DOI: 10.1017/S1431927605504513
Microsc Microanal 11(Suppl 2), 2005
Copyright 2005 Microscopy Society of America

Analysis of chemical composition of the cube-shaped phase in the Al-Mg-Si alloy by EFTEM and SLEEM

K. Matsuda*, Y. Ishida**, I. Mullerova***, L. Frank***, S. Ikeno*

*Faculty of Engineering, Toyama University, 3190, Gofuku, Toyama, 930-8555, Japan
**Graduate student, Toyama University, 3190, Gofuku, Toyama, 930-8555, Japan
*** Institute of Scientific Instruments ASCR, CZ-61264 Brno, Czech Republic

It is well known that the β-Mg_2Si phase is the equilibrium one in the Al-Mg-Si alloy. Several authors have also reported the cube-shaped phase in the Al-Mg-Si alloy with excess Mg and in quasi-binary alloys and discussed whether it is a precursor of the β-phase or not [1-2]. Application of the energy filtering TEM (EFTEM) to quantifying chemical composition of fine precipitates is very successful [3] and for the case of precipitates in Al-Mg-Si alloys this has been reported in our recent works [4]. Also the scanning low energy electron microscopy (SLEEM) is a useful way to obtaining information about the sample surface [5]. In this study the cube-shaped phase (cube-phase) in Al-Mg-Si with excess Mg was investigated by EFTEM and SLEEM techniques in order to determine the crystal structure and its chemical composition.

In the present experiments, samples of the Al-1.0mass%Mg_2Si-0.4mass%Mg (excess Mg) alloy were solution heat-treated at 848K for 3.6ks and quenched in chilled water at 277K. The aging process was performed at 623K. TEM specimens were prepared by conventional electrolytic polishing method and studied in an EFTEM (JEOL-4010T) equipped with a post-column energy filter and EDS, and operated at 400kV. Elemental maps were obtained by using the three-window method with higher energy loss electrons. SLEEM detector was manufactured in ISI Brno and installed into the SEM (Hitachi S-3500H).

Fig. 1(a) shows the zero-loss image of the cube-phase in an excess Mg alloy aged at 623K for 6ks. It is about 60 nm in size and its sides were found parallel to the [100] and [010] directions of the Al-matrix. Fig. 1(b) and (c) are the Mg-K and Si-K maps. Fig. 1(d) and (e) are the quantitative results calculated from intensities of these elemental maps [3]. The calculated ratio of Mg/Si was 3.09 instead of 2.0 and this result was approximately confirmed by the EDS analysis. Fig. 2(a) shows the SEM image at 20 keV. The precipitates may be identified at low contrast but their shapes cannot be recognized. In Fig. 2(b), acquired in the SLEEM mode at a landing energy of 1.6 keV, the precipitates are not only well observable in their correct shapes, but they can be even sorted according to additional details. The EDS analysis, performed on precipitates marked with 1 to 4, resulted for #1 and #2 in the Mg/Si ratio near 2.0 but for #3 and #4 it grew near to 3. This result supported the EFTEM data and confirmed the SLEEM imaging sensitive to differences in chemical composition of tiny precipitates.

https://doi.org/10.1017/S1431927605504513 Published online by Cambridge University Press

References

[1] H.Westengen and N.Ryum, Z. Metallkde. 70 (1979) 528.
[2] Y.Ohmori, L.C.Doman, Y.Matsuura, S.Kobayashi and K.Nakai, Mater. Trans. 42 (2001) 2567.
[3] R.F.Egerton: Electron Energy-Loss Spectroscopy in Electron Microscopy, 2nd ed., Plenum Press, 1996.
[4] K. Matsuda et al., Scripta Mater., 47 (2002) 833.
[5] I. Mullerova and L. Frank, Advances in Imaging and Electron Physics 128 (2003) 309.

