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ScienceDirect

Scripta Materialia 75 (2014) 66–69

Scripta MATERIALIA

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In situ measurements on stability of long-period stacking-ordered structures in $Mg_{85}Y_9Zn_6$ alloys during heating examined by multicolor synchrotron radiation small-angle scattering

Hiroshi Okuda,[a,*] Toshiki Horiuchi,[a] Michiaki Yamasaki,[b] Yoshihito Kawamura[b] and Shinji Kohara[c]

[a] *Department of Materials Science and Engineering, Kyoto University, Sakyo-ku, Kyoto 606-8501, Japan*
[b] *Department of Materials Science and Engineering, Kumamoto University, Kurokami, Kumamoto 860-8555, Japan*
[c] *Japan Synchrotron Radiation Research Institute, Kohto, Sayo 865-8555, Japan*

Received 26 September 2013; accepted 19 November 2013
Available online 23 November 2013

Changes in long-period stacking-ordered (LPSO) structures in $Mg_{85}Y_9Zn_6$ samples during heating up to melting has been examined by in situ synchrotron radiation small-angle scattering with X-rays containing higher harmonics to simultaneously obtain wide-angle diffraction spots. The 10H structure was less stable than the 18R, and the collapse of 18R and melting occurred simultaneously. The lattice parameter for in-plane ordering changes differently from the others with temperature, suggesting that the average structure of the in-plane order changes during heating.

Keywords: Small-angle X-ray scattering (SAXS); Synchrotron radiation; Magnesium alloys; Ordering; Long-period stacking-ordered (LPSO) structure

Owing to their potential application for lightweight structure materials, unique crystallographic structures and mechanical properties, long-period stacking-ordered (LPSO) structures in Mg–rare earth–transition metal alloy systems have recently attracted great interest as Mg-based lightweight alloys [1–4]. The Mg–Y–Zn ternary alloy system is known as a typical alloy system giving LPSO structures, and is classified as a type-I LPSO; in this system, LPSO structures are found to form just after casting, without any annealing. To understand the formation mechanism and the stability of LPSO structures, two approaches are important. One is a kinetic approach to the formation of the structure, i.e. how the stacking fault, solute segregation, periodicity formation and in-plane ordering are related each other. The other approach is static stability, namely, the phase diagram and detailed stable structure studies. Intensive transmission electron microscopy studies [3,5–8] have revealed the most stable periodicity for given compositions and temperatures – for example, 18R structures with in-plane ordering for the present alloy composition – which, in turn, requires further examination of the reported phase diagram [9–12]. Examining the nanostructural changes occurring during heating at a constant rate, such as the calorimetric scan often used in phase diagram studies [10], is a convenient approach to understanding what is happening during the process. Such examination might be pursued only by in situ measurements with the temperature range covering up to the melting point of the materials. It is also emphasized that in situ measurement is particularly important in the present alloy, where the LPSO phase is reported to appear directly from the liquid phase. In the present measurements, order spots from LPSO structures, in-plane ordering and fundamental Bragg spots were recorded up to well above the expected liquidus line by small-angle scattering/diffraction with higher harmonics.

An $Mg_{85}Y_9Zn_6$ polycrystalline alloy ingot was prepared by casting and provided by Kumamoto University. The composition is reported as an LPSO

* Corresponding author. Tel.: +81 757535193; fax: +81 757534835; e-mail: okuda@materials.mbox.media.kyoto-u.ac.jp

1359-6462/$ - see front matter © 2013 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.scriptamat.2013.11.021

