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HRTEM OBSERVATION OF AGE-PRECIPIATION IN Mg-Gd-Y ALLOYS

OBSERWACJA WYDZIELEŃ W STARZONYCH STOPACH Mg-Gd-Y TECHNIKĄ WYSOKOROZDZIELCZEJ TRANSMISYJNEJ MIKROSKOPII ELEKTRONOWEJ

Precipitation in Mg-Gd-Y alloys which have the different total amount of RE were investigated by HRTEM and SAED technique, and calculation of HRTEM images and electron density by first principles to understand the relationship between precipitation in these alloys and HRTEM images. The diffuse scattering by SAED was obtained in as-quenched samples in each alloy, and mono-layer zones have been confirmed by HRTEM observation. The atomic position just consisted of the RE and Mg columns show the black contrast, and the bright dots correspond to the space surrounding by the six Mg columns of hexagonal network with 0.18 nm. This is also corresponded the distribution of electron density of the cluster according to calculation by first principles.

Keywords: Mg-Gd-Y alloy, precipitation, HRTEM

W stopach Mg-Gd-Y o różnej zawartości metali ziem rzadkich badano procesy wydzielenia za pomocą technik HRTEM i SAED oraz symulowania obrazów HRTEM i gęstości elektronów przy użyciu pierwszych zasad w celu zrozumienia korelacji między wydzieleniami w tych stopach i obrazami HRTEM. W przesyconych próbkach we wszystkich stopach widoczne było na obrazach SAED rozpraszanie dyfuzyjne, a monowarstwowe strefy zostały potwierdzone podczas obserwacji HRTEM. Pozytcje atomowe złożone tylko z kolumn atomów ziem rzadkich i Mg dają czarny kontrast, a jasne punkty odpowiadają pozycjom otoczonym przez sześć kolumn Mg o sieci heksagonalnej o odległości 0.18 nm. Odpowiada to również rozkładowi gęstości elektronowej klastra zgodnie z obliczeniami metodą pierwszych zasad.

1. Introduction

Magnesium alloys containing rare earth elements are known to show good heat resistance [1]. Mg-Gd-Y alloys have been expected to improve mechanical properties at the room temperature [1]. The precipitation sequence in Mg-Gd-Y alloys aged at 473 K has been investigated using high resolution transmission electron microscopy (HRTEM) in previous reports. It is described as super-saturated solid solution(S.S.S.S.) \rightarrow β'' (D0₁₉) \rightarrow β' (cbco) \rightarrow β_1 (fcc) \rightarrow β (fcc) [2]. However, the precipitation behavior of the Mg-Gd-Y alloys at early stage of aging after quenching was not understood clearly. In this study, Mg-2.9at%Gd-0.8at%Y and Mg-2.1at%Gd-0.6at%Y alloys including the same ratio of Gd/Y=3 have been investigated to clarify the effect of the Gd/Y ratio and total amount of solute atoms on age-hardening and precipitation using HRTEM and calculations of images and electron density by first principles.

2. Experimental procedure

Mg-2.9at.%Gd-0.8at.%Y (2.9Gd-0.8Y) and Mg-2.1at.%Gd-0.6at.%Y (2.1Gd-0.6Y) alloys were prepared by casting using 99.9%Mg, Gd and Y purity ingots. The obtained alloys were homogenized at 773 K for 43.2 ks, and then hot rolled at 773 K to 1 mm in thickness. The sample was capsulated into the pyrex glass tube with argon gas. Then, it was solution heat-treated at 773 K for 3.6 ks and quenched in hot water at 353 K, and aged in a silicone oil bath at 473 K. TEM specimens were cut from plate samples and thinned by the twin-jet electro polishing technique. HRTEM observations were performed by using a TOPCON EM-002B operated at 120 kV. The HRTEM image was calculated using the multi-slice method and the electron density was calculated using the DV-Xa program [3].

3. Results and discussion

Fig. 1 shows age-hardening curves of two alloys aged at 473K. Two age-hardening curves are parallel to each ot-

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her, and the both alloys appeared two stage hardening which belong to a typical Y-group, and 2.9Gd-0.8Y alloy showed higher hardness than that of 2.1Gd-0.6Y alloy. Fig. 2 shows SAED patterns and HRTEM images in as-quenched specimens. In SAED patterns, the diffuse scattering was obtained in as-quenched samples in each alloy as indicated by an arrowhead, and the row contrasts consisted of bright and dark dots lying on the $\{1100\}_{Mg}$ planes as indicated by black arrows which were considered to mono-layer zones have been confirmed by HRTEM observation. Fig. 3 shows HRTEM images of two alloys aged for 7.2ks. The β' phase ($a=0.64$ nm, $b=2.22$ nm, $c=0.521$ nm) [4] was confirmed in the 2.9Gd-0.8Y alloy by HRTEM, although the β'' phase ($a=0.64$ nm, $c=0.521$ nm) [4] was confirmed in the 2.1Gd-0.6Y alloy before the β' phase. Fig. 4 shows simulated images of a RE column in Mg-matrix. The atomic position just consisted of the RE and Mg atomic columns show the black contrast, and the bright dots correspond to the space surrounding by the six Mg columns of hexagonal network with 0.18 nm.

