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DIRECT OBSERVATION OF CRYSTALLOGRAPHICAL CHANGES AT 10 K CAUSED BY THE APPLICATION OF VARYING STRESSES TO Nb_3Sn WIRES

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Résumé - Des mesures de diffraction aux rayons X sur des fils de Nb_3Sn mono- et multifilamentaires ont été effectuées sous tension mécanique variable dans le domaine de températures $5 \leq T \leq 300$ K à l'aide d'un nouveau dispositif. La variation des modifications de la phase A15 dans ces fils a pu être détectée en fonction de l'élongation ε.

Abstract - X-ray diffraction measurements of Nb_3Sn mono- and multifilamentary tapes have been performed in the temperature range $5 < T < 300$ K under variable axial stress by means of a newly constructed device. The changes of the stress-induced crystallographical modifications of the A15 phase have been observed as a function of applied strain ε.

I - INTRODUCTION

It is well established that the observed maximum of J_c and B_{c2} in Nb_3Sn multifilamentary wires as a function of the applied stress is connected with the state of precompression of the Nb_3Sn filaments caused by the surrounding bronze, which posseses a larger thermal contraction coefficient than the A15 layer /1/. Recently, this behavior has been attributed to stress induced crystallographical modifications of the A15 phase. By means of neutron and X-ray diffractometry on Nb_3Sn wires in the temperature range between 10 and 300 K, two new modifications of the A15 phase were detected, which were called T_2 and T_3 for better distinction /2,3/. In this nomenclature, T_1 is the well-known spontaneous forming tetragonal phase characterized by $T_M = 43$ K and $1-c/a \approx 0.006$ /4/. T_2 is an elastic tetragonal distortion of the cubic A15 structure, which increases continuously upon cooling the wires from the reaction temperature, 1000 K, to the operation temperature, 4,2 K. In contrast to T_1, the elastic tetragonal distortion T_2 does not form by a thermodynamical phase transformation at a fixed temperature, T_M, but by a gradual change of the stress field. In addition, $1-c/a$ is not constant over the volume, but is dependent on the angle α, the orientation of the Nb_3Sn crystallites with respect to the wire axis /2/. So far, the variation $1-c/a$ of T_2 with α gives the only possibility to determine the stress distribution acting on the filament. As pointed out in Ref. 2, the maximum value of $1-c/a$ for crystallites having the [001]axis parallel to the wire axis is ~ 0.0028 at 104 K. Additional to T_2 a new stress induced phase, T_3, occurs below temperatures ranging between 80 and 150 K. depending on the individual wire configuration. In contrast to earlier attempts to describe the stress effects with a linear uniaxial model, neutron diffraction experiments /2/ have shown that the anisotropy of the stress field has to be taken into account. From the measurement of T_c in multifilamentary Nb_3Sn wires it can be seen that the stress induced crystallographical modifications exhibit lower T_c values. In previous crystallographical investigations in Nb_3Sn composite wires, the variation of stress was caused by a variation of the temperature, T. In the present work, we report the first observation of crystallographical effects on T_2 and T_3 upon external stress application, keeping T = const. in full analogy to the J_c versus ε curve.

II - EXPERIMENTAL

X-ray investigations using a Bragg-Brentano beam geometry require sufficiently flat sample surfaces. Therefore a band-shaped geometry of the conductor was chosen. The preparation by the bronze route started with a round mono- or multifilamentary wire wrapped by additional bronze, which was deformed to a band and reacted under conditions resulting in homogeneous A15 layers. Because of the limited penetration depth of the CuKα radiation the Nb_3Sn layer needed to be layed bare by etching as indicated in Fig. 1.

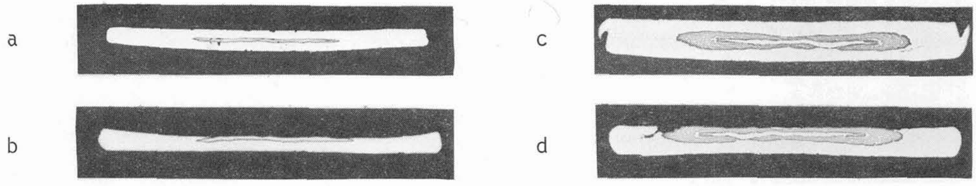

Fig. 1 - a, b: Monofilamentary Nb_3Sn wire; c, d: multifilamentary Nb_3Sn wire; in b, d the bronze was etched away.

