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Advanced Superconducting Materials

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Abstract

The superconducting properties of various materials are reviewed in view of their use in high field magnets. The critical current densities above 12 T of conductors based on NbN or $PbMo_6S_8$ are compared to those of the most advanced practical conductors based on alloyed Nb_3Sn. Different aspects of the mechanical reinforcement of high field conductors, rendered necessary by the strong Lorentz forces (e. g. in fusion magnets), are discussed.

Zusammenfassung

Fortgeschrittene supraleitende Materialien

Die supraleitenden Eigenschaften von verschiedenen Materialien werden diskutiert im Hinblick auf deren Verwendung in Hochfeldmagneten. Die kritischen Stromdichten der Supraleiter (Zr, Hf)V_2, NbN und $PbMo_6S_8$ bei Magnetfeldern über 12 T werden mit denen der zur Zeit fortgeschrittensten praktischen Supraleiter, Nb_3Sn mit Zusätzen, verglichen. Die vorliegende Arbeit umfaßt auch verschiedene Aspekte der mechanischen Verstärkung von Hochfeldsupraleitern, die wegen der starken Lorentzkräfte (z. B. in Fusionmagneten) notwendig wird.

I - INTRODUCTION

Although no new high Tc material was found after the discovery of the rhombohedral compound $PbMo_6S_8$ (Tc = 15K) nearly 10 years ago /1/, there is no stringent or definitive argument excluding the existence of new ternary or quaternary materials with T_c values close to 20K (or even higher). A possible limitation towards higher T_c values arises from theoretical arguments considering the strong electron-phonon coupling, which suggest a "saturation" of λ, the electron-phonon interaction parameter /2/. At present, the search for new superconductors has been extended to ternary, quaternary and even multinary compounds, including also nonmetallic elements as sulfur, carbon, nitrogen, oxygen and others /3/.

The wealth of the actual research in the field of superconductivity shows a pronounced trend toward applications, dictated by industrial requirements. The most successful application for industrial superconductors is actually doubtless represented by the full-body NMR magnets for clinical diagnosis, based on NbTi. For this purpose, mono- or multifilamentary NbTi wires must satisfy severe homogeneity requirements. They are produced by classical procedures, but do not need further improvements, the produced fields being always below 2T. There are other fields where the use of advanced superconductors is required: a) high field magnets, either for laboratory use or for future fusion applications, and b) superconducting microelectronics (Josephson computer). The present survey will be restricted to high field conductors, with a particular emphasis on emerging materials as $(Zr,Hf)V_2$, $PbMo_6S_8$ and NbN. A particular aspect of A15 conductors will also be treated, i.e. the behavior of J_c in mechanically reinforced wires, able to retain the strong Lorentz forces acting in fusion magnets.

II - COMPETING SUPERCONDUCTING MATERIALS

The materials which could hypothetically be used in superconducting wires (Table I) can be subdivided in three classes, following their values of the upper critical field, $B_{c2}(0) \leq 16T$, $16T \leq B_{c2}(0) \leq 30T$ and $B_{c2}(0) \geq 30T$ (No B_{c2} values could be found in the literature for the compound $Y_{.7}Th_{.3}C_{1.58}$, synthetized under high pressure/30/).