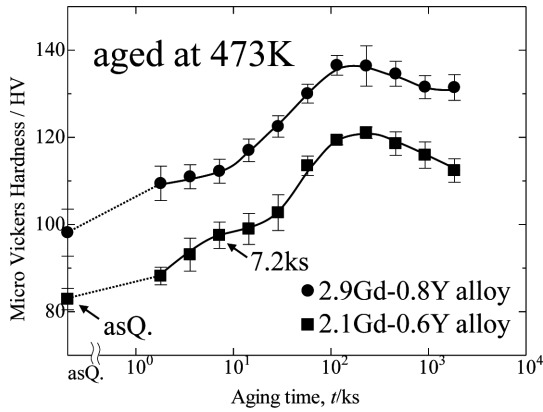


Fig. 1. Age-hardening curves of 2.9Gd-0.8Y alloy and 2.1Gd-0.6Y alloy aged at 473K

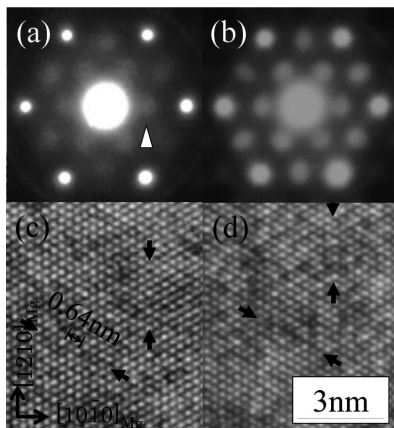


Fig. 2. SAED patterns and HRTEM images of as-quenched specimen in (a,c) 2.1Gd-0.6Y alloy and (b,d) 2.9Gd-0.8Y alloy

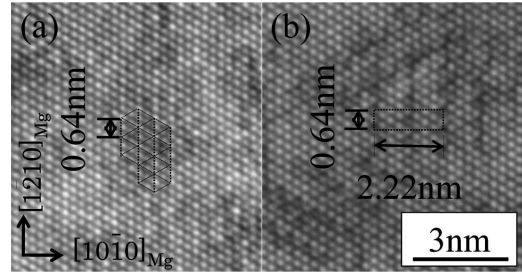


Fig. 3. HRTEM images of (a) 2.1Gd-0.6Y alloy and (b) 2.9Gd-0.8Y alloy aged at 473K for 7.2ks

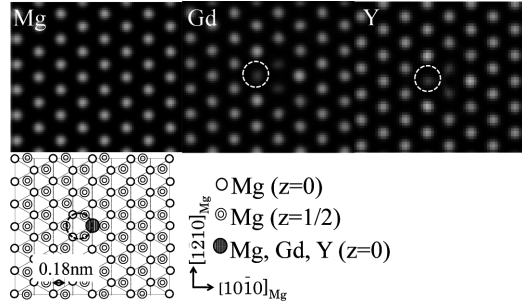


Fig. 4. Simulated HRTEM images of a RE column in Mg-matrix

4. Conclusions

1. The hardness of two alloys increased with two stages, and this behavior is typical in Mg-RE alloys including Y sub-group element.
2. The diffuse scattering was obtained in as-quenched samples in each alloy by SAED and the mono-layer zones have been confirmed by HRTEM observation.
3. The β' phase was confirmed in the 2.9Gd-0.8Y alloy by HRTEM, although the β'' phase was confirmed in the 2.1Gd-0.6Y alloy before the β' phase.
4. In simulated images, the space surrounded by the RE and Mg columns show the dark dots, and the bright dots correspond to the space surrounded by only Mg columns.

REFERENCES

- [1] S. Kamado, Y. Kojima, S. Taniike, I. Seki, S. Hama, Proc. of the International Conf. on Magnesium Alloys and Their Applications, 169-174 (1998).
- [2] S.M. He, X.Q. Zheng, L.M. Peng, X. Gao, J.F. Nie, W.J. Ding, J. Alloys and Compound **471**, 309-313 (2006).
- [3] H. Adachi, Y. Owada, I. Tanaka, H. Nakamatsu, M. Mizuno, Sankyo Shuppan, Introduction of DV-Xa Method, 3rd ed., Tokyo 2001.
- [4] H. Karimzadeh, Ph.D. Thesis, University of Manchester, UK, 97-100 (1985).