Our Nb_3Sn band especially prepared for the X-ray investigations had the following specifications:

1. Nb_3Sn monofilamentary band (1.9 x 0.13 mm cross section) reacted 100 h at 725°C, 5µm layer thickness; bronze/(Nb + Nb_3Sn) ratio = 8.7 : 1 (Fig. 1a), etched on one side, 7 : 1 (Fig. 1b).
2. Nb_3Sn multifilamentary band (originally wire of 0.91 mm Ø, supplied by Vacuumschmelze, deformed to 2.1 x 0.24 mm cross section), reacted 64 h at 700°C filament diameter about 1.5 - 2µm bronze/(Nb + Nb_3Sn) ratio = 12 : 1 (s. Fig. 1c) (including extra bronze), etched at one side, 8 : 1 (s. Fig. 1d).

The increased bronze/(Nb + Nb_3Sn) ratio was chosen for two reasons. First, it is well known that the amount of prestress is a function of the bronze/(Nb + Nb_3Sn) ratio /5/. Second, the danger of loosing too much of the prestress as a result of etching or polishing the bronze away on one side of the band, is reduced.

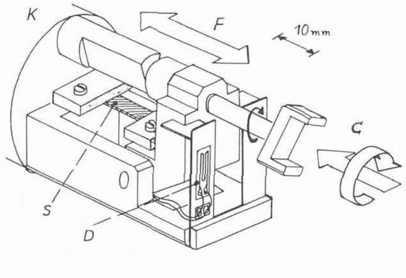

Fig. 2 - Strain rig (abbr. s. text)

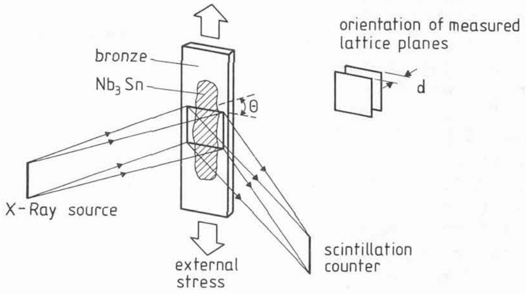

Fig. 3 - Bragg-Brentano beam geometry

For mounting, band segments of 15 mm length were soldered on flat copper bars (s. S in Fig. 2), which were fixed with screws on the low temperature strain rig. This newly constructed strain rig (s. Fig. 2) allows stress applications to the superconductor while keeping it at any desired temperature between 10 K and 300 K. The strain rig was mounted on a modified Oxford continuous flow cryostat. The CLTS temperature sensor was placed on the sample holder for accurate temperature control. A mechanical screwdriver-like feedthrough allows connection and disconnection to the screw of the strain rig (C in Fig. 2) at low temperatures. For the necessary short connection time of about 20 - 30 sec when varying the stress, the thermal contact leads to a negligible temperature raise of only 3 - 5 degrees. By turning the "screwdriver" an axial stress is applied to the probe as indicated by the arrow F (Fig. 2). The resulting strain values were measured indirectly with a gauged DMS-halfbridge circuit

(D in Fig. 2). This strain rig is suitable for strain values up to 2% without significant defocusing of the diffractometer beam geometry.

The measurements were performed in the step scanning mode using a high resolution equipment, including a secondary quartz monochromator for elimination of the CuKα_2-line. As the most suitable reflexes for studying the different low temperature crystallographical phases the (200), (210), (211) and (400) reflexes were chosen. From the beam geometry shown in Fig. 3 it is obvious that in our case the measured lattice plane spacings d are perpendicular to the longitudinal axis of the conductor. Therefore our data represent the stress induced radial effects on the crystal structure. The axial effects are subsequently calculated using data about the volume of the unit cell determined previously by neutron diffractometry /2/.

III - RESULTS

The shape of the (400) reflex at initial prestress (ε = 0) at 10 K (s. Fig. 4) looks very similar to the neutron diffraction data /4/. The reflexes of the T_3 phase (left line) and the tetragonal distortion T_2 (right line) are well resolved, the portion of T_3 phase apparent in this orientation being 40%. The formation of the T_3 phase begins at about T ≈ 80 K in agreement with previous results /2/. The vertical arrows in Fig. 4 indicate the peak belonging to T_3 and the horizontal arrows indicate the peaks corresponding to the tetragonal distortion T_2 and their deviation from the stress free lattice parameter of 5.279 Å (10 K) (determined from an etched filament). Applying a stress to the probe two kinds of drastical changes can now be observed (s. Fig. 4). First the tetragonal distortion T_2 is reduced with increasing strain, indicated by a shift of the line to higher angles with unaltered intensity. Second the intensity of the T_3 line decreases and appears instead at a lattice parameter equivalent to the stress free state. For strain values $\varepsilon > 0.5\%$ the reflexes of T_2 and T_3 fall together.