Compound	Structure	State of the Sample	Radiation Damage	Stress Sensitivity	Thermal Equilibrium	T_c (K)	γ (mJ/atgK^2)	$B_{c2}(0)$ (T)	Ref.
NbTi	A2	M	No	No	Yes	10.1		14	4
Nb-Ta-Ti	A2	M	No	No	Yes	9.0		16	5
VTi	A2	M	No	No	Yes	7		12	6
V-Ta-Ti	A2	M	No	No	Yes	6.5		16	7
ZrV_2	Laves	B	No	No	Yes	8.7			8
$(Zr,Hf)V_2$	(cub.)	M			Yes	9.7		21(4.2K)	8,9
Nb_3Sn	A15	B	Yes	Yes	Yes	18.0	13.2(60%c)		10
		B						∼30(c)	11
		B					13.0(t)		12
		M						20÷26(t)	11,13
Nb_3Sn + additions	A15	M	Yes	Yes	Yes	up to 18.2		25(4.2K)	14,42
V_3Ga	A15	B	Yes	Yes	Yes	15.9	24	24	15
Nb_3Ge	A15	S,C	Yes	Yes	No	23			16
		S				22.5		38	17
		V				20.8	7.58		18
Nb_3Al	A15	B	Yes	Yes	No	19.1			19
		B				18.4	9.0		18
		B				18.0		33	17
$Nb_3(Al,Ge)$	A15	B	Yes	Yes	No	20.7			20
		B				20.0	8.75		18
		B				21		44	17
		Q							21
$NbN^{+)}$	B1 (cub.)	B	No	Yes	No	15.7	2.04	8.5	22,56
		S				15.8		28	23
		S				16		35 to 50	24,55
		V				17		22(4.2K)	26
		S				17.8		16	25
		S				16		22	25
$PbMo_6S_8$	Rhombohedral	B	Yes	?	Yes	15	5	∼60	27
		M				13.4		∼50	28
		S				14.6			29
$Y_{.7}Th_{.3}C_{1.6}$	Pu_2C_3(c)	B	?	?	No	17	1.81	?	30

Table I. Superconducting properties of various intermetallic compounds after different preparation modes: B = bulk, M = multifilamentary, S = sputtered, C = coevaporated, V = CVD, Q = quenched from the liquid state and retransformed. The sensitivity to high enrgy irradiation is indicated, as well as the occurrence of equilibrium. +) NbN is stabilized by C or O: This symbol is used here for Nb(N,C,O).

A. $B_{c2}(0) \leq 16T$

The compounds NbTi, Nb-Ta-Ti, VTi and V-Ta-Ti are ductile, in contrast to all other materials listed in Table I, but exhibit relatively low T_c values ($T_c \leq 10K$). The highest values of $B_{c2}(0)$ for the ternaries Nb-Ta-Ti and V-Ta-Ti reach 16T /5,7/. The adequate operation range for magnets based on these conductors is thus 1.8K, where the corresponding values of B_{c2} are close to 15T.
Multifilamentary wires based on these materials are prepared by a succession of extrusion and wire drawing steps without intermediate annealings. The highest J_c values being obtained after the greatest amount of cold working. The recent progress with hydrostatically extruded NbTi /31/ (instead of the ordinary hot extrusion) confirms the beneficial effect of further cold working, which is thought to act on microstructural properties.

B. $16T \leq B_{c2}(0) \leq 30T$

This class of materials includes the ternary compound $(Zr,Hf)V_2$ crystallizing in the cubic Laves phase, C15, and the A15 type compounds Nb_3Sn (with and without additions) and V_3Ga. In both cases, multifilamentary wires are obtained by a diffusion reaction at the end of the plastic deformation, at temperatures at the vicinity of 700°C.

A15 phases at equilibrium. At 20T, V_3Ga still has higher J_c values than Nb_3Sn, in spite of the improvements achieved in the last years by introducing the additions Ti/32/, Ta/33/ or Ni + Zn/34/, which led to overall values of J_c = 1 X 10^4 A/cm² at 19T (Fig.1). This value of J_c at a field B_0 is generally considered as the necessary criterion for reaching B_0 in a magnet wound with the same conductor: The construction of small laboratory magnets reaching 19 or even 20 T on the basis of V_3Ga or alloyed Nb_3Sn can thus positively be envisaged. The improvement of J_c at high fields in alloyed Nb_3Sn wires is mainly correlated to an enhanced value of B_{c2}, as a consequence of the enhanced electrical resistivity, ϱ. A further progress could be reached by acting on the pinning mechanism, as suggested by the recent success of the powdermetallurgical ECN technique/35/: The high Sn reserve arising from $NbSn_2$ leads here to particularly high growing rates, thus causing a considerable enhancement of J_c up to 15T (see Fig. 1).

