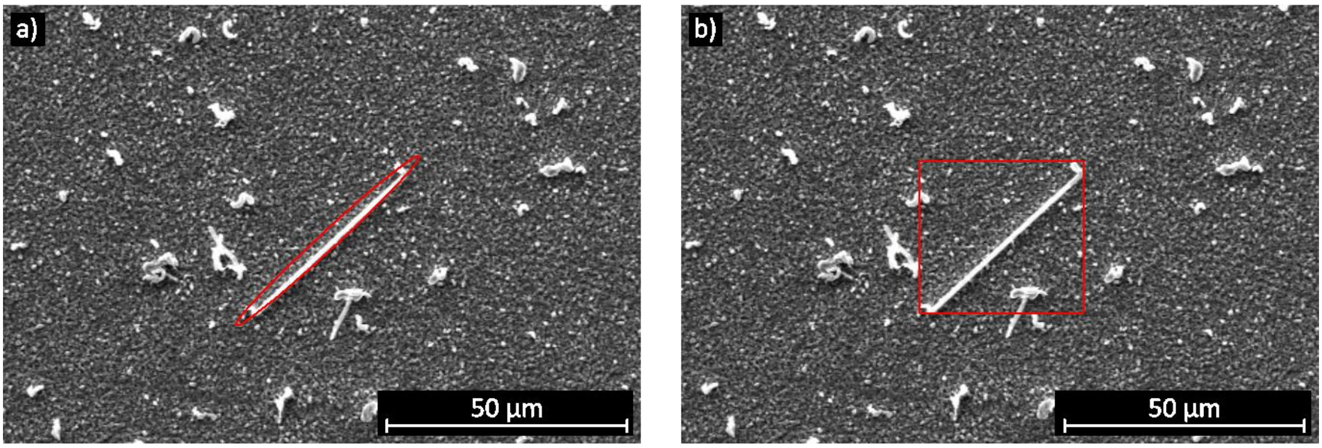

Fig. 3 Measuring the length of whiskers: a) by the major axis of ellipse (length is 52 μm), b) by the diagonal of rectangle (length is 46 μm) – only one whisker is measured for illustration purposes

3. RESULTS

To begin with, thresholding quality was evaluated visually. The self-developed method, Otsu's method and Kapur's Entropy-based method provided binarised images, which are suitable for analysis. Contrary, Weszka's and Pun's thresholding method could not separate the objects (whiskers) from the background (substrate); the provided binarised images could thus not be analysed. The binarised images obtained by different thresholding methods are illustrated in Fig. 4.