single-phase composition after long-time annealing [8]. The samples for in situ measurements were cut from the ingot as a plate with a thickness of about 0.5 mm. The in situ sample chamber was evacuated and the samples, shielded with carbon windows, were heated up to 650 °C during the measurements after rotating the sample to the desired orientation for the initial microstructure. Preliminary microstructural analysis was undertaken at beamline 6A of Photon Factory, KEK Tsukuba, Japan. In situ measurements were made at beamline 04B2 of SPring8, with a photon energy of 37.8 keV. With a single (111) Si monochromating crystal, the incident beam contained relatively strong λ/3 and λ/4 higher harmonics. A two-dimensional image-intensifier charge-coupled device (II-CCD) detector was placed in a standard small-angle X-ray scattering (SAXS) geometry designed to cover the scattering vector up to 10 nm^{-1}, which contains the LPSO peaks for 18R and 10H up to the second order, and the first in-plane order spots. Figure 1 illustrates the experimental set-up of the present measurements. A furnace was placed on a rotation stage inside a vacuum chamber evacuated by a turbo molecular pump, and an II-CCD was placed downstream of the vacuum path/beam stop of the SAXS apparatus. The scattering angle was calibrated by silver behenate for SAXS and Si powder for higher harmonics. With the higher harmonics, the same area of the SAXS detector covers up to 40 nm^{-1}. Therefore, we can monitor the fundamental Bragg reflections overlapping the standard small-angle scattering/diffraction pattern obtained for LPSO [13]. This overlapping measurement utilizing higher harmonics is a convenient way to monitor the melting/solidification of the sample, since the small-angle diffraction patterns up to 10 nm^{-1} only tell us about the formation/dissolution of the LPSO structure, and measuring the diffraction at a large angle up to 40 nm^{-1} with the same wavelength as for the SAXS requires too large an exit window of the furnace to control the temperature and evaporation from the sample. This is an important way to confirm if the LPSO appears simultaneously with the solidification, and also if the in-plane ordering occurs simultaneously with the formation of the LPSO.

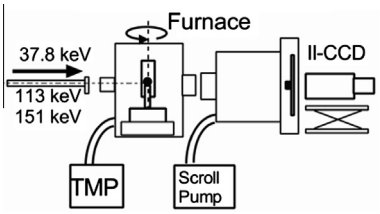

Figure 1. Schematic illustrations of the present in situ measurements.

In the present work, the heating rate of the sample was fixed as 10 K min^{-1}. The intensity was recorded every 15 s, with 13 s of exposure. It captured the structural change for every 2.5 K during heating. The temperature was monitored in the copper block of the sample holder in the furnace.

Figure 2 shows the radially averaged profiles for the in situ measurement. As shown in the figure, the profile gives the first diffraction peaks of 18R and 10H LPSO around 4 and 4.8 nm^{-1}, respectively, and a broad peak corresponding to the in-plane ordering around 6 nm^{-1}. The peak corresponding to the in-plane ordering is much broader than the LPSO peaks of 18R and 10H owing to the smaller in-plane domain size. These peaks agreed well with the results of previous studies [13,14]. At a low q, below 2 nm^{-1}, peak patterns appear because of the higher harmonics of λ/3, and correspond to the

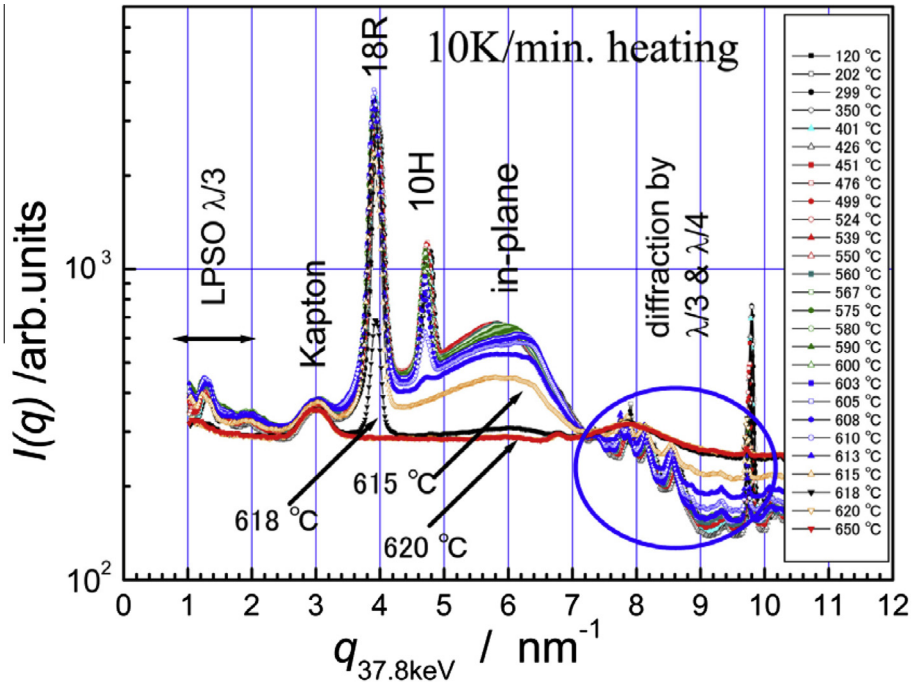

Figure 2. Radially averaged intensity profiles obtained during heating.