Evolution of α phase in metastable β titanium alloys studied by small-angle X-ray scattering

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Abstract

Small-angle X-ray scattering (SAXS) is a technique which makes use of elastic scattering of X-ray radiation on inhomogeneities in electron density in the studied material. In particular, a difference in chemical compositions between individual phases can be detected. In this research, SAXS was used to study the evolution of α particles in aged samples of a metastable β titanium alloy, Ti-6.8Mo-4.5Fe-1.5Al (LCB). In order to obtain scattering patterns for a known crystallographic orientation, the experiments were carried out on single crystals grown by a floating zone technique. Aged single-crystalline samples were measured in three different orientations, namely (001), (110) and (111) planes of the bcc β matrix oriented perpendicularly to the primary beam. Resulting scattering patterns exhibited symmetries which correlated with the orientation of the studied sample. A simple theoretical model was developed to interpret the shape and orientation of the observed scattering streaks. Good qualitative agreement between experimental data and simulation was found and the first results of the model are presented in this paper.

1. Introduction

Titanium alloys are attractive materials for a number of applications due to their low density, high strength and corrosion resistance [1]. Pure titanium is an allotropic element which crystallizes in a hexagonal close-packed α phase at low temperatures, while above the β-transus (882 °C for pure Ti), its structure changes to a body-centred cubic (bcc) β phase [2]. Additions of alloying elements which tend to stabilize the high-temperature β phase to lower temperatures (e.g. Mo, V, Fe, Nb) can suppress the martensitic β → α transformation below room temperature. Alloys with such composition are known as metastable β titanium alloys and upon quenching from temperatures in the β phase field, they retain the bcc β phase in a thermodynamically metastable state [3]. The metastable β phase can undergo a number of phase transformations which can be utilized to tailor the microstructure and mechanical properties of the material to a specific application. In some metastable β titanium alloys, nanometre-sized particles of metastable ω phase form during quenching. The ω phase has a hexagonal structure and is created by a diffusionless process accompanied by a collapse of two $(111)_\beta$ planes into their intermediate position, while the neighbouring $(111)_\beta$ plane remains at the same position [4]. Nevertheless, the collapse might not be complete, producing a trigonal crystallographic structure [5, 6]. During ageing at relatively low temperatures (approx. 200 – 450 °C, depending on the particular alloy composition), the ω phase grows by a diffusion-assisted process [7]. Ageing at higher temperatures results in precipitation of the thermodynamically stable α phase. α particles exhibit typically a lamellar shape and they obey the so-called Burgers orientation relationship with the parent β phase [1]: $(0001)_\alpha \,||\, \{110\}_\beta$, $\langle 11\text{-}20\rangle_\alpha \,||\, \langle 1\text{-}11\rangle_\beta$. There are twelve crystallographically equivalent α variants within a parent β grain allowed by the Burgers orientation relationship. The α phase can nucleate at inhomogeneities in the interior of β grains, e.g. particles of the ω phase or chemical inhomogeneities in the β phase. A dense distribution of nucleation sites results in a fine dispersion of small α lamellae. This morphology has a positive impact on the strength and toughness of the material, maintaining a satisfactory ductility [8].

In this paper, we present results of a small-angle X-ray scattering (SAXS) experiment which focused on characterization of α phase particles and on investigation of their morphology with respect to different heat treatment conditions. SAXS is a non-destructive technique which provides information on inhomogeneities in electron density in the studied sample (in our case, a difference in the chemical compositions of α phase particles and the β matrix). From SAXS data, structure characteristics of the inhomogeneities, such as the size and shape, can be determined [9, 10]. Our previous SAXS studies focused on the investigation of ω phase and showed that ω particles in aged titanium alloys are spatially ordered in a cubic superlattice with the main axes parallel to $\langle 100\rangle_\beta$ directions [11]. The growth kinetics of ω particles was investigated in an in situ experiment in [12, 13]. Evolution of ω particles in other metastable β titanium alloys was also investigated by SAXS in [14, 15, 16]. SAXS was also employed to characterize structure changes in magnesium [17] and aluminium alloys [18] or Ni-based superalloys [19].