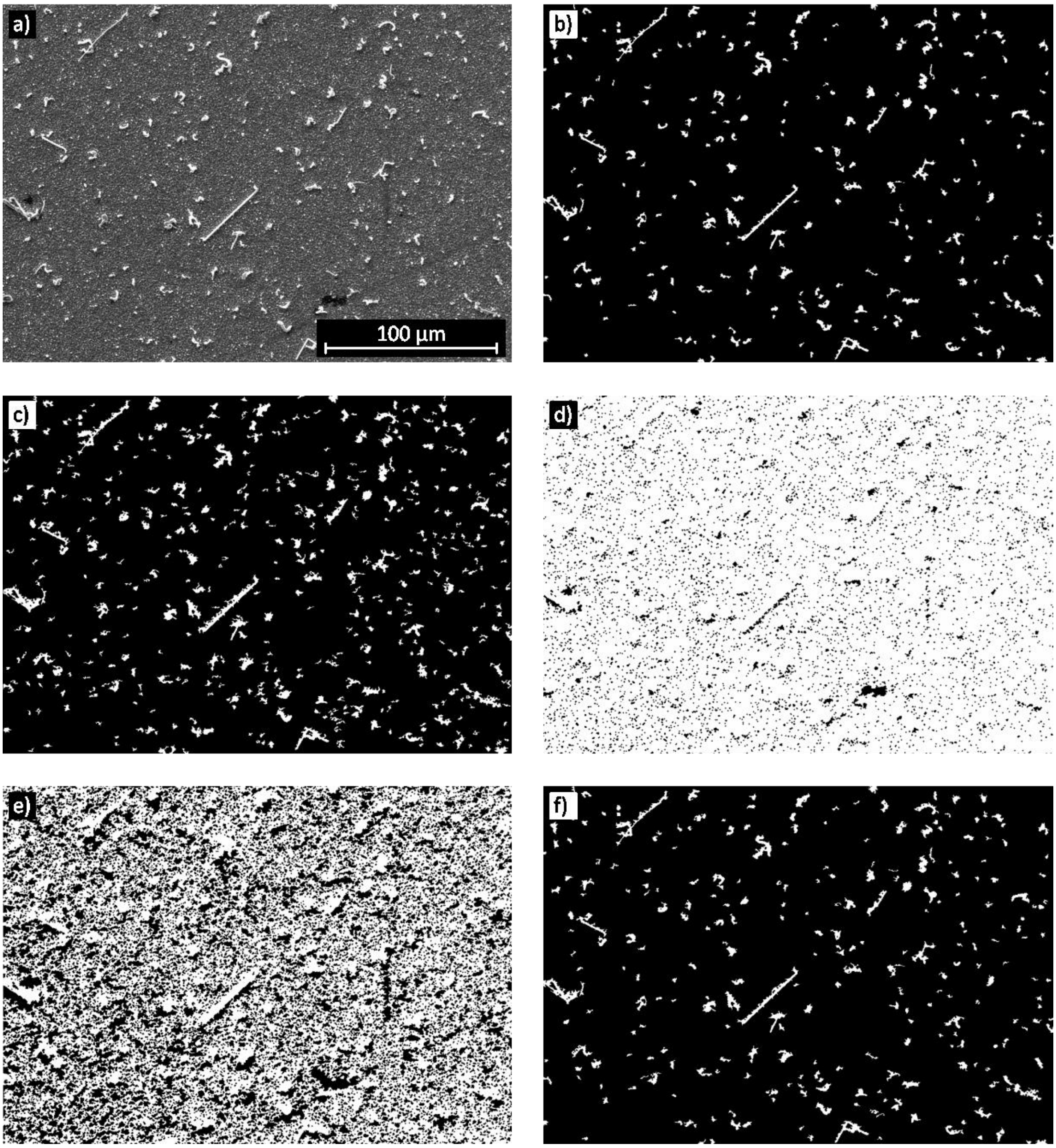

Fig. 4 Automatic thresholding methods comparison: a) original image, b) self-developed, MIL-based, method, c) Otsu's method, d) Weszka's method, e) Pun's Entropy-based method, f) Kapur's Entropy-based method

The density of whiskers was calculated (Fig. 5), and the results were compared to the results by manual counting in 35 images. The comparison was performed by calculating the MAPE (Mean Absolute Percentage Error) between the automatic thresholding methods and the manual counting (Fig. 6).