Laves phases. The superconducting properties of Laves phase compounds are relatively insensitive to irradiation with high energy particles /35/, which could be a decisive advantage over A15 or rhombohedral compounds in view of applications in future fusion magnets. Two approaches for producing multifilamentary $(Zr,Hf)V_2$ wires have been reported. The first one is based on the retransformation of amorphous $(Zr_{.7}Hf_{.3})_{.60}V_{.40}$ ribbons produced by rapid quenching /8/ into the C15 phase by a subsequent anneal of 72 hours at 600°C. It is thought that the melt spinning process to produce continuous ductile ribbons of amorphous Zr-Hf-V would be promising.

The second approach for producing multifilamentary $(Zr,Hf)V_2$ conductors by a diffusion reaction has already been developped to a level where an industrial production of long lengths of material seems possible. By the composite process, Kuroda et al./9/ have prepared wires containing 1634 $(Hf,Zr)V_2$ filaments. Groups of 7 or 19 Zr-Hf rods containing up to 40 at.% Hf were inserted in a V - 1 at.% Hf matrix and cold drawn, with intermediate anneals at 800°C after 50% area reduction. After several steps, including cutting, packing and further cold drawing, the composite wire was inserted into a Cu tube, drawn to the final diameter and reacted at temperatures between 850 and 1000°C to form Laves phase filaments. Since components of matrix and core contribute to the formation of the C15 layer, the layer formation rate will essentially show no saturation, in contrast to Nb_3Sn (or V_3Ga), where the depletion of Sn (or Ga) in the bronze matrix causes a saturation.

As shown in Fig. 1, the overall critical current densities of such wires are appreciable: The wire characterized by Zr - 40 at.% Hf, reacted 30 hours at 950°C exhibits a J_c value of 1 X 10^4 A/cm² at 14.3T and 4.2K (in the layer: J_c = 7 X 10^4 A/cm²).

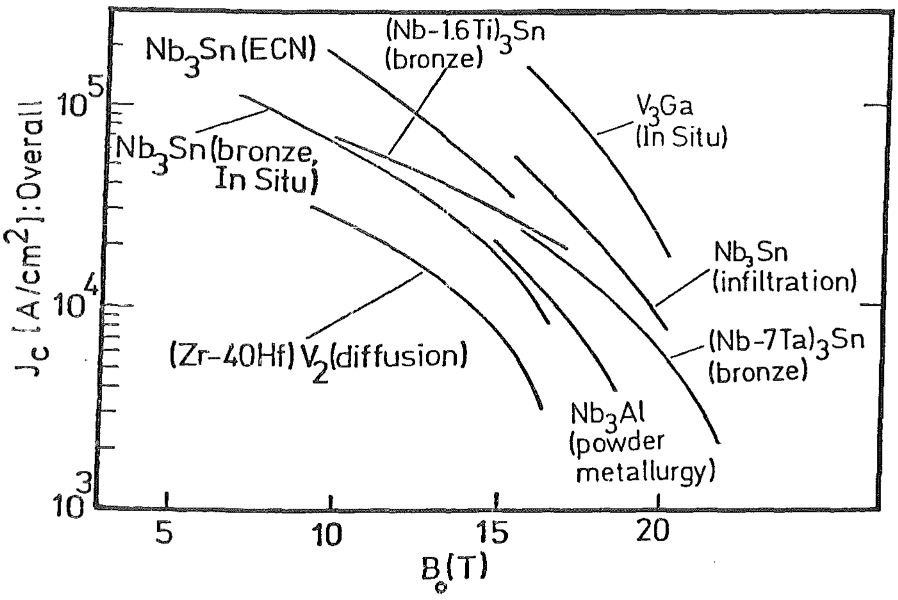

Fig. 1

Overall critical current densities for multifilamentary wires based on different materials, prepared by different methods:
Nb_3Sn(ECN/35/), Nb_3Sn(bronze /52/), Nb_3Sn(In Situ/53/), Nb_3Sn(infiltration/50/), V_3Ga (bronze/54/), Nb_3Al(powder metallurgy/48/), $(Nb-1.6Ti)_3Sn$ (bronze/32/), $(Nb-7Ta)_3Sn$ (bronze/34/), $(Zr-40Hf)V_2$ (composite diffusion/9,42/).