2. Material and experiments

In this study, single-crystalline samples of a metastable β titanium alloy, Ti-6.8Mo-4.5Fe-1.5Al (LCB), were investigated. Single crystals were grown in an optical floating zone furnace FZ-T-4000-VI-VPM-PC, Crystal Systems Corp., equipped with four halogen 1000 W lamps. The details of the growth procedure and characterization of the resulting ingot can be found in [20]. The orientation of the obtained single crystal was arbitrary and it was determined by Laue backscatter diffraction to be approximately $[131]_\beta$. The single-crystalline ingots were solution treated at 860 °C for 4 h in an evacuated quartz tube and quenched in water. Sample slices were cut perpendicularly to the growth direction of the solution-treated ingot and aged in molten salt baths for 0.5 h and 16 h at the temperatures of 460 °C, 490 °C, 510 °C and 540 °C, which promoted the growth of α phase lamellae. Each ageing treatment was terminated by a water quench. The samples were polished from both sides using SiC emery papers to the thickness of approximately 200 μm. The final polishing was carried out using a vibratory polisher.

Our experiment was carried out at the Advanced Photon Source (APS; Argonne National Laboratory, USA) at a dedicated ultra-small-angle X-ray scattering (USAXS) facility at 15-ID beamline (now the instrument is moved to 9-ID) [21]. The primary beam energy was 25 keV and the beam size at the sample surface was 150×150 μm^2. The radiation scattered by the sample was detected by a two-dimensional CCD detector Mar165 with 2048×2048 pixels and the pixel size of 79 μm. The sample-detector distance was 3.73 m. The measured samples (of an arbitrary, but known orientation) were mounted on a goniometer, allowing to measure each sample in three orientations: $(001)_\beta$, $(110)_\beta$ and $(111)_\beta$ planes of the bcc β matrix perpendicular to the incident beam. The acquisition time of a single detector frame was 1 – 30 s, depending on the particular sample and its orientation.

All measured data were calibrated to absolute scale using a glassy carbon standard and corrected for acquisition time, primary beam intensity, effective sample thickness (i.e. the tilt of the sample with respect to the beam was taken into account) and background scattering [22, 23]. The scattering angle was calibrated using a silver behenate standard [24].

3. Results

Measured scattering patterns exhibited characteristic streaks arising from the lamellar shape of α particles, see Fig. 1, which shows scattering patters arising from the sample aged at 510 °C for 0.5 h. Furthermore, the patterns exhibited different symmetries depending on the orientation of the sample with respect to the incident beam. When the $(001)_\beta$ plane of the cubic β matrix was oriented perpendicular to the primary beam, a four-fold symmetry was observed, while for the $(110)_\beta$ and $(111)_\beta$ orientations, two- and six-fold symmetries were detected. This is due to a specific spatial orientation of α phase lamellae with respect to the parent β matrix.

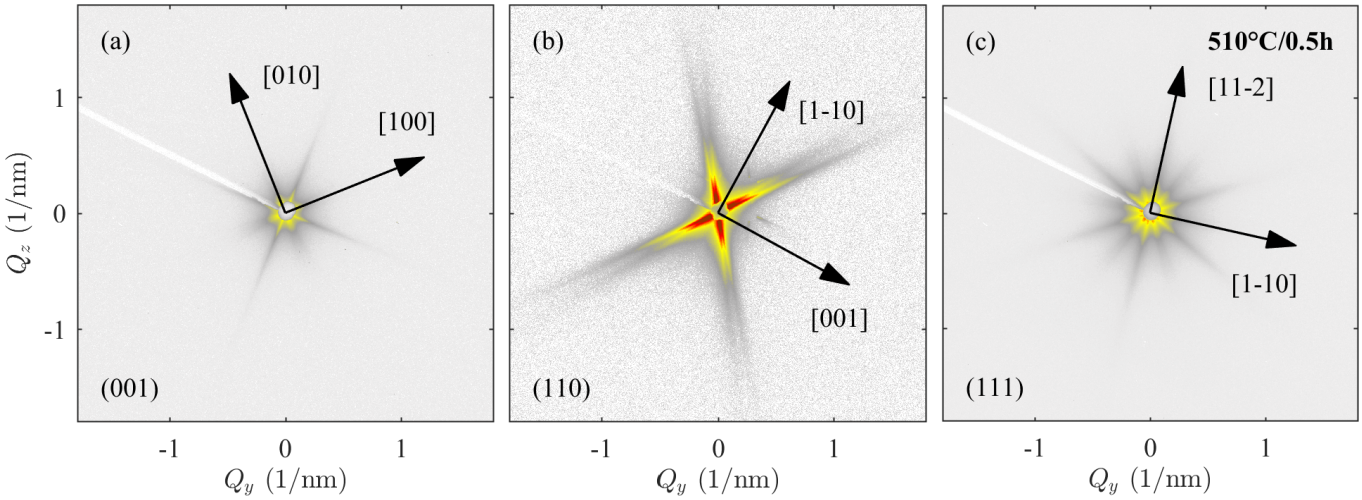

Fig. 1 An example of measured SAXS patterns from the sample aged at 510 °C for 0.5 h in three different orientations of the bcc β matrix with respect to the incident beam; the β crystallographic plane perpendicular to the beam is indicated in the bottom left corner. The colour scale is logarithmic and spans over five decades.