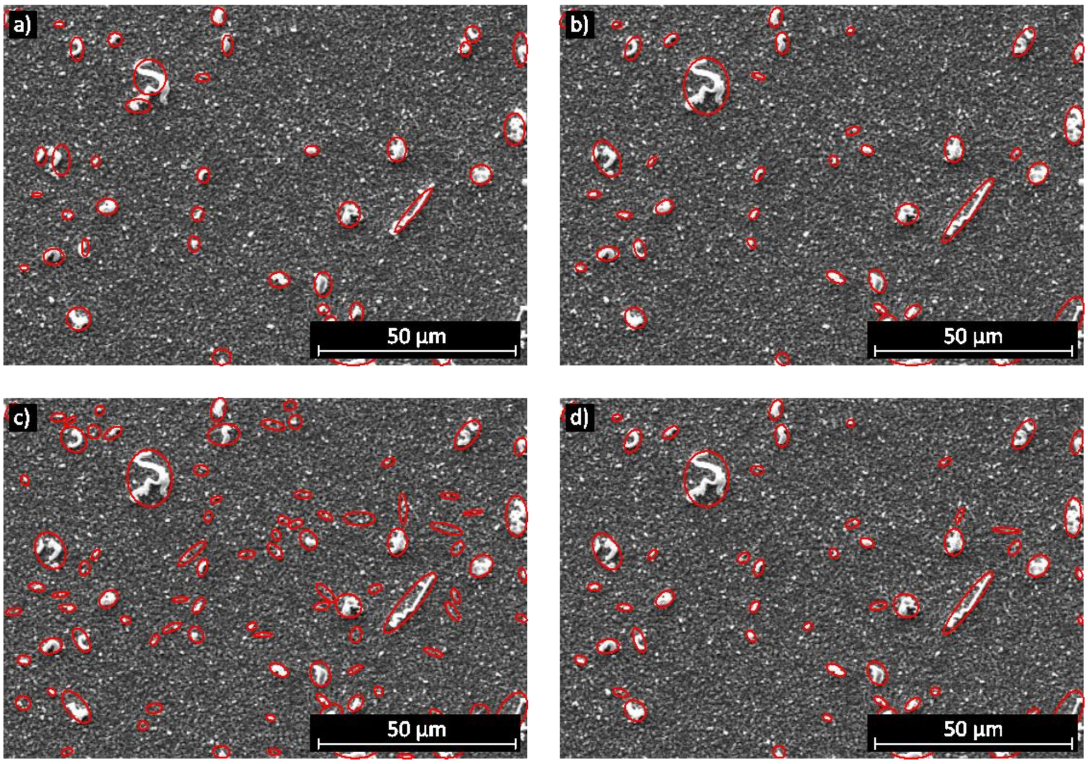

Fig. 5 Calculating the whisker density: a) manual selection and counting (density is 3441 pcs./mm2), b) self-developed, MIL-based, method (density is 3359 pcs./mm2), c) Otsu's method (density is 7538 pcs./mm2), d) Kapur's Entropy-based method (density is 4342 pcs./mm2)

It can be seen that Otsu's method measured significantly more whiskers than were present due to the inclusion of many artefacts resulting from roughness of the substrate. Kapur's method estimates better the whisker density, but still, it is also sensitive to the noise induced by the surface roughness of the substrate.

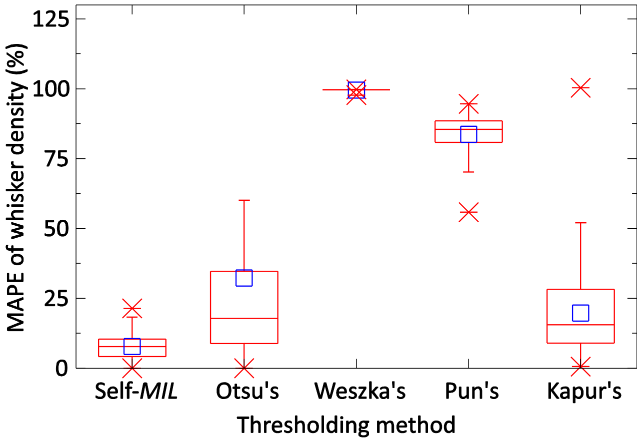

Fig. 6 Mean Absolute Percentage Error of whisker density obtained by different automatic thresholding methods

The automatic thresholding methods were compared also from the viewpoint of measured whisker lengths. The mean and the maximum length of whiskers were also determined by Otsu's and Kapur's methods: by calculating the length of the major axis of overlaid ellipses and by the diagonal length of overlaid rectangles. The MAPE for the two reference automatic methods (Otsu's and Kapur's) was calculated by comparing their results to the results obtained by the self-developed *MIL*-based method (Fig. 7). The comparison was performed by the automated analysis of 91 images with whiskers.

