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On the analysis of diffuse intensity and striation contrast in electron microscopy images of Al–Cu–Co–Si decagonal phases

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Abstract

Electron microscopy and diffraction experiments were carried out to analyze diffraction features and correlate with the striation contrast observed in the images of Al–Cu–Co–Si decagonal phase. There are two important diffraction features identified which include the diffuse streak and the extra satellite spots. The extra satellite spots can indicate some sort of superlattice chemical ordering among the clusters while the diffuse streaks indicate presence of some kind of phason disorder leading to the observation of striation contrast in the imaging modes. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Decagonal quasicrytal; Diffuse intensity; Electron microscopy; Ordering; Striation contrast

1. Introduction

The basic structure of decagonal quasicrystal (DQC) was proposed by Steurer and Kuo [1] for Al–Cu–Co. It was later refined for Al–Co–Ni and a cluster model (with 2.0 nm clusters) has been advocated [2–5]. All these models have considered only 0.4 nm periodicity along *c*-axis. But it has now been established that the stable quasicrystals in Al–Cu–Co and Al–Ni–Co exist in two different variations with *c*-axis parameter of ∼0.4 and ∼0.8 nm respectively [6]. Ritsch et al. [6] have elegantly demonstrated the relations between these variable periodicities via ordering reaction in the decagonal structure. They have also reported two different types of superstructure having 0.8 nm periodicity. These superstructures transform to disordered structures with 0.4 nm period after electron beam irradiation. Pramanick et al. [7] have discussed an alternative model to explain the two periodicities on the basis of 6D structure [8]. Earlier Edagawa et al. [9,10] reported the existence of an ordered superlattice in decagonal Al–Ni–Co and established an order–disorder transformation at 800°C. However, superlattice order has not been observed by them in Al–Cu–Co. Many other ordering possibilities such as Type I, Type II superstructures have been reported in Al–Ni–Co [11]. Song et al. [12] proposed the model of twinning of the microcrystalline (MC) phases in order to interpret the tweed contrast in transmission electron microscopy (TEM) images. Recently, Zhang et al. [13] have reported diffuse intensity and tweed contrast in Al–Cu–Co DQC phase and interpreted this as due to strain arising from the formation of localized crystalline phases in the DQC structure. The aim of the present paper is to study diffuse intensity in electron diffraction pattern in order to understand the origin of striation contrast observed in electron microscopy images of DQC phase in $Al_{62}Cu_{20}Co_{15}Si_3$ system.

2. Experimental

An Al–Cu–Co–Si alloy, prepared by melting the constituent elements (close to the $Al_{62}Cu_{20}Co_{15}Si_3$ composition) was remelted and solidified slowly in order to synthesise large size single crystal of DQC phase. The individual needles formed during solidification were extracted and used for X-ray diffraction and for TEM. Thin slices of the needles (transverse section of the individual needle) were prepared by mechanical polishing, dimple grinding and ion milling. The thinned sample was observed in a Philips CM-12, transmission electron microscope.

3. Results and discussions

Fig. 1 shows a 10-fold diffraction pattern displaying many subtle features. Diffuse streaks are observed around the most intense spots along all the P directions {1 0 0 0 0 0}.

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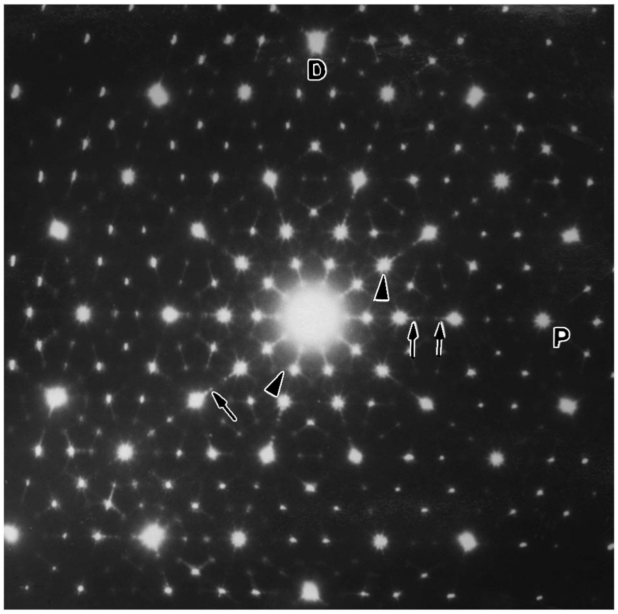

Fig. 1. Tenfold diffraction pattern obtained from an ion milled specimen. Diffuse streaks arrowhead, distortion (marked as D) and satellites (arrowhead) spots are noted.