By optimizing the heat treatment conditions, the packing ratios and the Hf content in the rods, Inoue et al. /42/ recently showed that (Zr, Hf)V_2 wires may exhibit even higher J_c values lying very close to those of unalloyed Nb_3Sn wires.

C. $B_{c2}(0) > 30T$

Even in the case of future improvements on J_c for the wires discussed in the paragraphs A and B, the limited value of $B_{c2}(0)$ (or better, $B_{c2}(4.2K)$) constitutes a stringent limitation. Other A15 type compounds, as Nb_3Al, Nb_3Ga, Nb_3Ge, or $Nb_3(Al,Ge)$ are known to exhibit $B_{c2}(0)$ values above 30T, while for NbN and $PbMo_6S_8$, values above 50T have been reported (see Table I). Unfortunately, these high $B_{c2}(0)$ values correspond either to bulk or non equilibrium states of the respective materials and not to the required multifilamentary state. For each one of these materials, characteristical difficulties are encountered when preparing multifilamentary wires, but there is no doubt that in the future, one or another will be available in the multifilamentary configuration with $B_{c2}(0)$ values approaching those listed in Table I.

Nonequilibrium A15 type systems. In the case of A15 type systems with $B_{c2}(0)$>30T, two main difficulties are encountered when comparing with Nb_3Sn or V_3Ga: i) the stoichiometric composition is metastable (for a review of the corresponding phase diagrams see Ref. 41) and ii) the diffusion reaction starting from the Cu bronze is not applicable. Thus, all approaches to prepare multifilamentary wires based on materials with $B_{c2}(0)$>30T will have to start from a nonequilibrium state. The stoichiometric composition can be reached by CVD (Nb_3Ge /18/), coevaporation (Nb_3Al /37/, Nb_3Ge /16,13/), sputtering (Nb_3Ge /16/) or quenching from the liquid state. The latter is particularly interesting, since it allows to retain the alloy in a ductile state, e.g. bcc /39,40,41/ or amorphous /42/. In the case of Nb_3Al, the bcc phase was retained at compositions above 22 at.%Al, either by liquid quenching /39/ or by splat cooling /40/ After retransformation at 900°C, Bevk et al. /40/ obtained extremely high J_c values, 2.2 X 10^5 A/cm^2 at 20 T, demonstrating the potential for Nb_3Al. A bcc phase with such high Al contents being not ductile enough for extended plastic deformation needed for industrial applications. A significant improvement was recently realized by Togano et al. /21/, who were able to produce amorphous $Nb_3(Al,Ge)$ ribbons by splat cooling on a Cu surface heated at 600°C. The (still unresolved) problem is now the same as in the amorphous (Zr,Hf)V_2 ribbons described above /8/: The production of long lengths of homogeneous amorphous ribbons by melt spinning, as a basis for multifilamentary wires. Another way for obtaining A15 phase filaments in a nonequilibrium state was discovered by Ceresara et al. /43/ on Nb3Al. If alternate layers of Nb and Al of less than 1 μm thickness are formed, a reaction between 700 and 900°C leads to an A15 phase containing 22 at.% Al., i.e. 1.5 to 2 at.%Al above the equilibrium value /41/. The mechanism leading to this favourable shift in composition is not yet understood, but all approaches in fabricating Nb_3Al wires reported in the meantime are based on this process. A spectacular one is the powdermetallurgical approach, first reported by Larson et al. /44/: A mixture of Nb and Al powders is "mechanically alloye[d]" by means of high energy ball milling and subsequently drawn to fine wires and reacted. Later on, the "cold powdermetallurgical" processing, developped by the author /45,51/ for producing ultrafine Nb_3Sn filaments was extended to Nb_3Al /46,47/ and was recently considerably improved by Thieme et al. /48/ (see Fig. 1). This method is still under progress.

