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The Stress Induced Cubic-Tetragonal Phase Transformation and the Strain Behavior of J_c in Cu-Nb_3Sn Multifilamentary Wires

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Abstract

The low temperature Nb_3Sn phase diagram has been determined, as well under zero pressure as under the precompression conditions occurring in Cu-Nb_3Sn multifilamentary wires prepared by different techniques. Low temperature X-ray and neutron diffraction data furnish the evidence for a stress induced cubic-tetragonal phase transformation in Nb_3Sn wires and tapes. The Nb_3Sn phase of wires containing Ta or Hf+Ga additions did not transform. These observations serve as a basis for a model which explains the behavior of J_c under uniaxial stress by a gradual tetragonal-cubic-tetragonal phase transformation. This model is supported by a great number of literature data. As a consequence, the so-called "prestress effects" on J_c of Cu-Nb_3Sn wires can be practically avoided by choosing the appropriate additions to the Nb and the bronze, thus stabilizing the cubic phase. This fact is of great importance for large scale applications.

Der spannungsinduzierte kubisch-tetragonale Phasenübergang und das Dehnungsverhalten von J_c von Cu-Nb_3Sn Vielkernsupraleitern

Zusammenfassung

Die Phasenbeziehungen im System Nb_3Sn bei tiefen Temperaturen wurden bestimmt sowohl ohne äußeren Druck als auch unter den Vorspannungsbedingungen, die in verschiedenen Cu-Nb_3Sn Drähten vorherrschen. Mittels Röntgen- und Neutronendiffraktion bei tiefen Temperaturen wurde eine spannungsinduzierte kubisch-tetragonale Phasenumwandlung in Nb_3Sn Drähten und Bändern nachgewiesen. Unsere Drähte mit Ta und Hf+Ga Zusätzen zeigten keinen Phasenübergang. Diese Beobachtungen dienen als Basis für ein Modell, das das Verhalten von J_c unter uniaxialem Zug durch eine graduelle tetragonal-kubisch-tetragonale Phasenumwandlung erklärt. Dieses Modell wird auch durch eine große Anzahl von Literaturdaten bestätigt. Es folgt, daß die sogenannten "prestress" Effekte auf J_c von Cu-Nb_3Sn Drähten durch geeignete Wahl von Zusätzen zum Nb und zur Bronze praktisch vermieden werden können, sofern diese Zusätze die kubischen Phasen stabilisieren. Diese Tatsache ist sehr wichtig für Anwendungen im großen Maßstab.

THE STRESS INDUCED CUBIC-TETRAGONAL PHASE TRANSFORMATION AND THE STRAIN BEHAVIOR OF J_c IN Cu-Nb_3Sn MULTIFILAMENTARY WIRES

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Summary

The low temperature Nb_3Sn phase diagram has been determined, as well under zero pressure as under the precompression conditions occurring in Cu-Nb_3Sn multifilamentary wires prepared by different techniques. Low temperature X-ray and neutron diffraction data furnish the evidence for a stress induced cubic-tetragonal phase transformation in Nb_3Sn wires and tapes. The Nb_3Sn phase of wires containing Ta or Hf+Ga additions did not transform. These observations serve as a basis for a model which explains the behavior of J_c und uniaxial stress by a gradual tetragonal-cubic-tetragonal phase transformation. This model is supported by a great number of literature data. As a consequence, the so-called "prestress effects" on J_c of Cu-Nb_3Sn wires can be practically avoided by choosing the appropriate additions to the Nb and the bronze, thus stabilizing the cubic phase. This fact is of great importance for large scale applications.

I. Introduction

The critical current density, J_c, of multifilamentary Nb_3Sn superconductors under externally applied uniaxial tensile stress at 4.2 K is known to increase and to go through a maximum J_{cm} at a strain ε_m.[1-5] This effect is connected with the compressive strain on the Nb_3Sn filaments resulting from the larger thermal contraction of the surrounding bronze.[2] The ratio J_{cm}/J_{co}, where J_{co} is the value of J_c at $\sigma = 0$, characterizes the state of precompression (also called prestress) in the superconducting wire and depends strongly on the preparation technique of the latter. The value of J_{cm}/J_{co} increases with the applied magnetic field. For multifilamentary wires with comparable bronze/Nb_3Sn ratios, the value of J_{cm}/J_{co} at 16 T is 2.2, 3.0, and 4.5 for wires prepared by the bronze process, the "In Situ" technique[3] or the powdermetallurgical technique,[4] respectively. This behavior is in sharp contrast to that observed in V_3Ga wires,[5,7] where a very small precompression effect on J_c is observed.

