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FURTHER EVIDENCE OF THE LOW TEMPERATURE PHASE
TRANSFORMATION IN Nb_3Sn AND V_3Si

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ABSTRACT

Low temperature x-ray diffraction studies show that the cubic $\rightarrow$ tetragonal martensitic transformation in Nb_3Sn and V_3Si is accompanied by a change in volume and is therefore a first order thermodynamic phase change. The axial ratio of the Nb_3Sn martensite is confirmed to be less than unity.

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Conflicting views have recently been expressed in the literature⁽¹⁻⁶⁾ concerning the thermodynamic order of the low temperature cubic $\rightarrow$ tetragonal martensitic transformation in the superconductors Nb_3Sn and V_3Si . In addition, it is known that the occurrence of the phase change is determined by the nature and condition of the sample. Although evidence of the martensite is usually observed in X-ray powder specimens,⁽¹⁻³⁾ Batterman and Barrett⁽²⁾ found that not all single crystals cut from the same bar of V_3Si transform on cooling, while Mailfert, Batterman and Hanak⁽³⁾ only detected the transformation in single crystals of Nb_3Sn after a heat treatment which lowered the tin content. In a previous investigation,⁽⁷⁾ we observed that bulk samples of V_3Si and Nb_3Sn showed no surface tilts indicative of a martensitic shear transformation yet powdered specimens from the same source showed X-ray line broadening.

In subsequent studies a tin-rich sample of Nb_3Sn , prepared at A.E.R.E. Harwell by diffusion of molten tin into niobium rods, showed no X-ray line broadening when examined at helium temperatures in a modified version of the X-ray diffractometer cryostat described by King and Preece.⁽⁸⁾ After a quantity of this sample was heated under a vacuum of 3×10^{-7} mm Hg for eight days at 920°C , the lattice parameter at 299°K was lowered from $5.2961 \text{ \AA}$ to $5.2903 \text{ \AA}$ ($\lambda\text{CuK}\alpha_1 = 1.54051 \text{ \AA}$). Broadened diffraction profiles were observed when this sample was cooled to 9.4°K , confirming that the occurrence of the phase change can be related to the room temperature lattice parameter of the A15 cubic phase. The low temperature line broadening is reversible on heating, as illustrated in Figure 1 by the repeated scans through the 520, 432 diffraction profile. The $\text{K}\alpha_2$ component can be distinguished at 21°K and the sharp cubic profile is recovered at 28.5°K .

Since the reverse transformation may not occur simultaneously throughout the powder specimen, this recovery interval is understandably greater than the 2°K reported for single crystal specimens. (1-3)

Many of the broadened back reflection profiles in Figure 1 are composed of up to six different $K\alpha_1$ and six $K\alpha_2$ tetragonal components and for this reason powder diffraction results have in the past been regarded as ambiguous. (1-3) The 440 tetragonal profile, however, can definitely be resolved into a relatively weak $K\alpha_1$ peak attributable to the 440 component, and more intense $K\alpha_1$ and $K\alpha_2$ peaks composed of superimposed 404 and 440 reflections. The shift of the low l index tetragonal component towards lower Bragg angles indicates that the axial ratio of the martensite is less than unity. This was confirmed from the ratio of the $\sin \theta$ values for 404 and 440 $K\alpha_1$ peaks, which gives c/a as 0.9964 in good agreement with the single crystal results of Mailfert et al. (3)

It is also apparent from the Figure that both the 404 and the 440 $K\alpha_1$ tetragonal peaks occur at lower Bragg angles than the cubic 440 $K\alpha_1$ peak. A careful examination of the diffraction data has shown that this effect is quite general, i.e., the centroids of all the unresolved tetragonal profiles are shifted to Bragg angles lower than those of the respective cubic reflections. Similar centroid shifts accompanying line broadening have been observed in a further sample of Nb_3Sn ($a = 5.2896 \text{ \AA}$ at 298°K) and also in V_3Si ($a = 4.7248 \text{ \AA}$ at 298°K). After eliminating geometrical errors by extrapolation procedures, (9) it can be shown that the centroid positions of the broadened diffraction profiles indicate that the volume of the tetragonal cell at 9.4°K is of the order of 0.2% greater than that of the Al_5 cubic cell at 77°K . This clearly demonstrates that the

martensitic transformation involves a change in volume and must therefore be regarded as a first order thermodynamic phase change in support of the theoretical models of Labbé and Friedel⁽⁵⁾ and Cohen, Cody and Holloran.⁽⁶⁾

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Fig. 1 Low temperature X-ray diffraction profiles of powdered Nb_3Sn ($a = 5.2903 \text{ \AA}$ at 299°K).

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