$PbMo_6S_8$. The possibilities of powdermetallurgical processing in preparing superconducting multifilamentary wires with $B_{c2}(0)\geq 30T$ are far from being completely explored. As illustrated by the work of Seeber et al. /28/ on $PbMo_6S_8$ wires, prepared using prereacted powders of the rhombohedral phase. This method is possible since the rhombohedral Mo chalcogenides are relatively soft, with a Vickers microhardness of 150kg/mm^2 /49/, which is considerably lower than that of Nb_3Sn, ~ 1000 kg/mm^2. The method can be described as follows: A Mo tube is filled with $PbMo_6S_8$ powders with sizes ≤20 μm and is hot drawn to a compact wire of 0.3 mm diameter. T_c is considerably lowered during the deformation, from 13.8 to 8K, and must thus be recovered by a final annealing at temperatures ranging from 800° to 1000°C, the annealing time varying between 10 and 100 hours. A reaction between the $PbMo_6S_8$ and the external tube wall during the recovery annealing must be avoided, which explains the choice of Mo as external tube material. In order to get a better compaction during the cooldown to 4.2K, a stainless tube was placed outside the Mo. The critical current density of such a wire at 6.5T and 4.2K was $J_c = 3 \times 10^3 A/cm^2$ /28/. This value is obviously one or two orders of magnitude smaller than the expected value in $PbMo_6S_8$, due to cracks and insufficient compaction, as demonstrated by J_c measurements on hot pressed, bulk $PbMo_6S_8$ samples with grain sizes <5 µm (see Fig. 2). A tendency toward considerably higher J_c values for submicron grain sizes can be recognized (grain boundary pinning), leading to the extrapolated values represented by the dotted line in Fig. 2. It appears, however, that the production of $PbMo_6S_8$ wires with submicron sizes is precisely the main difficulty. Indeed, not only the rhombohedral Mo chalcogenides are very stable, with melting points 1500°C /49/, but the tendency to form large grains is evident: sizes of > 100 µm can be easily obtained as the product of the sintering reaction at 1000°C. The strong influence of the grain size on J_c is confirmed by the work of Hamasaki et al. /29/ on thin $PbMo_6S_8$ films with 1 µm grain size: about 5×10^4 A/cm^2 at 8T and

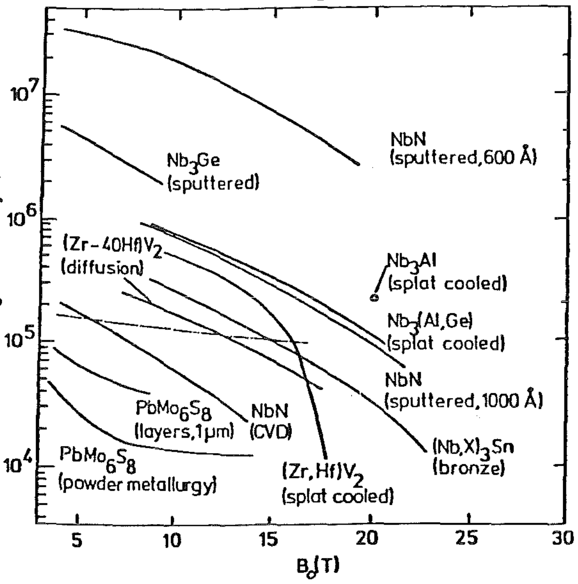

Fig. 2.
J_c vs. B_0 for the layer of superconducting material in wires or tapes prepared by different methods: $(Nb,X)_3Sn$ /32,34/, Nb_3Ge /38/, $Nb_3(Al,Ge)$ /21/, $(Zr,Hf)V_2$ /9/, $PbMo_6S_8$, powders, 5 µm /28/, layers, 1 µm /29/, NbN, sputtered, 600 and 1000 Å /24/, CVD /26/. The dotted line represents an extrapolated upper limit for $PbMo_6S_8$ /28/.

4.2K were reported. Thus, the success of future $PbMo_6S_8$ wires will depend on the ability of further reducing the grain size. A very stimulating point is the very high value of $B_{c2(0)}$, ~ 50T for the sample analyzed in Ref. 28: the curve J_c vs. B_0 decreases very slowly in the range between 8 and 14T. Thus, an increase of J_c in the layer by a factor of 5 would be sufficient to exceed the values of the best A15 wires above 20T.