Hoard et al.[8] formulated the idea that this different behavior in Nb_3Sn and V_3Ga could be correlated to the low temperature phase transformation in Nb_3Sn, but no experimental proof has been reported so far. It is the aim of the present work to study the correlation between the changes J_{cm}/J_{co} in Nb_3Sn and the occurrence of the low temperature tetragonal phase by means of low temperature X-ray and neutron diffraction.

II. Experimental

In order to get a general view, the present investigation includes wires prepared by the bronze technique (supplied by Vacuumschmelze) as well as "In Situ", powdermetallurgical and Cu matrix processed wires (Table I), prepared in our laboratories.

It is obvious that neutron diffraction would be more appropriate to the wire geometry, since the limited penetration depth of the X-ray beam is avoided. However, due to the minimum required quantity of 10 g wire material, neutron diffraction analysis was only performed on the bronze wire on the FR 2 reactor in Karlsruhe. This wire of 0.6 mm diameter was mounted in a bundle of 260 wire pieces having 5 cm individual length. All other wire samples were studied by X-ray diffraction (Cu K_α radiation). This analysis requires

Preparation Technique	Composition	Filament Diameter
Bronze (Vacuumschmelze)	Bronze/Nb = 2.5	~ 3 µm
"In Situ"	Cu-25Nb-14Sn	$\lesssim$ 0.2
	Cu-30($Nb_{.90}Ta_{.10}$)-11Sn	$\lesssim$ 0.3
Cu matrix	Cu-25Nb-13Sn	~ 0.4
	Cu-25($Nb_{.93}Ta_{.07}$)-12Sn	~ 0.3
	Cu-25($Nb_{.96}Hf_{.04}$)-13Sn	~ 0.3
	Cu-25($Nb_{.96}Hf_{.04}$)-13($Sn_{.78}Ga_{22}$)	~ 0.5
Powder Metallurgy	Cu-35Nb-20Sn	~ 0.5

Table I: Different wires analyzed in the present investigation (all compositions wt.%). The bronze wire was reacted at 700°C, all other wires between 2 and 5 days at 650°C.

only small amounts of sample but the surface exposed to the X-ray beam must be even. This was obtained by rolling the Sn coated wires of originally 0.25 mm diameter into tapes of 100 µm thickness prior to reaction. These tapes were then disposed in the sample holder of 12 x 8 mm size. They were pressed into a soft paste which not only held them in place without introducing additional stresses, but also enabled to obtain a well-

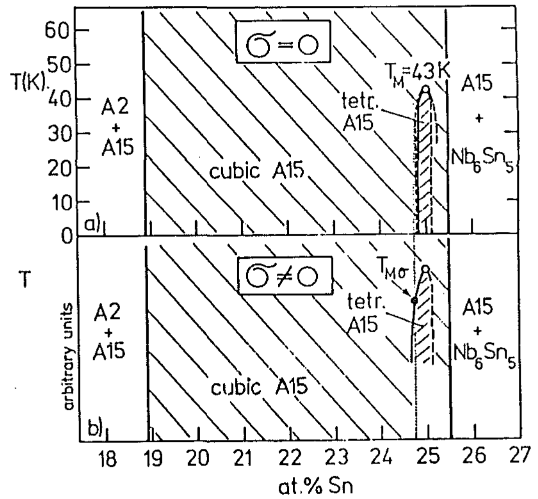

Fig. 1: The low temperature phase diagram of Nb-Sn at zero pressure (a) and in the state of precompression $\sigma \neq 0$ (b). (a) reflects the situation in bulk samples, (b) in multifilamentary composite wires with filament diameters well below 1 µm, formed after 3 days at 650°C. A slight widening of the tetragonal phase field at $\sigma \neq 0$ leads to a compression-induced phase transformation (vertical dotted line). For $\sigma \neq 0$, $T_{M\sigma} > T_M$ was found. In this figure, the c/t phase limit has been temptatively drawn: it merely describes the onset of the transition.

defined, nearly even surface. A Thor low temperature diffraction camera with a flow cryostat, mounted on a Philips generator was used, the sample temperature being controlled by a He gas stream.

