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Formation of multiple orientation relationships of Q precipitates in Al–Mg–Si–Cu alloys

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Precipitation of Q laths/rods in some age-hardenable aluminium alloys has been traditionally accepted to occur with a single orientation relationship with the α-Al matrix. In this study, the orientation relationships of Q precipitates are characterized by atomic resolution high-angle annular dark-field scanning transmission electron microscopy. Contrary to the long-standing view, multiple orientation relationships are found between Q precipitates and the α-Al matrix. These orientations are correlated by rigid-body rotations about the long axes of the Q laths/rods.

Keywords: Aluminium alloys; Precipitates; Orientation relationship; HAADF-STEM

The Q phase is found in a wide variety of aluminium alloys, including 2xxx series Al–Cu–Mg–Si alloys [1], 6xxx series Al–Mg–Si wrought alloys with Cu additions [2–8], and Al–Si–Mg–Cu casting alloys such as A319 [9,10]. Solid-state precipitates of Q phase form as long laths or rods with their long axes parallel to $<0\,0\,1>_\alpha$. The crystal structure of the Q phase is generally accepted to be hexagonal ($P\bar{6}$ $a = 1.039$ nm, $c = 0.402$ nm), with 21 atoms in the unit cell [11], but the reported stoichiometric composition varies from $Al_4Cu_2Mg_8Si_7$, $Al_5Cu_2Mg_8Si_6$ to $Al_4Cu_1Mg_5Si_4$ [12]. In the literature, the Q phase is often quoted as Q', which is regarded as a precursor phase of Q. The crystal structure of the Q' phase is similar to that of Q and the Q' differs from the Q phase only by a small change in lattice parameters [12,13] or composition [5]. The relationship between Q' and Q is analogous to that of S' and S phases reported in Al–Cu–Mg alloys [14] and in this paper no attempt is made to distinguish Q' and Q. It is commonly known in the literature [15] that the Q phase has a $\{5\,1\,0\}_\alpha$ habit plane, and that its orientation relationship (OR) with the α-Al matrix is $[0\,0\,0\,1]_Q$ // $[0\,0\,1]_\alpha$ and $(1\,1\bar{2}\,0)_Q$ // $(5\,1\,0)_\alpha$, or $[0\,0\,0\,1]_Q$ // $[0\,0\,1]_\alpha$ and $(2\,1\bar{3}\,0)_Q$ // $(1\,0\,0)_\alpha$.

While the Q phase is a key strengthening phase in a group of commercially important aluminium alloys, its formation mechanism is still poorly understood. Any attempts to gaining improved understanding of the formation mechanism inevitably requires a knowledge of the ORs between the Q phase and its surrounding α-Al matrix and hence the transformation strains involved in its precipitation inside α-Al. Given the technological importance of the Q precipitates in Al–Mg–Si(–Cu) and Al–Cu–Mg–Si alloys, a clear understanding of the ORs of the Q phase is essential. In this study an atomic-resolution high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) imaging technique is used to examine the ORs of Q precipitates formed in aged samples of an A6111 (Al–Mg–Si–Cu) alloy. Contrary to the single OR that has been reported in the literature [12,15], we found multiple ORs between Q precipitates and α-Al matrix.

Samples of AA6111 alloy with a nominal composition of Al–0.75 Mg–0.63Si–0.75Cu–0.25Fe–0.2Mn (wt.%) were solution treated at 550 °C for 30 min, water quenched and aged at 250 °C for 16 days. Discs 3 mm

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in diameter were punched from the aged sheets, ground to a thickness of ~0.2 mm, and twin-jet electropolished in a solution of 33% nitric acid and 67% methanol, at −25 °C and 14 V. HAADF-STEM imaging was performed using a dual-aberration-corrected FEI Titan^3 80–300 microscope operated at 300 kV, under strong atomic number (Z) contrast conditions and with an incident electron probe size of ~1 Å, a convergence semi-angle of 15 mrad and an inner collection semi-angle of ~50 mrad. The enlarged HAADF-STEM images were the result of Fourier filtering with an aperture encompassing all the visible spots in the Fourier transform, in order to remove high-frequency noise.

All Q precipitates in the aged AA6111 alloy had a lath- or rod-like shape with their long axes parallel to directions of $[0001]_Q$ and $<001>_\alpha$, in which there is a negligible mismatch between atomic rows of Q and α-Al lattices. Inspection of cross-sections of "end-on" Q precipitates revealed that some laths or rods had a habit plane or facet parallel to $\{510\}_\alpha$ while others had their facets deviated from $\{510\}_\alpha$ or had no obvious facets. Figure 1a–c show a $[0001]_Q$ HAADF-STEM image and an enlarged inverse fast Fourier transform (FFT) image of an end-on $\{510\}_\alpha$ lath and the corresponding FFT pattern. In the HAADF-STEM image, Figure 1a, individual atomic columns can be clearly resolved and those containing predominantly Cu atoms can be identified as the much brighter dots. This is because Cu has a much higher atomic number ($Z = 29$) than the other three elements ($Z = 14$ for Si, 13 for Al, and 12 for Mg) and the atomic density in each column is approximately the same for this crystal orientation. A hexagonal honeycomb network of Cu columns (red) was observed and marked in the enlarged inverse FFT image (Fig. 1b). For the hexagonal unit cell of the Q phase outlined (red diamonds) in the figure, each of the three $<11\bar{2}0>_Q$ directions had an angle of ~11.5°, ~18.5° or ~41.5° with respect to its nearest $<100>_\alpha$ direction. The FFT pattern in Figure 1c could be indexed according to the $[0001]_Q$ zone axis of the Q structure ($a = 1.04$ nm, $c = 0.405$ nm). The interface between Q and α-Al phases was parallel to a $\{510\}_\alpha$ plane, and the OR implied by the FFT pattern was such that:

$$[0001]_Q // [001]_\alpha \text{ and } <11\bar{2}0>_Q // <510>_\alpha. \qquad \text{(OR1)}$$

This OR has been commonly reported in the literature [12,15]. In this OR, the lattice misfit between the $<11\bar{2}0>_Q$ and $<510>_\alpha$ directions was very small (~0.7%) and Q laths having a $\{510\}_\alpha$ habit plane would maximize the number of matched atomic rows in the habit plane.

Closer examination of Figure 1a, and Figure 1b in particular, also revealed the existence of another hexagonal atomic network (blue). The angles of the triangles within this hexagonal network were measured to be ~60°. The atomic columns that constitute the hexagonal network appeared to be slightly brighter than the other atomic columns except the Cu columns, suggesting that these columns are likely to be Si, as the atomic number of Si is slightly higher than that of Al or Mg, or the atomic density of these columns are higher. Using the α-Al lattice

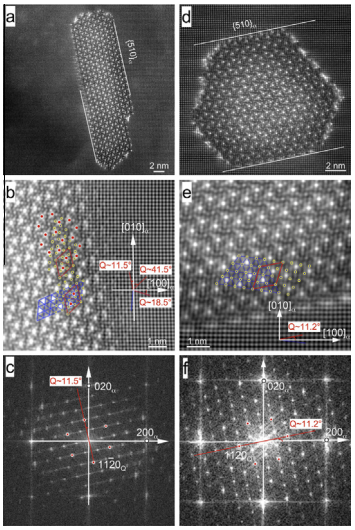

Figure 1. (a, d) $<001>_\alpha$ HAADF-STEM images of cross-sections of Q laths/rods; (b, e) enlarged inverse FFT images of (a) and (d), respectively; and (c, f) corresponding FFT patterns of (a) and (d), respectively. In (b) and (e) the red and yellow dots mark the location of the Cu and presumably Si-rich columns, respectively, and the Q unit cell is indicated by red lines, and Si network by blue lines. In (c) and (f) the red dots mark the $\{11\bar{2}0\}_Q$ reflections. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

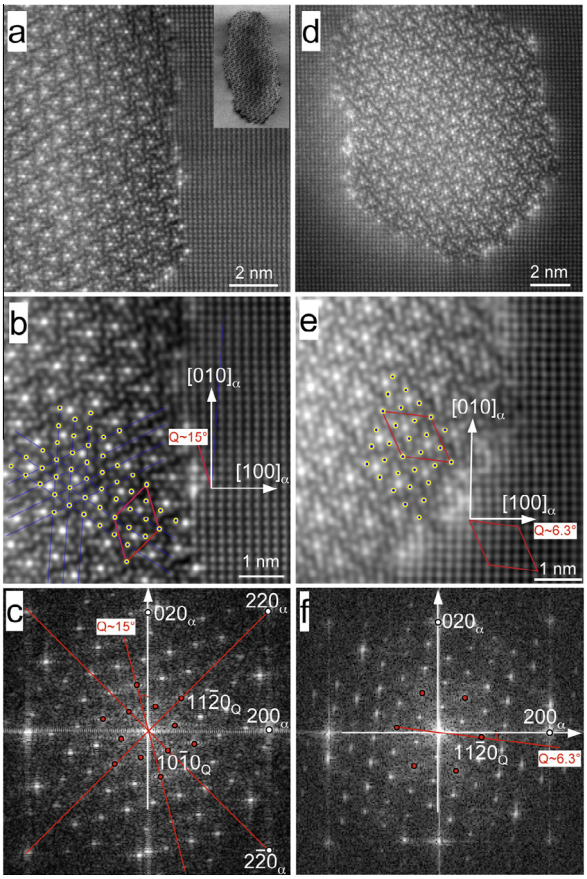

Figure 2. (a, d) $<001>_\alpha$ HAADF-STEM images of cross-sections of Q laths/rods; (b, e) enlarged inverse FFT images of (a) and (d), respectively; and (c, f) corresponding FFT patterns of (a) and (d), respectively. In (b) and (e) the yellow dots mark the location of the presumably Si-rich columns, and the Q unit cell is indicated by red lines. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)