<u>NbN.</u> There are similarities between wires based on the Laves phase $(Zr, Hf)V_2$ discussed above and NbN wires: i) little or no radiation effects on T_c are observed /36/, and ii) J_c exhibits no stress dependence /23/ (NbN stays here for simplicity, while NbCN or other formulas are used in the literature). Among the numerous methods used so far for preparing NbN tapes or layers, only reactive sputtering and CVD will be selected for the present discussion. Most work has been performed on reactive sputtering /23, 24, 25, 55/ on a substrate, with Nb or (Nb+C) targets and (N_2 + Ar) or (C_2N_2 + Ar) gas. At very low deposition temperatures, the deposited NbN layers were found to be amorphous, no X ray diffraction lines being visible /24/. The B1 phase appears after deposition above 450°C /24/ or after subsequent annealing at T ≳450°C /23, 55/. The value of T_c varies between 16 and 17.8K, while the electrical resistivity is comprised in the range between 80 and 300 µΩcm /23,24,25,55/. Strong variations of B_{c2} have been observed as a function of the sputtering conditions, the main parameter being the B1 grain size. Like in $PbMo_6S_8$, grain boundary pinning seems to be the dominant mechanism. In ≥ 2 µm films, deposited at 900°C, B_{c2} values between 8.5 and 20T were found by Gavaler et al. /24/, the lower value being close to the NbN produced by a bulk diffusion process /56/.
The highest B_{c2} values are found in thin films (≤ 5000 Å) deposited at 450°C /24/: These conditions are a compromise between an extremely high B_{c2} /24/ and a pinning active columnar void microstructure. J_c values for 600 and 1000 Å film thicknesses (Fig. 2) demonstrate the potential of NbN. For the 600 Å film, an extrapolation of the critical data near T_c yields B_{c2} ~ 50T (see Table I). For NbN recrystallized from the amorphous state, B_{c2} around 35T were reported /23,55/. Thus, reactive sputtering allows to determine the ultimate limits of J_c at high fields in NbN wires, but there is still

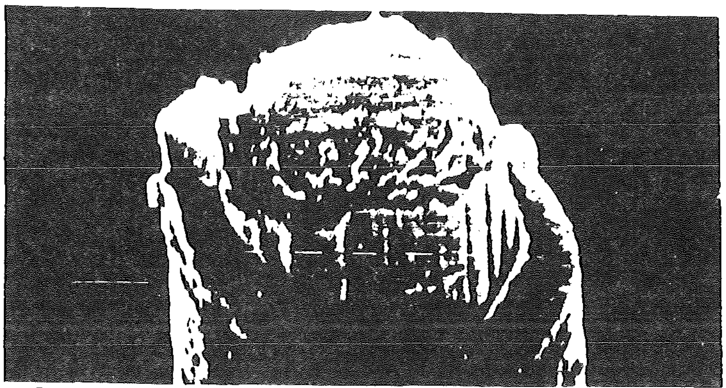

Fig. 3
Carbon fibre of 6.5 µm diameter, coated with a 1 µm NbN layer, and a very thin SiC interlayer. Bar length: 0.5 µm.
(Dietrich et al. /26/).

a long way to go up to a practical superconductor.
A further step in this direction is the deposition of NbN on carbon fibers by CVD and magnetron sputtering, by Dietrich et al. /26/, combining the mechanical strength of the carbon fibre and the superconducting properties of the layer. This method is rendered possible by the coating of a SiC layer on the carbon fibre prior to NbN coating, which considerably improves the adherence, thus avoiding cracks in the layer /26/. A B_{c2} value of 22T at 4.2K was reached so far by these methods. A typical J_c vs. B_o curve is shown in Fig. 2, while a carbon fiber, coated with SiC and NbN, is shown in Fig. 3. Further progress is expected from a lowering of the deposition temperature.