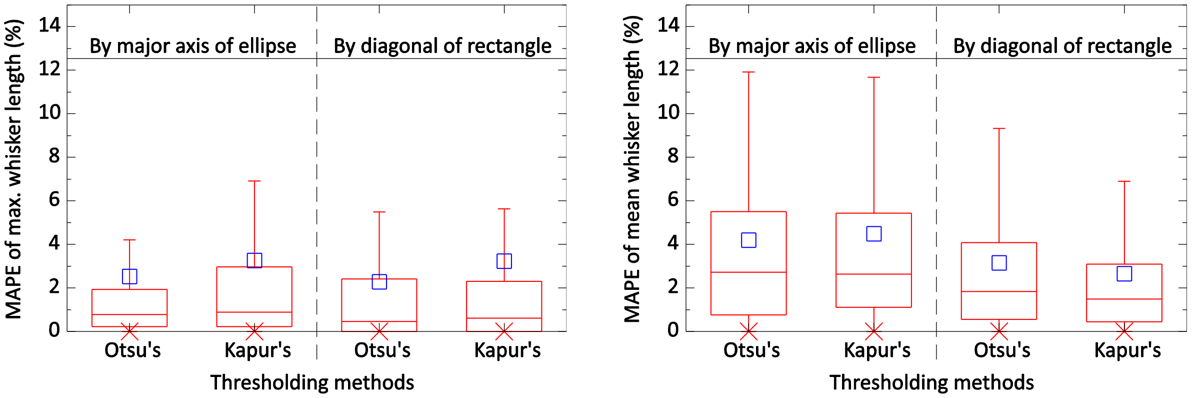

Fig. 7 Mean Absolute Percentage Error of whisker lengths. Comparison of two reference thresholding methods (Otsu's and Kapur's) to the self-developed one: a) maximum length of whiskers, b) mean length of whiskers

As can be seen in Fig. 7, the two reference methods performed nearly the same as the self-developed method; the determined whisker length differs only by few percent (maximum length by 2–3%, mean length by 3–4%). However, there were some outliers (~43%) especially in the MAPE of maximum whisker lengths. The reason for this is illustrated in Fig. 8. It can be seen that there is some contamination exactly in the middle of a long whisker causing an image intensity drop. The reference methods interpreted this whisker as two individual whiskers, which resulted in a large error in the maximum whisker length calculation.