The evolution of scattering patterns with ageing time is demonstrated in Figs. 2 and 3 which show images obtained from samples aged at 490 °C for 0.5 h and 16 h, respectively. After the short ageing time at 490 °C (Fig. 2), the streaks are already well visible but quite weak, indicating a low volume fraction of α lamellae. After 16 h at 490 °C (Fig. 3), the character of the streaks remains similar but their intensity significantly increases. Scattering pattern of a sample aged at a higher temperature (510 °C, ageing time 0.5 h) is shown in Fig. 1.

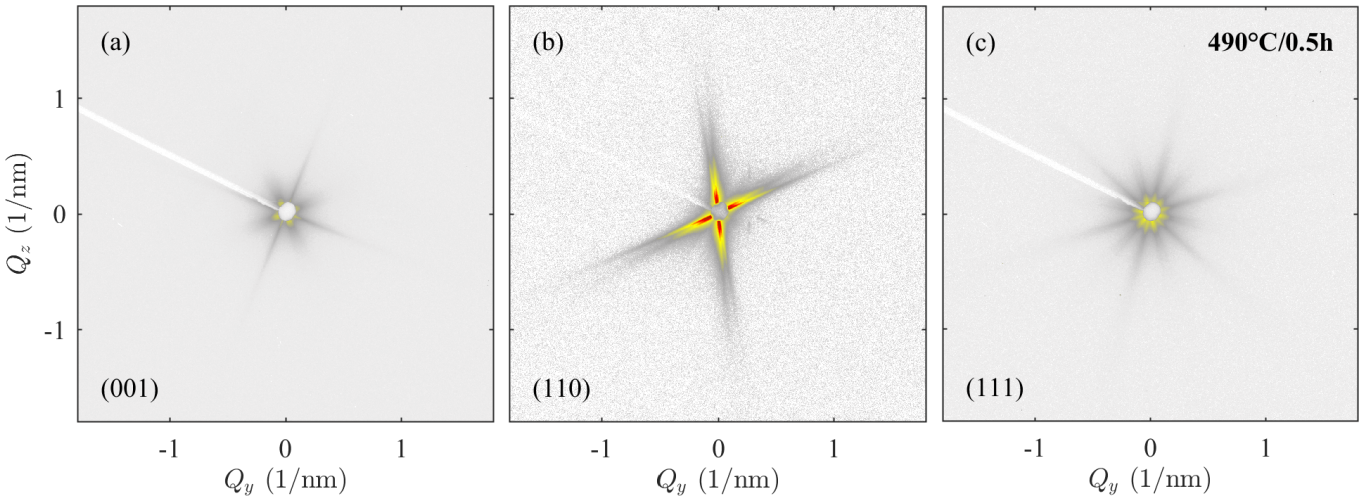

Fig. 2 SAXS patterns from the sample aged at 490 °C for 0.5 h for three measured orientations of the sample with respect to the beam. The colour scale is the same as in Fig.1.

