EXPERIMENTAL RESULTS ON Nb 25 WT.% Ta 45 WT.% Ti SUPERCONDUCTING WIRE

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Abstract. -Several small billets with NbTi binary and Nb 25 wt.% Ta 45 wt.% Ti ternary alloy filaments were made and fabricated into fine wire after subjecting the alloys to different treatment conditions. J_c results at 4.2 K and 1.8 K from binary and ternary materials, which were prepared under the identical conditions, are compared. The results of an α-Ti precipitation study on the two alloys are also presented. The J_c results are compared with those from some earlier work on a Nb 15 wt.% Ta 44 wt.% Ti [1] material.

I. INTRODUCTION

There has been a limited effort for some time to develop improved materials for operation in the field range 8-11 T for several applications, particularly special dipole and quadrupole magnets. Ductile materials such as ternaries based on the NbTi system, particularly those containing Ta, have considerable appeal if they can be shown to exhibit significantly improved J_c's in this field region.

Some of the earlier investigations have been referred to previously [1] and will not be reviewed again here. There has been apparent disagreement in the more recent literature [1,2] on how effective Ta is in increasing the J_c at 4.2 K and/or at 1.8 K.

In work reported by one of the authors in 1988 [1], the J_c properties of a ternary alloy of composition Nb 15 wt.% Ta, 44 wt.% Ti, were compared with those of the binary Nb 46.5 wt.% Ti. The two alloys were prepared in three different conditions prior to extrusion. These were: 1. as cold worked, 2. annealed at 800°C for 1.5 h, 3. heat treated for 40 h at 375°C. Four heat treatments, each of 40 h at 375°C were given after extrusion, to material in all the above conditions.

From this work it appeared that, while the cold worked NbTi material gave slightly improved properties when compared with the annealed, the difference was not great, particularly at higher fields. In the case of the 15 wt.% Ta ternary, however, it appeared that the J_c of the material annealed at 800°C for 1.5 h. was superior to that of the cold worked composite. When the material was heat treated for 40 h. at 375°C before the multifilamentary extrusion, the properties were reduced in the case of both the ternary and the binary. In all cases the ternary alloy appeared to be more sensitive to the various treatments than did the binary.

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Work has been reported [2] on a conductor developed for the Fermilab Low Beta Quadrupole requirements. Tab. I shows the actual data from Ref. 2 compared with that from Ref. 1 at 4.2 K. The results obtained in Ref. 2, while encouraging, were not superior to those of the binary (Col. 2 in Tab. 1), although the conductor was not optimized, as stated by the authors in the abstract. In the work for Fermilab, the ternary alloy was in the cold worked state before the multifilamentary extrusion. In the earlier investigation, [1] carried out on 50.8 mm diameter billets, while the average results were lower than those obtained from the larger billet for comparable heat treatments, the effects observed with cold worked monofilaments were similar. The optimum monofilamentary condition for the ternary, annealed for 1.5 h. at 800°C, (Col. 3 in Tab. 1), was not investigated in the Fermilab work. In the binary alloy the cold worked material is frequently superior to the annealed and this fact probably led the workers in Ref. 2 to omit an examination of the properties of the annealed material.

Unfortunately measurements of H_{c2}, T_c and the amount and size of the precipitate after the various treatments, were not reported in these earlier investigations. Work of this type has, however, now been carried out, particularly at the University of Wisconsin, and some results on microstructure-property relationships will be reported elsewhere at this conference [3].

Since Ta is a weaker β-stabilizer than Nb [4], when Ta substitutes for Nb, the alloy would be expected to behave as if it had a lower Nb % If this favors more α-phase precipitation, it will possibly lead to higher J_c's. We, therefore, decided to investigate a higher Ta containing alloy than that used in Ref. 1. The only alloy readily available was Nb 25 wt.% Ta, 45 wt.% Ti and this was therefore chosen, even though the reported H_{c2} and T_c for bulk material of this composition were not as high as had been reported for other alloys [5,6,7&8].