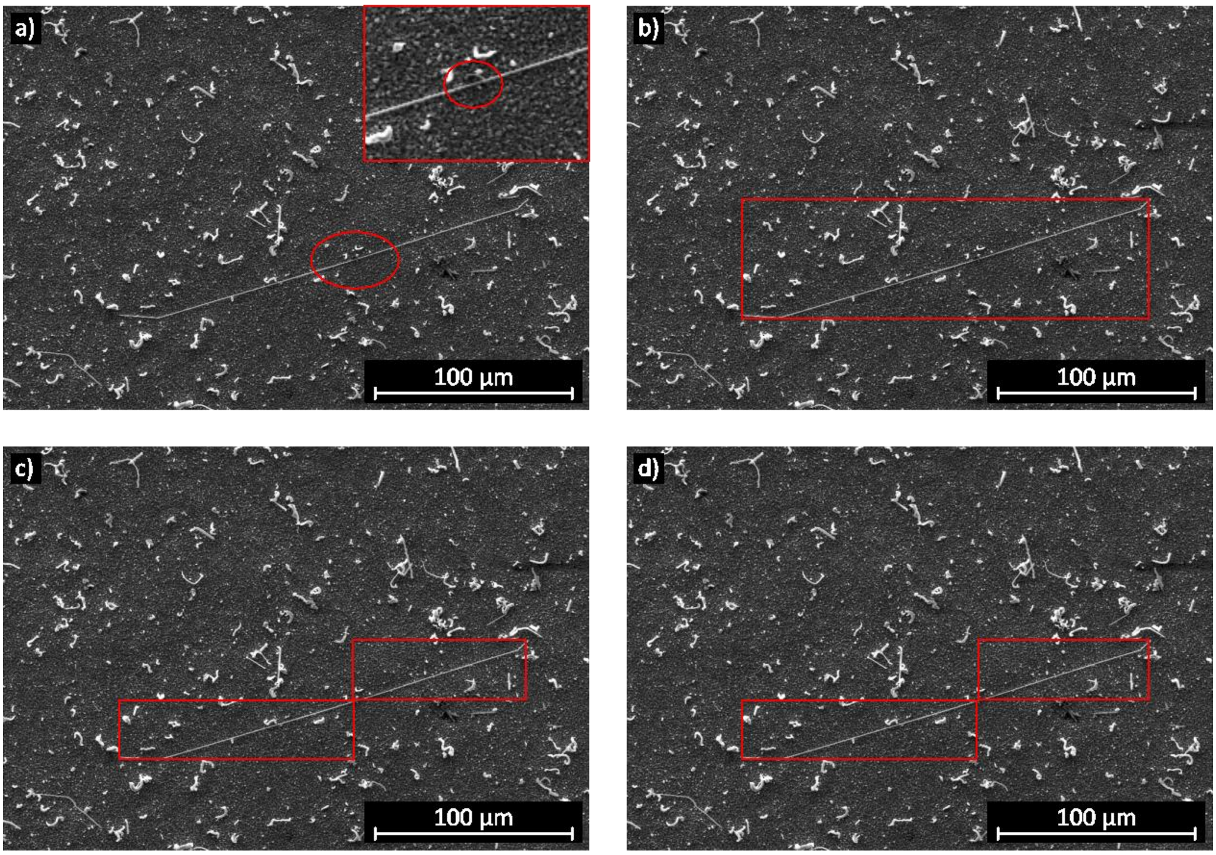

Fig. 8 Measuring the maximum whisker length: a) original image, b) self-developed, MIL-based, method (length is 216 µm), c) Otsu's method (max. length is 123.5 µm), d) Kapur's Entropy-based method (max. length is 123.2 µm) – only one whisker is evaluated for the convenience of the readers

4. DISCUSSION

Our experiment indicates that two reference automatic methods (Weszka's and Pun's) were not at all suitable for whisker characterisation. Weszka' method is based on producing a transformed histogram of the image to make the distinction between the peak of the objects and the peak of background easier. The transformation is performed by calculating the rate of change of grey-level (edge value) around each pixel in the image (A.2), and excluding pixels having high edge value. After the histogram transformation, the optimum threshold for the object separation can be determined by selecting the x-axis value, intensity value, belonging to the appropriate peak. The problem with the usage of this method for tin whisker evaluation is that the peak of the background (substrate) and the peak of the objects (whiskers) are heavily overlapping, even in the transformed histogram (Fig. 9), that the x-axis value belonging to the peak in the histogram is not optimal for thresholding; it is not appropriate for separating the objects.

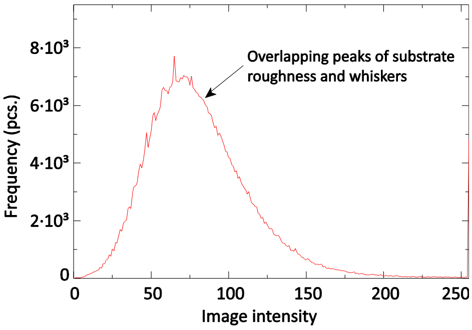

Fig. 9 Transformed histogram of image presented in Fig. 4.a)

Pun's method is based on calculating the a posteriori entropy H_n' of the image and determining an evaluation function $f(s)$ (A.12) from the probability distribution of grey-levels. The optimal threshold value is the s which maximises the evaluation function $f(s)$. The disadvantages of this method according to Kapur [32] are: the maximisation of $f(s)$ does not achieve a priori maximisation of a posteriori entropy H_n'; and since H_n' is majorised by another function, the algorithm does not always use the statistical properties of the grey-level histogram.