It has to be noted that the behavior of the filaments in a tape is not exactly the same as in a wire, as follows from the anisotropy of J_c in tapes recently reported by Bevk et al..[12] In powder metallurgical and Cu matrix processed wires the bronze at the surface had to be partly etched away in order to limit the absorption of X-rays. This may alter somewhat the stress distribution near to the surface, but the effect on the $c \rightarrow t$ transformation was found to be negligible (c stays for cubic, t for tetragonal). This problem is not encountered in "In Situ" wires which can be analyzed in the as-reacted state.

The results obtained above on tapes and wires have been compared to those of bulk Nb-Sn samples melted by electromagnetic levitation in a Cambridge crucible[13] under a high argon pressure, followed by a homogenization heat treatment at high temperatures.[14]

III. Results

1) Nb-Sn low temperature phase diagram at $\sigma = 0$

In order to understand a mechanism based on a phase transformation, the knowledge of the phase diagram of the investigated compound is essential. The starting point of the present investigation was thus the determination of the low temperature relationships in bulk Nb-Sn, by applying the phase rules on the $c \rightarrow t$ phase transformation, which is the first order.[9,10] An attempt was then made to draw the Nb-Sn phase diagram under precompression, as it results from the wire data. Since the precompression involves non-hydrostatic components of stress[11], this diagram may be different from that determined under hydrostatic conditions, but reflects the actual situation in multifilamentary wires and is thus useful for the understanding of stress effects on J_c.

The principles applied for the present determination of the low temperature Nb-Sn phase diagram are based on the phase rules and were recently applied to the low temperature phase diagram of the system $Cu_xMo_6S_8$, which is even more complex.[15] According to Mailfert et al.[16], Vieland[17] and Chu[18], the tetragonal phase at zero stress forms at $T_m = 43$ K. King et al.[9] and Vieland et al.[10] demonstrated that the tetragonal phase forms by a first order phase transformation. From the specific heat data of Junod et al.[19] on Nb_3Sn samples of different compositions and with different degrees of transformation, it follows that the tetragonal phase has invariantly a sharp superconducting transition, the width being of the order of 0.4 K. Recent data of Devantay et al.[14] show a linear variation of T_c of ~1.85 K/at.% Sn in the cubic A15 phase field. Assuming a similar variation for the tetragonal phase, the width of its phase field is estimated to be less than 0.2 at.%. There are two ways how the tetragonal phase can be formed by a first order phase transformation at 43 K, either by a congruent formation from the solid cubic A15 phase or by a peritectoidical formation. From the analysis of two bulk samples with 24 and 24.4 at %Sn, described in detail by Devantay et al.,[14] if follows that in both cases the dominant phase at 10 K is the cubic phase, thus excluding the peritectoidical formation. All these observations lead to the low-temperature Nb-Sn phase diagram plotted in Fig. 1a. The two-phase region c+t, characteristical for a first order phase transformation, is temptative.

2) Precompression induced cubic tetragonal phase transformation

We have found a precompression-induced phase transformation in multifilamentary Nb_3Sn wires, prepared either by the "In Situ", the powdermetallurgical or the Cu matrix technique. All these techniques are based on the external Sn diffusion process, the reaction conditions being 3 days at 650°C. The phase transition was detected by comparing the 400, 420, 520/421, 521, and 440 peaks at 300 K and 10 K. In practice, the peak (400) is the simplest to study by virtue of its reasonable intensity, absence of degeneracy in $\Sigma(h^2+k^2+l^2)$ and sufficient resolution of the tetragonal splitting. In addition to the line splitting, the cubic-tetragonal transformation is marked by a line broadening and a lowering of the peak intensity.

