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THE CRYSTALLOGRAPHIC ORIENTATION OF $\text{Al}_5\text{Ga}_3\text{V}$ AND $\text{Nb}_3\text{Al}$

PRECIPITATES IN BCC MATRICES

By

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Convergent-beam transmission electron diffraction was used to determine the crystallographic orientation of sub-micron sized Al5 structure $V_3^5$Ga and $Nb_3^2$Al precipitates formed by solid state precipitation from supersaturated BCC solutions. In each case the crystallographic orientation relation was (to within an uncertainty of $\pm 5^\circ$): $[001]_{Al5} \parallel [111]_{BCC}$; $(100)_{Al5} \parallel (110)_{BCC}$. This lattice correspondence is intuitively plausible since it ensures that the closest packed planes and directions of the precipitate parallel those of the matrix.
Crystalline compounds of the Al$_5$ structure are of scientific and engineering interest because of their excellent superconductiong properties. The promising Al$_5$ compounds are, however, invariably brittle, and therefore cannot be directly formed into superconducting wire or tape. Al$_5$ superconducting materials are hence usually manufactured in a two-step process, in which wire or tape containing the metallic constituents of the Al$_5$ structure is rolled, drawn, or extruded, and the Al$_5$ compound is subsequently introduced through an appropriate thermal treatment which causes its precipitation in the solid state. To establish metallurgical control over the processing of Al$_5$ superconducting materials it is important to achieve a fundamental understanding of the crystallography, mechanism, and kinetics of the relevant precipitation reactions.

The present investigation was undertaken to determine the common crystallographic relations which govern the orientation of Al$_5$ precipitates in BCC media. The two cases specifically studied were: V$_3$Ga (Al$_5$) in a vanadium-rich V-Ga solid solution (BCC) and Nb$_3$Al (Al$_5$) in a niobium-rich Nb-Al solid solution (BCC). Samples were made by casting and homogenizing solute-rich metallic solid solutions, deforming these into thin tapes by warm rolling, and aging at intermediate temperature to precipitate the Al$_5$ phase. The specific examples illustrated in the accompanying figures are: (1) V-18 at.% Ga, deformed 90\% and aged at 700°C for 12 hours, and (2) Nb-18 at.% Al, deformed 99\% and aged at 750°C for 3 hours. Thin foil specimens for transmission electron microscopic analysis were thinned from these tapes, and examined through both conventional and convergent beam electron diffraction analyses to determining the crystallographic relations between the Al$_5$ precipitates and the BCC matrix phase.

The analysis of the V$_3$Ga precipitate is illustrated in Fig. 1 (a-c). The transmission electron micrograph presented in Fig. 1a shows the precipitates
The $V_3\text{Ga}$ precipitates are lenticular in shape and have an average size of ~3000 Å length by 1300 Å width. A typical convergent beam diffraction pattern from this sample is shown in Fig. 1b: the disc-shape of the diffraction spots is due to the angular convergence of the incident electron beam. The diffraction pattern is indexed in Fig. 1c. The zone axes of the BCC matrix and the Al5 precipitates are [111] and [001], respectively. The rows of parallel matrix and precipitate spots in the diffraction pattern indicate parallel planes (within ±5°) since the corresponding reflecting planes are almost parallel to the electron beam (Bragg angle < 1°). It follows that the (100) plane of the Al5 precipitate is essentially parallel to the (110) plane of the BCC matrix. The complete orientation relation is hence given by the appealingly simple correspondence:

$$[001]_{\text{Al5}} || [111]_{\text{BCC}} ; (100)_{\text{Al5}} || (110)_{\text{BCC}}$$

The analysis of the Nb-Al system is illustrated in Fig. 2a-b. The transmission electron micrograph presented in Fig. 2a shows that the $Nb_3\text{Al}$ precipitates formed from Nb-18 at.%Al on aging at 750°C for 3 hours are morphologically different from the $V_3\text{Ga}$ precipitates illustrated in Fig. 1; the precipitates are more nearly equi-axed and tend to attach to one another rather than extending into the matrix (the morphological contrast is discussed in more detail in ref. 2). Nonetheless, selected-area convergent beam diffraction patterns (Fig. 2b) reveal an Al5-BCC lattice correspondence identical to that found in the $V_3\text{Ga}$ case and given by relation (1).

The Al5-BCC lattice correspondence found in this study is illustrated in Fig. 3. It is an intuitively plausible correspondence since it insures that the most closely packed planes and directions of the Al5 and BCC structure...
are parallel to one another, and in this sense resembles the familiar Kurdjumov-Sachs relation between the FCC and BCC structures. This relation does, however, differ from that previously suggested by Togano, et al. from an analysis of surface diffused $V_3Ga$ on $V$ tape: $[110]_{A15} \parallel [120]_{BCC}$; $(001)_{A15} \parallel (001)_{BCC}$. The Togano correspondence was never found in this work. Using X-ray and reflection electron diffraction (RED) techniques, Diadiuk, et al. studied the preferred orientations of Nb$_3$Sn diffusion layers grown by Sn-vapor reaction with single crystal Nb substrates; the preferred parallel planes were given: $(100)_{BCC} \parallel (110)_{A15}$, $(110)_{BCC} \parallel (100)_{A15}$, $(211)_{BCC} \parallel (110)_{A15}$, and $(111)_{BCC} \parallel (111)_{A15}$ for the four pre-selected BCC orientations. The respective parallel directions, however, could not be observed by their techniques. It has been known that the parallel planes and directions are both necessary to determine crystallographic orientation relationships between two crystal structures. It should also be noted that for a Sn-diffusion reaction on a bulk Nb substrate, the growth direction of the A15 Nb$_3$Sn layers is restricted by the orientation of the BCC Nb substrate, rather than being permitted to take a path to minimize the free energy.

The orientation relations presented here are uncertain by $\pm 5^\circ$ due to elongation of the reciprocal lattice in the electron diffraction patterns. This ambiguity can be removed by high-angle tilting experiments which are now in progress.

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REFERENCES

FIGURE CAPTIONS

1. (a) Transmission electron micrograph of V-18 at.\% Ga deformed 90\% and aged at 700°C for 12 hours. Regions A and B are A-15 and BCC phases, respectively. (b) The correspondent convergent beam electron diffraction pattern. (c) Index pattern of (b).

2. (a) Transmission electron micrograph of Nb-18 at.\% Al deformed 99\% and aged at 750°C for 3 hours. Regions A and B are A-15 and BCC phases, respectively. (b) The correspondent convergent beam electron diffraction pattern.

3. The superimposed conventional unit cells of BCC and A-15 structures with the parallel planes (heavy lines) and direction (large dash line) indicated.
Figure 3