The other two reference automatic methods (Otsu's and Kapur's) were appropriate for separating the objects (whiskers) from the background (substrate); but the results, whisker parameters obtained from the binarised images were not correct. Otsu's method searches for the threshold that minimizes the intra-class variance defined as a weighted sum of variances of the two classes (objects and background) computed from the image histogram. However, it has limitations according to Lee [35]. The optimal threshold level cannot be determined by this method if the area with objects (whiskers) is small in comparison to the area of the background (substrate) and if the histogram does not exhibit bimodality. Another case when the optimal threshold cannot be determined exists when an image is corrupted by additive noise, such as (in our case) strong surface roughness of the substrate. Consequently, Otsu's method is not suitable for measuring the whisker density, and had some problems with whisker length measurements also when some contamination covered the whiskers.

Kapur's method performed the best out of the reference automatic methods. This method considers the objects and background as two different signal sources [36], so that when the sum of the two class entropies reaches its maximum, the image is said to be under optimal thresholding. In this method, two probability distributions (e.g., object distribution and background distribution) are derived from the original grey-level distribution of the image as follows (3):

$$\frac{p_0}{P_s},\frac{p_1}{P_s},\ldots,\frac{p_s}{P_s} \text{ and } \frac{p_{s+1}}{1-P_s},\frac{p_{s+2}}{1-P_s},\ldots,\frac{p_l}{1-P_s} \tag{3}$$

where *s* is the threshold and

$$P_s=\sum_{i=0}^{s} p_i \tag{4}$$

$$H_b(s)=-\sum_{i=0}^{s}\frac{p_i}{P_s}\ln\left(\frac{p_i}{P_s}\right) \text{ and } H_w(s)=-\sum_{i=s+1}^{l}\frac{p_i}{1-P_s}\ln\left(\frac{p_i}{1-P_s}\right) \tag{5}$$

The optimal threshold *s* is defined as the grey-level which maximises function (6):

$$f(s)=H_b(s)+H_w(s) \tag{6}$$

As it can be seen from equation (5), the results are also sensitive to the image noise caused by the surface roughness of the substrate; small shining features of the substrate can lower the value *s*, where $H_w(s)$ has its maximum. Moreover, this method cannot correctly cope with the presence of contaminations during whisker length calculations.

5. CONCLUSIONS

In this paper, an automatic method was developed to characterise whisker growth quantitatively in SEM images. The method was tested by characterising evaporated tin layers with Cu substrate because this thin tin layers on this substrate can form whiskers in many forms and in high quantity in a short period of time. The self-developed method was compared to manual counting and to four automatic reference thresholding methods. It was found, that the self-developed method can characterise the whiskers appropriately. Compared to the automatic reference methods, the advantage of this method is that finding the optimal threshold value is not image intensity (histogram) based, but geometry-based. The Mean Intercept Length of objects is calculated to determine it. This method is therefore not sensitive to image noise induced by the surface roughness of the substrate; it is able to appropriately and thoroughly separate anisotropic objects (e.g. whiskers) from the image background and can estimate the area density and the length of whiskers properly. To sum up, the method reported here is recommended to characterise whisker growth automatically and to obtain comparable results between different measurements. By using the method, whisker formation in numerous material systems can easily be investigated, and the reliability of electronics products can significantly be enhanced.

A fent közölt kutatási eredmények publikálásra kerültek:

O. Krammer, B. Illés, R. Bátorfi, K. Dušek, “Automatic characterisation method for statistical evaluation of tin whisker growth”, MICROELECTRONICS RELIABILITY, **73** (2017) 14–21

Készítette: Dr. Krammer Olivér

Budapest, 2018.01.31.