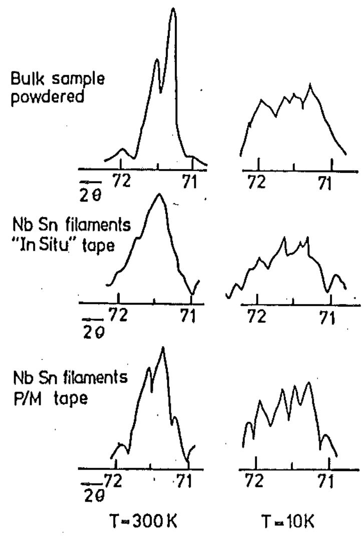

Fig. 2: The (400) X-ray diffraction line at 300 K and 10 K for the cases of bulk Nb_3Sn, "In Situ" and powdermetallurgically (P/M) processed Cu-Nb_3Sn tapes. In all three cases, a cubic-tetragonal low temperature phase transformation is observed. The oscillations are statistical and not correlated to line splitting due to the transformation.

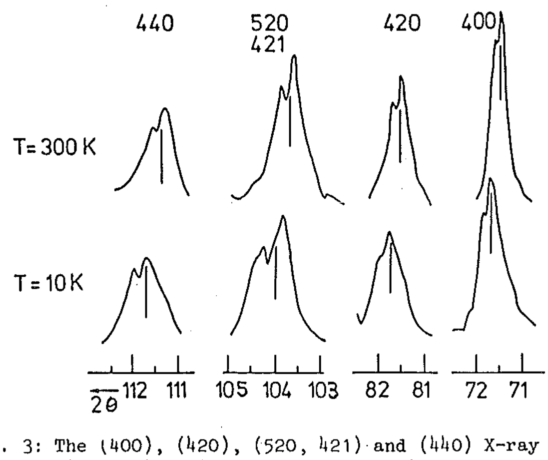

Fig. 3: The (400), (420), (520, 421) and (440) X-ray diffraction lines of the Nb_3Sn filaments of a Cu-25 wt.%Nb-14 wt.%Sn wire after etching away the bronze matrix. No cubic-tetragonal transformation is observed down to 10 K.

As shown in Fig. 2, the shape of the (400) peak of the Nb_3Sn filaments under precompression is strongly affected by the low temperature phase transformation. After etching away the bronze matrix, the Nb_3Sn filaments remain cubic down to 10 K. This is shown in Fig. 3 for the case of the "In Situ" wire initially produced by levitation melting[22] having the composition Cu-25 wt.% Nb-14 wt.% Sn. The onset of the transition temperature T_M is considerably increased with respect to that of unstressed Nb_3Sn: values $T > 60$ K were found. A detailed description of the variation of T_M will be given elsewhere.

Chu[18] has shown that T_M of Nb Sn is enhanced by a high hydrostatic pressure ($T_M = 46.5$ K at $1,8 \cdot 10^9$ N/m^2 (18 kbar)). More recently, the same author[20] observed in V_3Si a pressure-induced phase transformation at $2,9 \cdot 10^9$ N/m^2. Fasol et al [21] also observed a pressure-induced phase transformation in V_3Si, but at $4,5 \cdot 10^9$ N/m^2. A simple extrapolation of Chu's data shows that hydrostatical pressures higher than $5 \cdot 10^9$ N/m^2 would be required to get $T_{M\sigma} > 60$ K and T_c values around 16.5 K as obtained in our wires. This is in agreement with recent results of Thorwarth et al.[29] However, the maximum calculated pressure exerced by a bronze shrinkage leading to $\epsilon_m = 0.7\%$ is only of the order of $5 \cdot 10^8$ N/m^2.

This is an indication for the important role of the shear stresses (of smaller magnitude) in stabilizing the tetragonal phase. It may be added that the shear stresses are certainly enhanced by the irregular tape size of the filaments due to the texturing during the deformation process.

