

Surface effect on GP zone formation in an Al-Ag single-crystal sample examined by GI-SAXS

Hiroshi OKUDA^{1*}, Shojiro OCHIAI¹,
¹IIC,Kyoto University, Sakyo-ku, 606-8501 Japan

Introduction

The kinetics of phase separation is strongly affected by the existence of surface. For example, effect of surface segregation and depletion of quenched-in vacancies at the surface may be quite important to understand the microstructural evolution near the surface. In particular, the latter is interesting also from the viewpoint that it may be related to the formation mechanism of precipitate-free-zone (PFZ) in age-hardenable aluminum alloys. However, experimental methods to inquire into the microstructure near the surface are limited. In particular, nondestructive measurement has been almost impossible.

Small-angle scattering with grazing incidence is a useful tool to inquire into the microstructure with an average over macroscopic volume with limited depth. For example, the penetration depth can be controlled from about 10 nm to several hundreds of nanometers by controlling the angle of incidence of X-ray between 0.2 and 0.5 degree at the photon energy of 8.3keV.

Experimental

Small-angle scattering experiment with grazing incidence was performed at a synchrotron radiation facility, Photon Factory, Tsukuba Japan. Single crystalline Al-1.5mol%Ag samples were solution treated and then quenched into iced water. After electrochemical polishing to accomplish mirror surface finishing, the samples were aged at 423K to form GP zones

The incoming focused X-ray beam with a photon energy of 8.3 keV was shaped in rectangular with 0.1mm height x 1.0mm width at the sample position, and the angle of incidence was controlled by the rotation of the sample holder. The scattering intensity was measured by a one-dimensional position-sensitive detector.

Results and Discussions

Radius of gyration obtained from GI-SAXS measurement is shown as a function of incident angle of X-ray in Fig1. The horizontal line in the figure shows the radius obtained by the transmission small-angle scattering of the same sample, which corresponds to the average over 0.15mm of thickness. When we are interested in the GI-SAXS from a sample whose spatial dispersion of a second phase is nearly uniform in three dimension, one may express the scattering intensity by[1][2],

$$I(q) = T_i(\theta_i) T_f(\theta_f) S(q)$$

with

$$S(q) \sim \exp(-q^2 R_g^2 / 3)$$

Then, when the average size is depth dependent, the intensity might be approximated by

$$I(q, \theta_i, \theta_f) = \int_0^\infty I(q, z) \exp(-\mu z / \sin \theta_i) \exp(-\mu z / \sin \theta_f) dz$$

$$\sim \int_0^\infty \exp(-R_g(z)^2 q^2 / 3) \exp(-\mu z / \sin \theta_i) \exp(-\mu z / \sin(2\theta_G - \theta_i)) dz$$

Figures 1 (a) and (b) show the change in the Guinier radius as a function of grazing angle and the depth profile of average size of Guinier Preston zones. The present result suggests that the average size of GP zones is smaller at the near-surface regions. The present picture agrees with the vacancy depletion model of diffusional phase transformation.

References

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- *okuda@mtl.kyoto-u.ac.jp

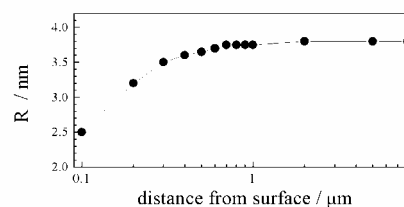
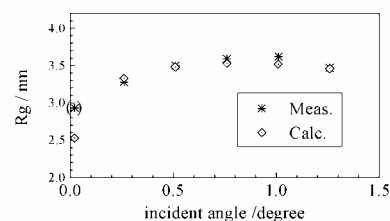


Figure 1. Estimated distribution of average size as a function of distance from the surface. The distribution reproduces the angular dependence of R_g except for the lowest incident angle.