The intense spots in the D directions {1 1 0 0 0 0} (marked as D) are non-circular in shape and show diffuse streaks radiating along the P directions. It is now well understood that this shape distortion is a result of the superposition of reflections from five variants of the B2 phase with the DQC spots [14]. The spots contain diffuse streaks along all the P directions and thus become star shaped in general. Diffuse streaks are also present at positions marked by the arrowhead. These are distinguished from the other streaks by the fact that only three of the five pronged sides of the pentagon of spots are connected by the streaks. The spread of the diffuse streaks corresponds to a length of 2.0–2.4 nm in real space. Sateuite spots can also be observed (marked by small arrow), which appear to follow closely Type II superstructure reflections [11]. Fig. 2 shows a bright field image in a near two-beam condition ($\mathbf{g}_p$), where some weak spots of the systematics row survive, revealing tweed contrast along with one set of lines lying perpendicular to $\mathbf{g}_p$. The other sets of lines generally seen in 10-fold orientation are not visible in this image. The important features of this image are the prominent rhombic domain structure, the fine parallel lines (hereafter referred to as striations) within the rhombic

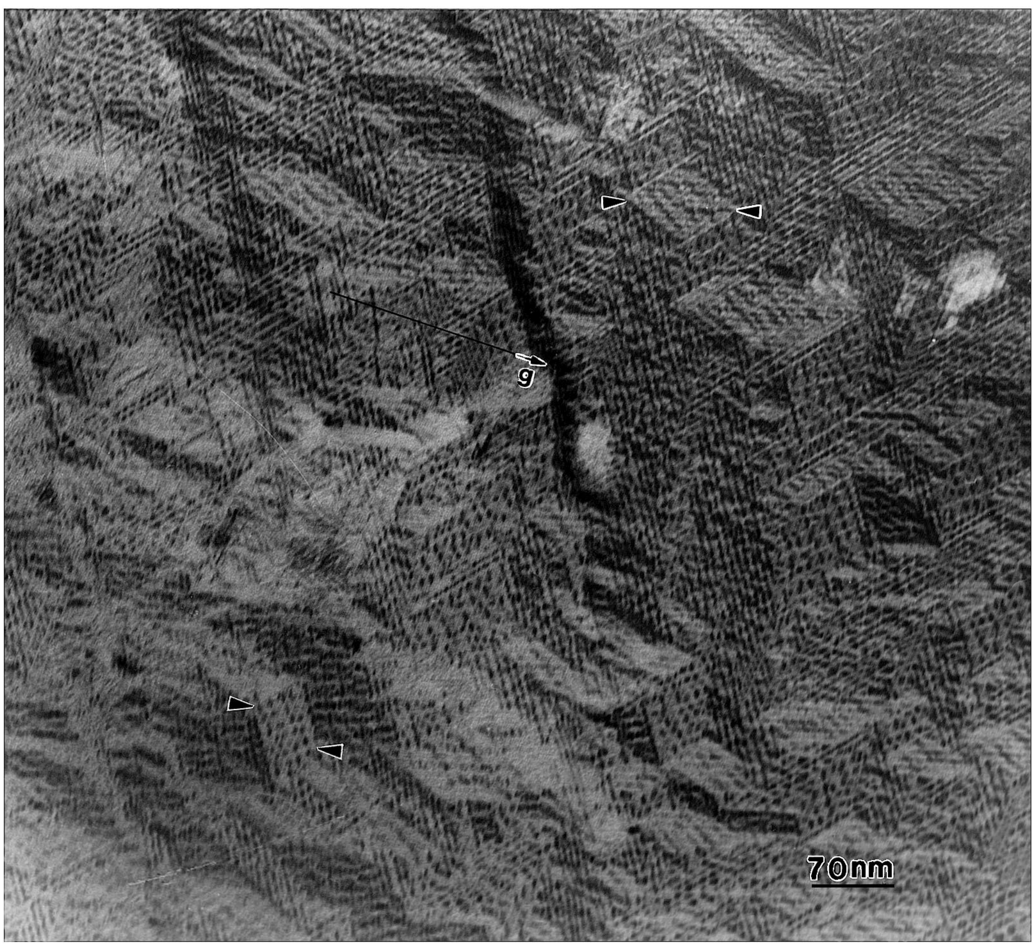

Fig. 2. Bright field image taken in nearly two beam condition showing the linear fringe type contrast perpendicular to $\mathbf{g}_p$ vector. Rhombic domain has been indicated (arrowheads).

