Scanning Confocal Laser Microscopy of the Surface of Anodized Al5052 Alloy

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Introduction

Surface morphology of aluminum alloys has been investigated conventionally by scanning electron microscopy (SEM) and scanning probe microscopy (SPM).[1,2] Quantitative information on the surface morphology can not be obtained by SEM, and the application of SPM is limited to small working range in height less than 1 μm. Scanning confocal laser microscopy (SCLM) gives quantitative information on the surface morphology through tomography in the working height range between 0.1 μm and 500 μm.

In this work, porous-type anodic oxide films were formed on Al5052 alloy in a sulfuric acid solution, and the change in the surface morphology of the alloy during anodizing was studied by SCLM, focussing on the behavior of second-phase particles.

Experimental

A Al5052-H32 alloy sheet (Cu:0.02, Si:0.07, Fe:0.25, Mn:0.01, Mg:2.66, Zn:<0.01, Cr:0.19, Ti:0.02, Al:bal.) was cut into 2 x 3 cm^2 as specimens. The specimens were degreased in ethyl alcohol with ultrasonic wave for 10 min, and then electropolished for 2 min in a mixture of acetic acid and 70% perchloric acid (4 : 1) at 11 ± 1 °C. After electropolishing, the specimens were anodized in 16 wt% sulfuric acid at 20 V and 1 ± 0.5 °C. The specimen surface was observed after anodizing for different periods (t_a) with SCLM and SEM, and electron probe for microanalysis (EPMA) was applied to examine the composition of the second phase particles. The vertical cross section of the specimens was also observed by SCLM to measure the film thickness.

Results and Discussion

Figure 1 shows a) two-dimensional contrast SCLM image and b) SEM image of the specimen before anodizing at the same area. Black and gray spots are observed in the SCLM image, as designated by black and white arrows, respectively. Three-dimensional height SCLM images showed that the black spots are associated with extrusions of second-phase particles, and the gray spots with pores formed by the dissolution of the particles. The height of the particles and the depth of pores were between 0.1 – 5 μm.

The SEM image at the same area shows bright particles and dark particles as indicated by white and black arrows, respectively. The EPMA analysis showed that the bright and dark particles contain mainly iron and magnesium, respectively. Comparison of Fig. 1(a) with (b) suggests that SCLM gives more information on surface morphology than SEM, and that the particles beneath the surface are distinguishable in the SEM image.

Figure 2 illustrates two-dimensional SCLM contrast images of the specimen anodized for t_a = a) 1, b) 10, and c) 30 min at the same area as in Fig. 1. Figure 2-d) shows the image of the t_a = 30 min-specimen coated with a thin Pt layer. The image at t_a = 1 min (Fig.2-a) shows a distribution of spots similar to that before anodizing (Fig.1-a), and shows grain boundary, due the difference in the film thickness from grain to grain. The size and number of the black spots increase with t_a in the SCLM image without Pt coating. The image of t_a = 30 min-specimen with Pt coating, however, is similar to that with t_a = 1, and shows a fringe pattern, suggesting a slight difference in the height from grain to grain again.

This implies that SCLM images obtained from anodized-Al alloy specimen without Pt coating result from not only the outer oxide surface but also inner part of the oxide film. Increase in the number of black spots with t_a can be explained as results of the increase in the number of second-phase particles included in anodic oxide films.

Three-dimensional SCLM height images showed that iron-containing particles are included in the anodic oxide film and magnesium containing particles dissolves preferentially into solution during anodizing. Analysis of three dimensional SCLM images of the specimen partly coated with Pt enabled an accurate determination of the thickness of anodic oxide films formed on Al, assuming the refractive index of anodic oxide films.

References

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2. P. Shmutz and G. S. Frankel, J. Electrochem. Soc. 146, 4461 (1999).

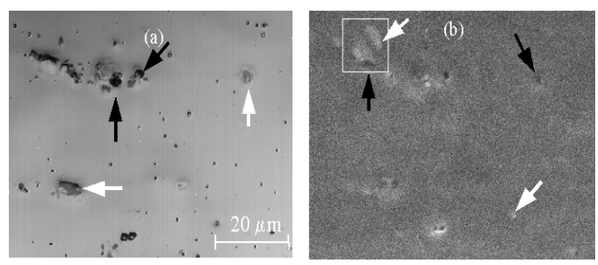

Fig. 1 a) Two-dimensional SCLM contrast image and b) SEM image of the surface of electropolished Al5052 alloy .

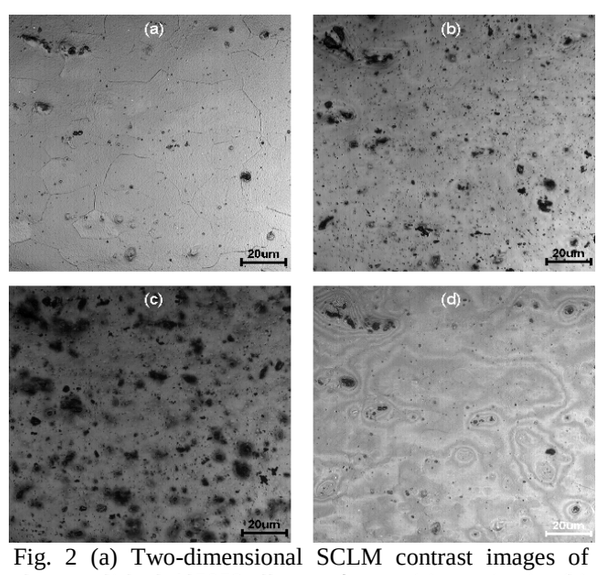

Fig. 2 (a) Two-dimensional SCLM contrast images of electropolished Al5052 alloy surface at (a) t_a = 1 min, (b) t_a = 10 min and (c) t_a = 30 min before Pt coating, and (d) t_a = 30 min after Pt coating.