3) The low temperature Nb-Sn phase diagram under precompression ($\sigma \neq 0$)

A temptative low temperature Nb-Sn diagram under precompression ($\sigma \neq 0$) is drawn in Fig. 1b. Since the degree of precompression and thus the value of $T_{M\sigma}$ may vary from wire to wire, this diagram is only schematic, and is based on the following data:

a) Like for hydrostatic pressure, precompression leads to values $T_{M\sigma} > T_M = 43$ K.

b) Since the unstressed Nb_3Sn in our samples after etching does not transform, the effective Sn content must be below 24.8 at.%, the Nb rich limit for the tetragonal phase at $\sigma = 0$. Precompression thus induces a bradening of the tetragonal phase field.

c) The inductively measured T_c value after etching, 17.6 K, indicates that the maximum Sn-rich limit under precompression reaches ~ 24.7 at.%, using the T_c vs. Sn content relationship of Devantay et al.[14]

d) It is not known how the stresses influence the two phase region c + t. In Fig. 1b, the limit is not drawn, since is not at all certain that the transformation is still of the first order. It has indeed been shown by Ullrich et al.[23] that V_3Si undergoes a first order transformation at zero pressure, but a second order phase transformation at $\sigma \neq 0$.

A comparison with the line distribution of a fully transformed bulk sample, added in Fig. 2 shows that for "In Situ" wires the majority, for powdermetallurgical wires perhaps even the totality of the filament volume is tetragonally transformed. As shown by neutron diffraction[24], only a small part of the analyzed bronze wires are transformed.

4) The influence of Ta and Hf additions

Ta additions to the Nb, 7 wt.% with respect to the Nb (Cu matrix method) and 10 wt.% with respect to the Nb ("In Situ" method) were found to suppress the formation of the tetragonal phase. A Cu matrix processed wire sample with 4 wt.% Hf with respect to the Nb was found to transform partially (~ 30%), but the simultaneous addition of 3 wt.%Ga with respect to the copper again suppresses the tetragonal phase.

The reason why these additions suppress the tetragonal phase is unclear at present. A slight deviation of the formed A15 phase from stoichiometry is sufficient to explain this effect. Further investigations are actually in progress in our laboratory in order to elucidate this question.

5) The cubic-tetragonal transformation in wires under uniaxial stress:a model

The present results permit to formulate a model for the phase balance in the filaments of a multifilamentary wire, shown in Fig. 4. The ratio V_c/V_c+V_t, where V_c is the amount of cubic phase in the volume and V_t the corresponding amount of tetragonal phase, changes as a function of increasing stress, the sequence being $t \rightarrow c \rightarrow t$. Two mechanisms can be invoked in order to

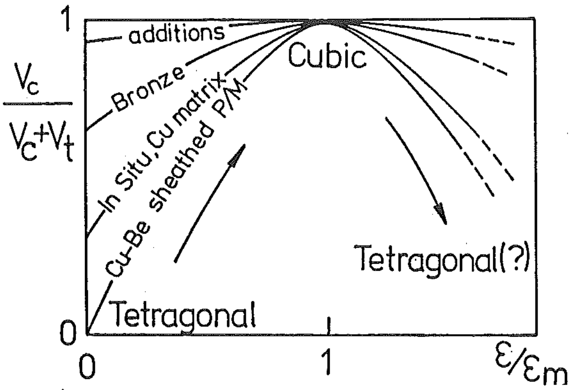

Fig. 4: A model for the effects of uniaxial tensile stress on the crystallographic structure of multifilamentary wires prepared by different techniques. V_c and V_t represent the volume of cubic and tetragonal phase respectively.

explain this gradual change of V_c/V_c+V_t under varying stress conditions: a) by a shear stress gradient in the Nb_3Sn filament, b) by the change of the two-phase region c + t passing from Fig. 1a ($\sigma = 0$) to Fig. 1b ($\sigma \neq 0$).

If the state at ϵ_m in the wire is really a stress-free state, comparable to our filaments after etching away the bronze matrix, the Nb_3Sn phase will be cubic. If we assume a tridimensional stress distribution[8], a certain amount of shear stress is even present at $\epsilon = \epsilon_m$, but this has little influence on the proposed model.

As indicated in Fig. 4, we found that the amount of tetragonal phase in the precompressed state increases in the sequence a) Ta or Hf+Ga additions, b) bronze processed wires,[24] c) "In Situ" and Cu matrix wires, d) powdermetallurgical wires with a Cu-Be jacket.