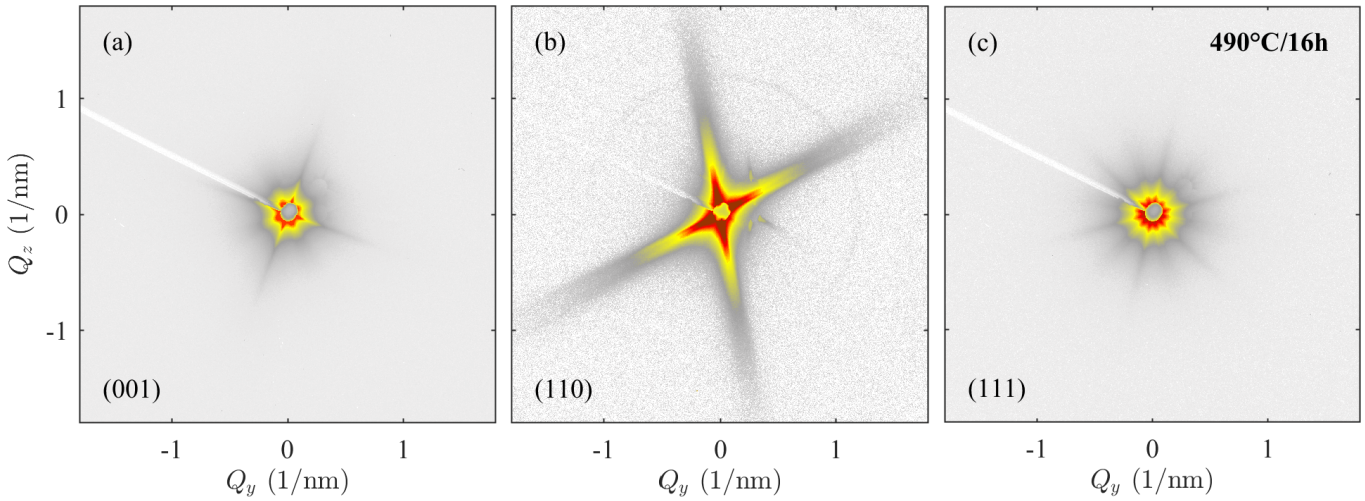

Fig 3 SAXS patterns from the sample aged at 490 °C for 16 h for three measured orientations of the sample with respect to the beam. The colour scale is the same as in Fig.1.

4. Discussion

In order to explain the shape of measured streaks and their symmetry with respect to the orientation of the sample, a simple numerical model was developed. In the model, α phase lamellae were simulated as triaxial ellipsoids, the lengths of individual axes being the parameters of the simulation. We assumed that the ellipsoids have random sizes; however, the ratios of their axes were kept fixed. Other parameters of the simulation included the order of Gamma distribution of the axes lengths m_R, the rotation of the pattern in the detector plane and a background correction. The simulation evaluated all three orientations simultaneously. The relative root-mean square deviation of the axis length is $\sigma_R/R = m_R^{-1/2}$; since the simulated intensities were not much sensitive to m_R, we kept $m_R = 10$ fixed, i.e. $\sigma_R/R \approx 0.32$. In the simulations we completely neglected possible correlations in the ellipsoid positions, represented by the position correlation function. Such function would modify the SAXS intensity distribution for small Q, not affecting the characteristic streaks.

The best agreement between the simulated and measured symmetries was achieved for α lamellae having the longest dimension along $[0001]_\alpha \,||\, \langle 110\rangle_\beta$, the shortest dimension along $\langle 11\text{-}20\rangle_\alpha \,||\, \langle 1\text{-}11\rangle_\beta$ and the remaining one along $\langle 1\text{-}100\rangle_\alpha \,||\, \langle 1\text{-}1\text{-}2\rangle_\beta$. An example of simulation results for the sample aged at 510 °C for 0.5 h (cf. Fig. 1 for experimental data) is shown in Fig. 4. The simulation was obtained for triaxial ellipsoids with the axis ratio of approximately 1 : 4 : 14. The determination of the absolute ellipsoid dimensions is complicated by the fact that due to a broad dispersion of ellipsoid sizes, the measured SAXS data do not exhibit a distinct transition between the Guinier and Porod regions [25], which is usually used for the determination of the mean particle size. In this case, the absolute dimension could be determined from the SAXS intensities carefully normalized to the primary X-ray beam and irradiated sample volume. This will be the subject of future studies.

The comparison of measured and simulated data is displayed in Fig. 5, which shows radial cuts (or more precisely data integrated along circular sectors with the central angle of 4°) in the directions of the dashed lines in Fig. 4 for measured (dots) and simulated (full lines) data. It can be observed that despite the model being quite simple, we were able to obtain a good qualitative agreement with measured data.