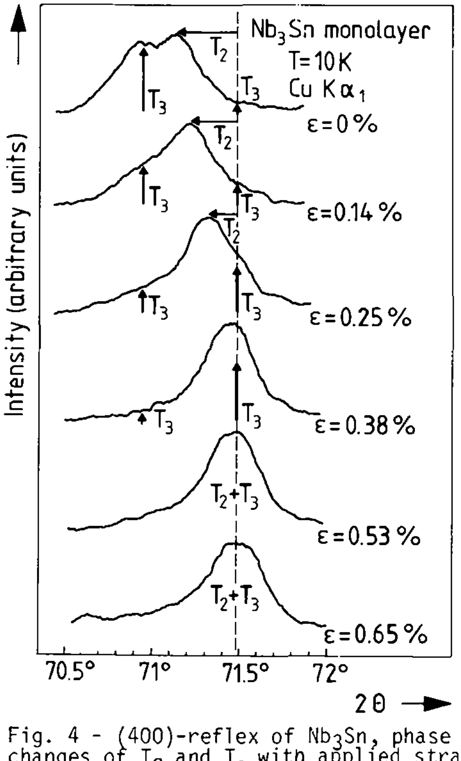

Fig. 4 - (400)-reflex of Nb$_3$Sn, phase changes of T_2 and T_3 with applied strain.

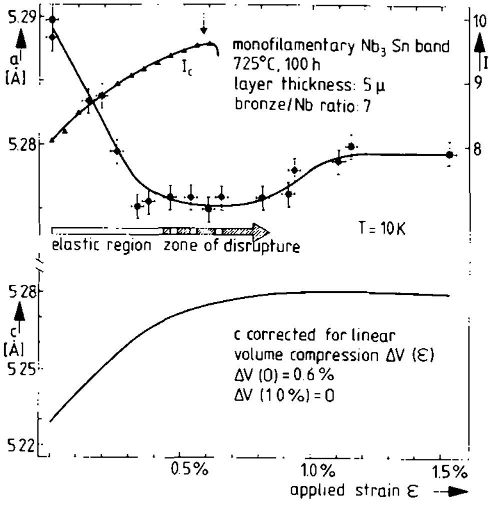

Fig. 5 - Critical current I_c, lattice parameters a (measured) and c (calculated, connected for volume compression) vs. strain ε.

In Fig. 5 the lattice parameter a of T_2 and the corresponding critical current I_c at B = 10 T are plotted as a function of the strain ε. Disruptures of the Nb_3Sn layer do not allow the measurement of the maximum of I_c, but results on wires with similar bronze/(Nb + Nb_3Sn) ratios /5/ suggest a maximum of I_c at ~ 0.8 to 0.9% strain. Starting from the measured lattice parameter, a, the shorter lattice parameter c can now be determined taking into account the elastic compression of the Nb_3Sn unit cell volume. During the cooling process the unit cell volume not only decreases with T, but also as a function of the increased prestress. From the temperature dependent neutron diffraction data (Fig. 6 in Ref. 2) the direct measurement of a and c leads to an observed elastic volume reduction of ΔV = 0.3% for a multifilamentary Nb_3Sn wire with a bronze/(Nb + Nb_3Sn) ratio of about 3. The volume compression was found to be a linear function of the strain. In our case with a bronze/(Nb + Nb_3Sn) ratio = 7 the prestress is incrased by a factor ~ 2, which leads to ΔV = 0.6%. This volume compression requires a pressure of 10 kbar which can easily be calculated via the bulk modulus (1730 kbar Ref. 6) using the definition formula.

Considering the linear decreasing of the volume compression upon stress application in the strain range 0 to 1% leads to the c versus strain curve in Fig. 5. In the beginning c raises strongly until it reaches the region of disrupture of the Nb_3Sn layer (compare with the I_c versus ε curve). For the multifilamentary binary Nb_3Sn tape a similar behavior to that of the monofilament was observed. Due to the larger bronze/(Nb + Nb_3Sn) ratio the (1-c/a) for ε = 0 of the tetragonal distortion was larger resulting in overlapping peaks of T_2 and T_3. With the application of stress similar effects as in the monofilamentary case were observed. The significantly weaker signal intensities of the multifilamentary band with respect to the monofilamentary allows only semiquantitative conclusions at this stage. Further results will be presented in a more detailed paper.

IV - CONCLUSIONS

The present experiments constitute the first direct observation of the influence of variable external stress on the crystallographical structure of Nb_3Sn. The behavior of the two stress-induced phases T_2 and T_3 upon stress application is quite different. The elastic tetragonal distortion T_2 shows an additional elastic volume compression in the range of 0.3 to 0.6%, depending on the bronze to Nb_3Sn ratio. The importance of radial effects represented by the variation of (1 - c/a) with the orientation α /2/ is confirmed by the present measurements, which yield a first quantitative information. The portion of the still unresolved phase T_3 is also a function of the precompression. In contrast to T_2, the volume fraction and not the lattice parameter of T_3 seem to be affected by stress. These results will be completed by our running investigations upon Nb_3Sn wires with ternary additions and internal reinforcement in view of the crystallographical reasons for the advanced critical properties of these technically meaningful superconductors.

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