6) The ratio J_{cm}/J_{co} and the cubic tetragonal transformation

We will now try to explain the different ratios J_{cm}/J_{co} in multifilamentary wires prepared by different techniques in the light of the transformation model described above. The basis of our discussion lies in the different values of $H_{c2}(0)$ of the cubic Nb_3Sn A15 phase, 30 T, and of the tetragonal phase, 24 T.[25] The discussion is restricted to applied fields above 12 T, where the critical current is mainly determined by the upper critical field, and possible microstructural differences between the cubic and tetragonal phase are less important for the pinning mechanism.[30] Following this model, the increase of J_c in Nb_3Sn multifilamentary wires with uniaxial tensile stresses is due to a gradually increased amount of cubic phase. This is confirmed by the following arguments:

a) The J_c values at high fields of wires with nontransforming (cubic) filaments as obtained by additions of Ta[22,27] or Hf+Ga[28] are known to be considerably higher than those of wires without additions (tetragonal).

b) At high fields, the slope dJ_c/dB_o of a Nb_3Sn "In Situ" wire containing 10 wt.% Ta with respect to Nb is considerably smaller than that of the corresponding wire without Ta [22], thus confirming results of Suenaga et al.[27] on bronze processed $(Nb, Ta)_3Sn$ wires. The smaller slope dJ_c/dB_o at the same field, B_o, simply reflects the smaller B_o/H_{c2} ratio for the cubic phase.

c) The ratio J_{cm}/J_{co} for the nontransforming "In Situ" wire Cu-30 wt.%$(Nb_{90}Ta_{10})$-11 wt.%Sn is only 1,45 at 16 T, independent from the applied field.[22]

d) If a tensile stress is applied to Nb_3Sn multifilamentary wires, the slope J_c vs. B_o for a given value of B_o increases, as shown by Foner et al.[26] on "In Situ" wires and by Flükiger et al.[4] on powdermetallurgical wires (Fig. 5). This reflects the increasing amount of cubic phase.

e) As recently shown by Seibt[31], the ratio J_{cm}/J_{co} for Nb_3Sn bronze wires (at 12.3T) at 2.2K is higher than that obtained at 4.2 K. Here, the change in the upper critical field, $\Delta H = H_{c2}(2.2\ K)-H_{c2}(4.2K)$ is higher for the cubic phase (2.2T)[25] which is sufficient for explaining the observed increase in J_{cm}/J_{co}.

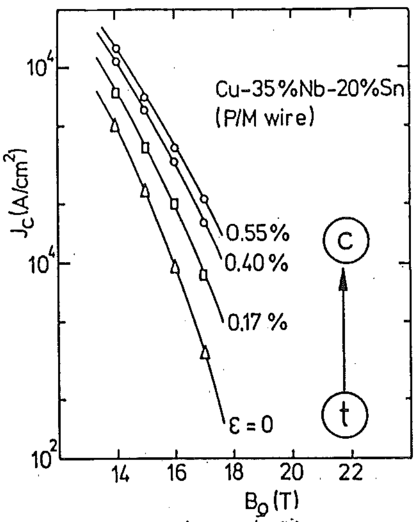

Fig. 5: The change of slope in a P/M wire of the composition Cu-35wt.% Nb-20wt.% Sn for different strain values, reflecting the gradual transformation into the cubic phase approaching ε_m. These values are extracted from Ref. 4.

IV. Conclusion

It was found that the ratio J_{cm}/J_{co} in Nb_3Sn multifilamentary wires is strongly correlated to the volume ratio between the cubic and the tetragonal phase, which in turn depends on the state of precompression. A model based on these results has been established which qualitatively describes the most important aspects of the behavior of J_c of Nb_3Sn multifilamentary wires as a function of the applied uniaxial stress. The very small ratios J_{cm}/J_{co} for nontransforming wires are of a great technical importance, in particular for large scale applications at high fields: very strong supporting structures with high Young's modulus and large linear thermal expansion coefficients can now be used in combination with nontransforming Nb_3Sn wires (obtained by a great variety of additions to the Nb and to the bronze): this combination drastically reduces the precompression (or prestress) effects on J_c.

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