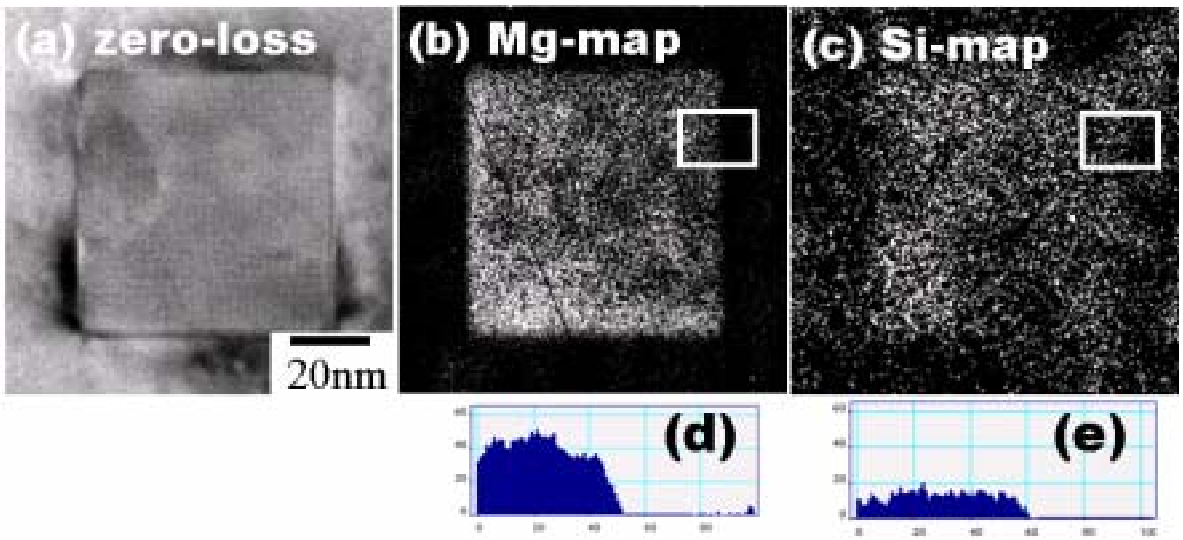

Fig. 1. EFTEM images obtained from the cube-phase in an excess Mg alloy aged at 623K for 6ks: (a) zero-loss image; (b) Mg-K (1305eV) and (c) Si-K (1839eV) maps; (d) and (e) are calculated quantitative profiles from intensities in (b) and (c), respectively, showing the numbers of Mg or Si atoms per unit volume.

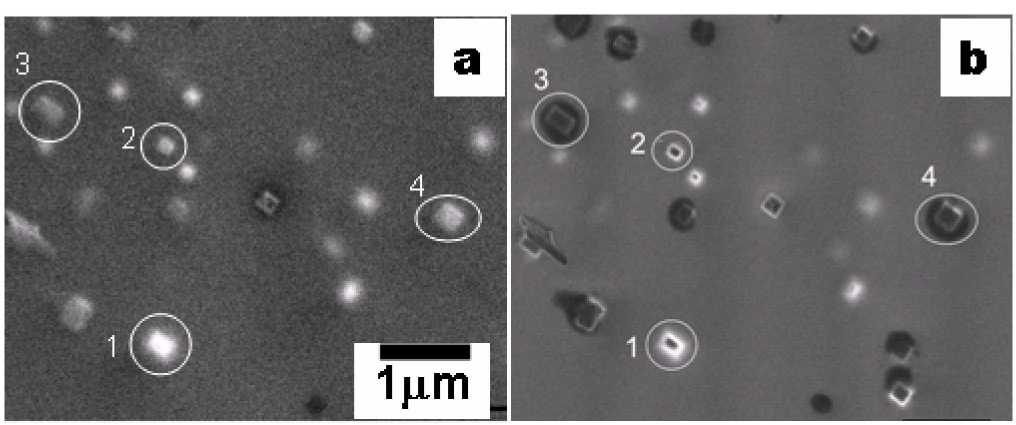

Fig. 2. SLEEM image of the cube-phase in an excess Mg alloy: (a) SEM image (20 keV), and (b) SLEEM image (1.6 keV).

https://doi.org/10.1017/S1431927605504513 Published online by Cambridge University Press