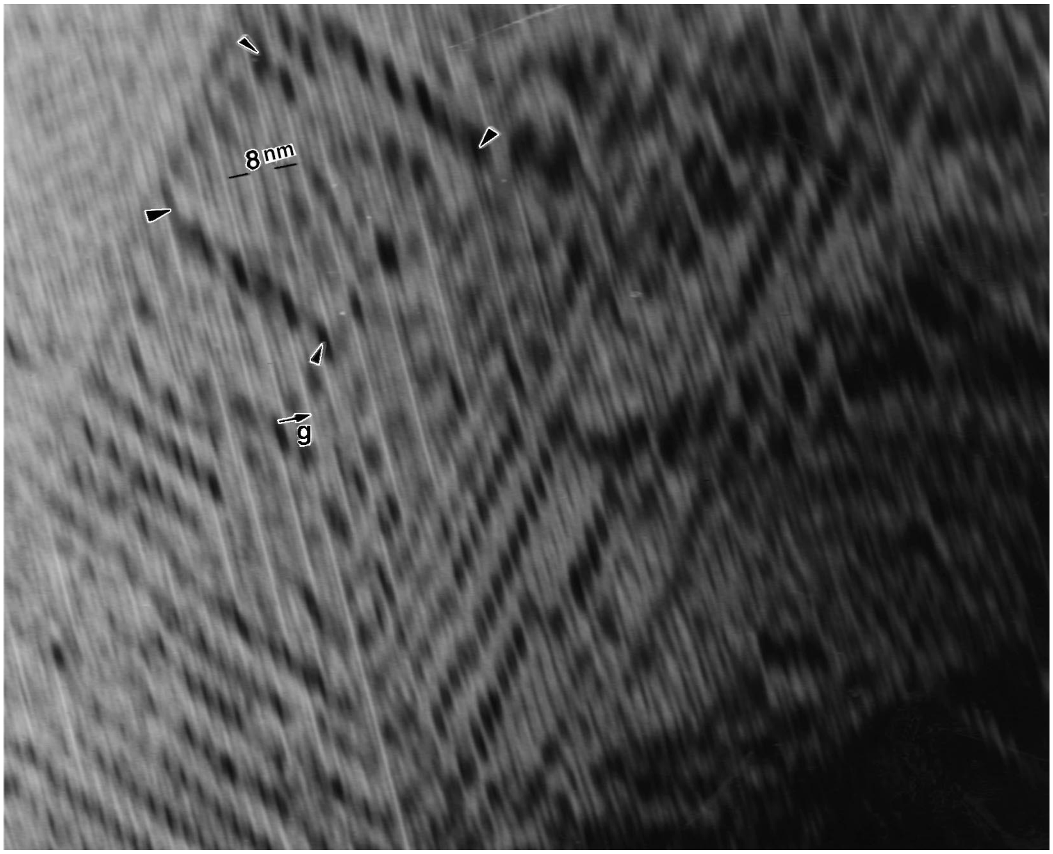

Fig. 3. High magnification dark field image of $\mathbf{g}_p$ vector, showing striations displaying various Fibonacci related spacings. Rhombic domains can be observed (marked arrow heads).

domain, perpendicular to $\mathbf{g}_p$ as noted already, and dark patches of contrast within the domains. In dark field imaging using a $\mathbf{g}_p$ reflection (0 1 1 0 1 0) type, we observe bright striations of variable spacing as shown in Fig. 3. The striations are perpendicular to the selected $\mathbf{g}_p$ vectors and they approximately define the rhombic domains (marked by arrowheads) described earlier. Similar DF imaging experiments were performed for other $\mathbf{g}_p$ vectors (rotated by 36°). The striations appear consistently in all orientations normal to the P directions. This image shows the lowest fringe spacing resolved in this study (∼0.5–0.6 nm) along with bright striations as observed in the other micrographs. The various spacings observed for bright striations in all the DF images were found to be in the order of ∼1, 2, 3, 5, 8, 13 and 21 nm with an accuracy of ±0.1 nm. The spacing varies and follows the Fibonacci relation, i.e, any larger spacing can be dissociated into a combination of smaller spacings. The striations are perpendicular to the diffuse streaking observed in Fig. 1.

Zhang et al. [13] attributed the contrast to the strain field associated with the small crystalline regions observed by them in their high resolution electron microscopy (HREM) pictures. In their study, the striations which were perpendicular to twofold **g** vectors were present up to a length scale of 100 nm. On the contrary, the fine, so-called crystalline regions in their HREM images extended only to 4–5 nm in length and were one unit cell thick. We disagree from the interpretation of the striation contrast given by Zhang et al. [13]. Similar rhombic domains were observed earlier by Song et al. [12] and interpreted as due to twinning of microcrystalline phases. However, their interpretation of the domains as microcrystals 20–500 nm in size was not supported by their HREM images. The rhombic domains in our case appear to contain striations with different spacings in different regions [15]. The spacings of the striations are τ related as noted earlier and display an angular relation of 72° between adjoining boundaries when all the boundaries are imaged. Thus, they conform to the geometry of the underlying quasiperiodic lattice. It is interesting to note that Hradil et al. [16] interpreted the satellite scattering in quasiperiodic layers in X-ray diffraction (XRD) of Al–Cu–Co–Si system, due to either by superordering in higher dimensional space or by lammellar periodic microdomain superimposition in quasiperiodic structure. The diffuse streaks starting from Bragg's reflection and ending up on satellites have been attributed to one dimensional disorder relative to superlattice structure. Our present electron microscopy analysis supports the idea of phason type disordering in superordered structures.

4. Conclusions

The diffuse streaks observed in the selected area diffraction patterns (SADPs) are attributed to the presence of phason boundaries evolving from phason disorder. The structural rearrangement in the DQC phase leads to an unusual striation contrast in the images, giving rise to features with phason disorder which causes anisotropic diffuse streaks. The existence of weak superlattice sattelite spots indicates onset of ordering in decagonal structures. In the present study we have obtained a DQC phase which exhibits partially ordered superlattice structure along with certain amount of phason disorder.

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