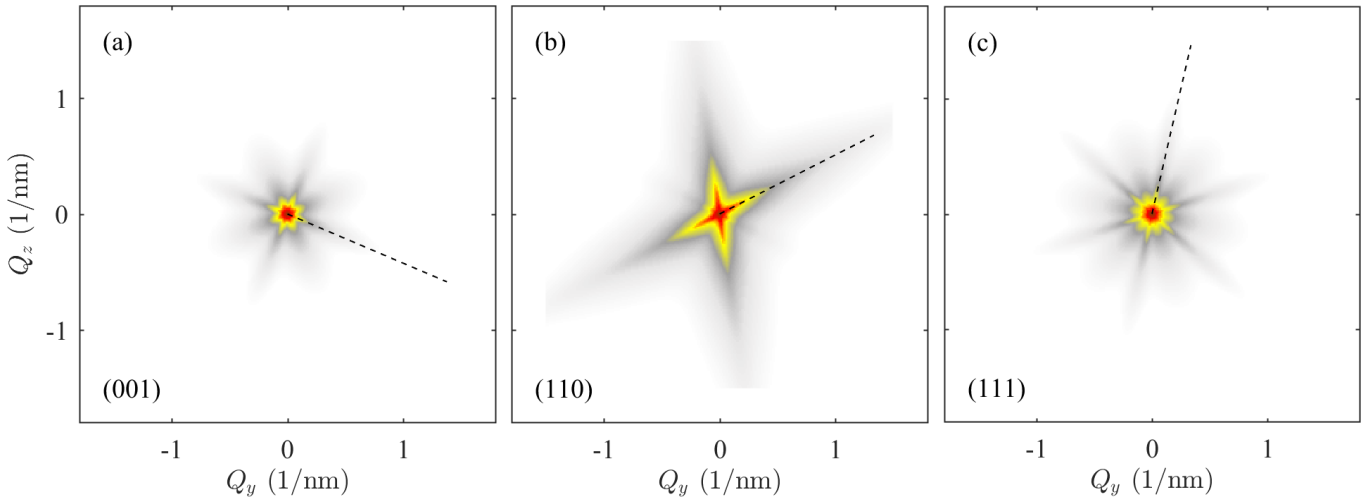

Fig. 4 Simulation results for sample aged at 510 °C for 0.5 h, cf. experimental data in Fig. 1. The intensity is logarithmic. Dashed lines denote the directions along which the radial cuts in Fig. 5 were extracted.

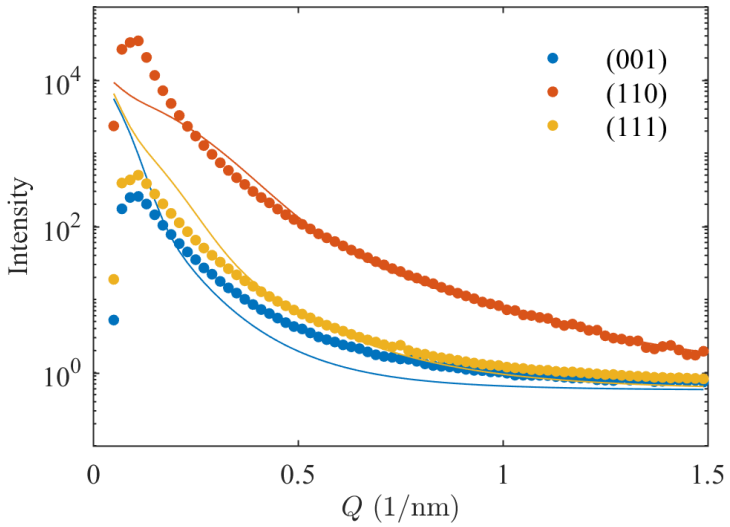

Fig. 5 Radial cuts along dashed lines in Fig. 4 comparing measured data (dots) and the simulation (full lines). Blue, red and yellow colours indicate the (001), (110) and (111) sample orientations, respectively.

5. Conclusions

Small-angle X-ray scattering (SAXS) was employed to study α phase particles in a metastable β titanium alloy, LCB (Ti-6.8Mo-4.5Fe-1.5Al). Characteristic streaks arising from the shape and spatial orientation of α particles in the β matrix were observed, the symmetry of the streaks was found to correlate with the orientation of the sample with respect to the incident beam. A simple simulation modelling α particles as triaxial ellipsoids was introduced. This model is able to qualitatively explain the observed streaks. It was found that the longest α lath dimension lies along $[0001]_\alpha$ || $\langle 110\rangle_\beta$ direction, while the shortest dimension is parallel to $\langle 11\text{-}20\rangle_\alpha$ || $\langle 1\text{-}11\rangle_\beta$. The remaining dimension lies along $\langle 1\text{-}100\rangle_\alpha$ || $\langle 1\text{-}1\text{-}2\rangle_\beta$.

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