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BNL-25949

CONF-790610B-12

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to be presented at the

INTERNATIONAL CONFERENCE ON MARTENSITIC TRANSFORMATIONS

(ICOMAT-79)

Massachusetts Institute of Technology

Cambridge, Massachusetts, USA

24-29 June 1979

*Research supported by the U. S. Department of Energy.

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Softening of Young's Modulus of Polycrystalline Nb_3Sn

D. O. Welch, J. F. Bussière, and M. Suenaga

Ultrasonic measurements show that the shear modulus $(C_{11}-C_{12})/2$ softens dramatically as single crystals of Nb_3Sn approach the martensitic transition near 50K. It is expected that Young's modulus of polycrystals will also soften, but previous ultrasonic measurements, which suffer from severe damping, fail to show the expected effect. We have measured Young's modulus of polycrystalline Nb_3Sn between 4.2K and 300K by static beam deflection methods, and observe marked softening. A value of $14.3 \pm 5 \times 10^{11}$ dyne cm^{-2} was obtained at 300K by the deflection of thin Nb_3Sn -Nb- Nb_3Sn composite strips by external stress. The variation of the modulus with T was obtained from the change in the radius of curvature of internally stressed Nb_3Sn -Nb composite strips. (This method is made possible by the near perfect match between the thermal expansion coefficients of Nb and Nb_3Sn .) The modulus is found to be proportional to $\ln T$ between 50K and 300K and is temperature independent below the superconducting T_c , resulting in a decrease by a factor of ~ 2 between 300K and 18K. The observed softening is somewhat less than that predicted by a polycrystalline average of the experimental single crystal elastic constants, but is much larger than that observed with ultrasonic measurements.

I. Introduction

Nb_3Sn undergoes a reversible cubic = tetragonal martensitic phase transition near 50K. This transition is sensitive to the degree of order, presence of defects, and other microstructural properties; a summary of the characteristics of the transition in Nb_3Sn and other Al₅ compounds can be found in [1]. The temperature dependence of the elastic constants of single crystals and its relation to the phase transition have been studied in detail, both theoretically and experimentally [1,2]. A most striking feature is the vanishing of the shear modulus $(C_{11}-C_{12})/2$, and hence of Young's modulus in the [100] direction, at the transition.

Since technological applications of Nb_3Sn as a superconductor utilize polycrystals, their elastic behavior and its temperature dependence are of interest. Measurements on polycrystals, however, are scarce and inconsistent with single crystal data. Old and Charlesworth [3] found a value for Young's modulus at 300K of 17.9×10^{11} dyne cm^{-2} using an ultrasonic "critical angle" technique, while the published sound velocity data of Testardi et al. [4] lead to a value of 23.6×10^{11} dyne cm^{-2} . These values are considerably higher than is obtained by a Voigt-Reuss-Hill polycrystalline average [5] of single crystal constants [6,7], 13.7×10^{11} dyne cm^{-2} . Furthermore the Young's modulus derived from sound velocity data [4] softens much less than the modulus derived from single crystal data [7], as may be seen in Figure 1.

Such discrepancies do not occur for the Al5 compound V_3Si , for which the softening predicted by polycrystalline averaging agrees with that from ultrasonic data [4]; dynamic resonance experiments on V_3Si polycrystals also show substantial softening [8]. In order to determine whether or not polycrystals of Nb_3Sn show substantial softening of Young's modulus as the temperature approaches that of the martensitic phase transition, we have measured the static deflection of bimetallic ($Nb+Nb_3Sn$) composite strips under the influence of internal and external stress and derived the Young's modulus of Nb_3Sn in the range of 4.2 to 300K.

II. Experimental Procedure

$Nb_3Sn/Nb/Nb_3Sn$ composite strips were made by a solid state diffusion process (the "bronze process"). Initially, a Cu-13wt%Sn bronze is cast around a 3.2mmx40mmx150mm plate of pure niobium. The bronze/niobium composite is then rolled to obtain a tape of niobium ($\sim 25\mu m$ thick) with $\sim 50\mu m$ of bronze on each side, which is then slit into 12mm x50mm pieces and reacted at 725°C for 50h. This results in Nb_3Sn layers of $\sim 3-4\mu m$ thickness at the interface between the niobium and the bronze.