III - PRACTICAL CONDUCTORS SUBMITTED TO STRONG LORENTZ FORCES

An inherent problem of large high field magnets (e.g. fusion magnets) is the occurrence of strong Lorentz forces, requiring a reinforcement of the conductor by high strength materials which can be incorporated either prior to or after the reaction heat treatment. In the case of collective heating of the already assembled reinforced conductor (realized for example in the "cable in conduit" concept/57/), the large thermal expansion coefficient of steel, $\alpha = 16 \times 10^6 K^{-1}$, is expected to exert an enhanced precompression on the superconducting filaments, which have ordinarily α values between 5 and 7 $\times 10^6 K^{-1}$. The question whether and to what extent J_c is influenced by the reinforcing structure can be answered by studying the correlation J_c vs. ε. It is known/23,59/ that NbTi, NbN and $(Zr,Hf)V_2$ in the multifilamentary configuration are essentially unaffected by external mechanical stresses up to strain values where filament disruptures occur, i.e. $\varepsilon = 0.6\%$ and higher, depending on the superconducting material. For $PbMo_6S_8$, where no J_c vs. ε data are available, an influence of ε is not excluded if the strong degradation of T_c after deformation of the wires is taken into account. Strong strain effect on J_c have only been observed in wires based on A15 type compounds/59/. The effect of the enhanced precompression in reinforced Nb_3Sn wires has recently been studied/58/ and can be summarized as follows: i) ε_m, the strain value at which $J_c = J_{cmax}$, increases in unreinforced wires is shifted to values close to 1%, and ii) the ratio J_c/J_{cm} de- from 0.3 to 1% for reinforced wires. The negative effect on J_c can be counterbalanced either by additions to Nb_3Sn or by using reinforcing materials with lower α values, which both lead to an increase of B_{c2}. In the case of additions, B_{c2} is raised by the enhancement of the electrical resistivity, while low α materials attenuate the precompression on the filaments, which also leads to higher B_{c2} values. As shown in Fig. 4,

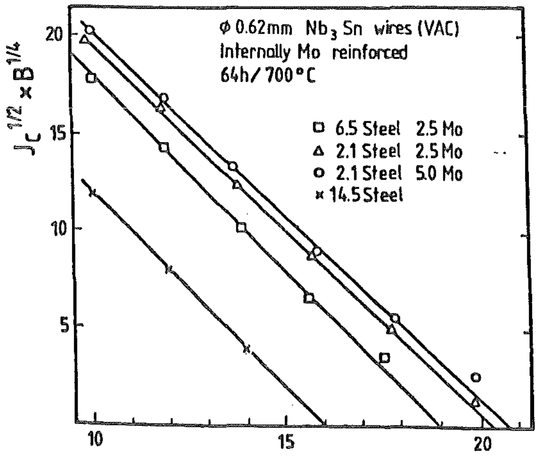

Fig. 4
Kramer plots for internally reinforced conductors base on the same VAC multifilamentary Nb_3Sn wire, with different combinations of stainless steel (high α) and Mo (low α material). As a consequence of the varying precompression, B^*_{c2} changes from 16 to 21 T (Flükiger etal. /58/).

the extrapolation of the Kramer plots shows continuously increasing values of B_{c2}^* when substituting stainless steel (high α) by Mo (low α). For a conductor based on the same multifilamentary Nb_3Sn wire, B_{c2}^* increases from 16 to 21T as function of the steel/Mo fraction. For fusion applications, it is of importance that the precompression in a reinforced conductor can be controlled by simply changig the ratio between the high α and the low α reinforcing material.

IV - CONCLUSION

Since no new high T_c materials could be found in the last decade, the development of advanced superconductors is now concentrated on the achievement of high J_c values at high magnetic fields on materials already known. This is mostly obtained by increasing the upper critical field (by optimization of the reaction heat treatment and/or by additions to the superconductor) or by reducing the precompression acting on the filaments. Setting a value of $J_c = 4 \times 10^4$ A/cm^2 in the superconducting layer as a criterion, it appears from Fig. 2 that superconducting magnets with fields up to 25T and more can be in principle expected in the future.

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