Because the thermal expansion coefficient of the bronze substantially exceeds that of the Nb and Nb_3Sn , cooling from the reaction temperature, 725°C, results in an internal stress. With bronze layers of the thickness in these experiments, the internal stress is sufficient to cause plastic flow in the unreacted Nb during cooling. Thus, an internal stress remains in the $Nb_3Sn/Nb/Nb_3Sn$ composite which is obtained by etching away the bronze layers in dilute nitric acid. It is possible to remove this internal stress by annealing the composite strip (without bronze) at 725°C for 1/2 hour.

The temperature dependence of Young's modulus was determined with internally stressed composite strips (unannealed). The presence of the internal stress is made manifest by etching away one of the Nb_3Sn layers in 4 parts HNO_3 , 2 parts H_2O , and 1 part HF, a solution which etches Nb_3Sn much faster than it does Nb. The resulting Nb_3Sn/Nb asymmetric composite curls with the Nb on the inside diameter, indicating that the Nb_3Sn was initially in compression. As described below, the thermal expansion coefficients of Nb and Nb_3Sn are very well matched; thus changes in the radius of curvature of the curled composites with temperature can be used to deduce the temperature dependence of the Young's modulus of Nb_3Sn .

The match between the thermal expansion of Nb_3Sn and Nb was determined by measuring the radius of curvature of a composite, annealed at 725°C for 1/2 hour (without the bronze matrix), where one layer of Nb_3Sn is removed. These measurements indicate that the mismatch in $\Delta L/L$ of the two materials between 725°C and room temperature is less than 10^{-5} and less than 5×10^{-6} between 300K and 4.2K. Thus any curvature in unannealed composites can be attributed to mismatch due to plastic flow in the Nb and this mismatch, Δ , is independent of temperature below room temperature.

With isotropic elasticity theory, it is straightforward to derive the resulting radius of curvature, R , of the bimetallic composite [9, 10]. The result is of the form:

$$\Delta = R^{-1} F(E_1, E_0, \nu_1, \nu_0, l_1, l_0) \quad (1)$$

where E is Young's modulus, ν is Poisson's ratio, l is the layer thickness, and the subscripts one and zero refer to Nb_3Sn and Nb , respectively. Because Δ is independent of temperature, the temperature dependence of the radius of curvature is of the form:

$$R(T)/R(300\text{K}) = F(300\text{K})/F(T) \quad (2)$$

where the temperature dependence of F comes primarily from that of the Young's moduli; we shall neglect the temperature dependence of Poisson's ratio (a source of $\sim 10\%$ error). Explicitly writing F yields the relation between the temperature dependence of R and E [9]:

$$\frac{E'_1}{E_1} = \frac{RE'_0}{R'E_0} \cdot \frac{2}{a} \left\{ 1 + \left[1 - 4k^{-2} a^{-2} L^2 (R/R')^2 \right]^{1/2} \right\}^{-1} \quad (3)$$

where:

$$L \equiv \frac{l_1}{l_0} \quad k \equiv \frac{E_0 l_0 (1 - \nu_1)}{E_1 l_1 (1 - \nu_0)}$$

$$a \equiv k^{-2} \{ (1+k)(L^2+k) + 3k(1+L)^2 - k(R/R') (4+6L+4L^2) \}$$

and the primed and unprimed symbols refer to values at low temperatures and room temperature respectively; the subscripts one and zero refer to Nb_3Sn and Nb , respectively.

The temperature dependence of R was measured by enclosing a $\text{Nb}_3\text{Sn}/\text{Nb}$ composite strip in a cryostat with windows through which the strip could be photographed at temperatures between 4.2 and 300K. The radius of curvature was obtained by measuring the negatives in an optical comparator. These values, when inserted in Eq. (3) together with elastic data for Nb , yield the temperature dependence of E for Nb_3Sn .

III. Results and Discussion

The radii of curvature of two curled $\text{Nb}/\text{Nb}_3\text{Sn}$ bimetallic strips, with characteristics summarized in Table I, were measured between 4.2 and 300K; changes were of the order of $\sim 40\%$. These results were used, as described above, to derive the data shown in Fig. 1. For simplicity, the Poisson's ratios of Nb and Nb_3Sn were taken to be equal and temperature independent (resulting in an error of $\sim 10\%$); a Voigt-Reuss-Hill average of the single crystal data of [11] was used for the temperature dependent Young's modulus of Nb . Fig. 1 shows that $E_{\text{Nb}_3\text{Sn}}$ softens by about a factor of two between 300K and 50K, in marked contrast with the sound velocity data of [4]. The softening is somewhat sample dependent and is somewhat less than that predicted by a Voigt-Reuss-Hill polycrystalline average of single crystal data. The modulus