Table I. Comparison of J_c's at 4.2 K in Refs. 1 & 2

	J_c A/mm^2		
	Ref. 2 Ø 254 mm Cold work	Ref. 1 Ø 50.8 mm Cold work	Ref. 1 Ø 50.8 mm Annealed
5T			
NbTi	3225	2990	2860
NbTiTa	3110	2960	3250
8T			
NbTi	1380	1150	1165
NbTiTa	1050	975	1290

II. EXPERIMENTAL PROCEDURE

A Sample preparation

The alloys used in this work were Nb 46.5 wt.% Ti and Nb 25 wt.% Ta 45 wt.% Ti. They were obtained from NRC and were plasma melted. The ternary was cast into a 203 mm. diameter ingot and the binary into a 254 mm diameter one. These castings were cold worked to a rod size of 33.3 mm diameter and this was the condition in which they were purchased.

These materials were clad in copper and cold drawn down to 16.3 mm diameter. At this point three different treatments were employed for both the binary and ternary materials. These treatments were: an 800°C, 1.5 h. anneal (Ann), a 375°C heat treatment for 40 h. (HT) and the third was left as "cold worked" (CW). For cold worked and heat treated materials, sample rods were then drawn from 16.3 mm through several dies before they reached a core diameter of approximately 6.6 mm. The copper was chemically removed. Since there was no Nb barrier on the annealed material, it acquired a compound layer during the anneal at the rod size of 16.3 mm. At this stage the copper was removed and 1.5 mm was ground off the radius of the alloy to remove this reacted layer. The cleaned rod was then reclad with copper and drawn to a core size of approximately 6.6 mm.

Fourteen 6.86 mm diameter holes were drilled in a single circle in each of six 50.8 mm diameter billets. Fourteen 6.6 mm diameter rods were cut from each of the alloys in the three different conditions. These rods were each wrapped with Nb foil and inserted into the holes in the copper billets. The billets were sealed, welded and HIP'd at 580°C for 2 h. Conventional extrusion was then performed at 600°C and an extrusion area ratio of 16:1. After the extrusion, all the materials were subjected to the same series of heat treatments, i.e. 4 HT's at 375°C each for 40 h with a strain (ε) of 1.56 before the first heat treatment and 0.93 before each of the succeeding heat treatments. The wire was then drawn down and the J_c properties measured at a series of strains from 2 to 6 after the final heat treatment.

III. RESULTS

A. Critical current densities

The J_c values at 4.2 K were determined at 5T, 7T and 8T for the binary and the ternary in the three different conditions. The resistivity criterion for the measurements was 10^{-12} Ω·cm. The J_c values peaked in the strain range of 3.6-4.6 after the last heat treatment. These peak values were plotted as a function of field in Fig. 1. This shows that the 25 wt.% Ta ternary exhibits significantly lower J_c values than the 46.5 wt.% Ti binary under all comparable conditions after 4 HT's of 40 h. at 375°C. The three different treatments (CW, HT and Ann) affected the binary and ternary materials in different ways. In the case of the binary, the Ann material has the lowest J_c at the low fields. The HT and CW materials were very similar but the HT'd material showed slightly superior

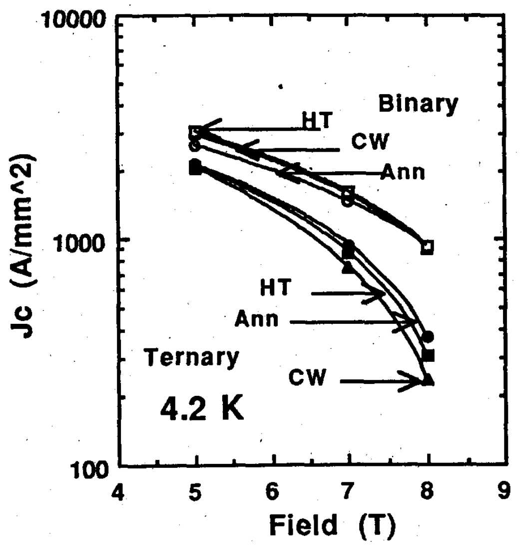

Fig. 1. Jc vs. Field (4.2 K) for binary and ternary alloys

results. At 8T the binary material in all conditions appeared to give the same J_c. In the case of the ternary, the annealed material showed the higher values as was the case with the 15 wt.% Ta alloy. The CW material had the lowest J_c's and the HT material was intermediate.

The J_c values at 1.8 K were determined at Yale University, in the range 2-7T for the binary and the ternary in different conditions. The results are shown in Fig. 2. While the ternary, in both the annealed and heat treated condition, shows similar properties to those of the binary in the annealed condition, the cold worked and heat treated binary materials were still significantly superior to the ternary materials

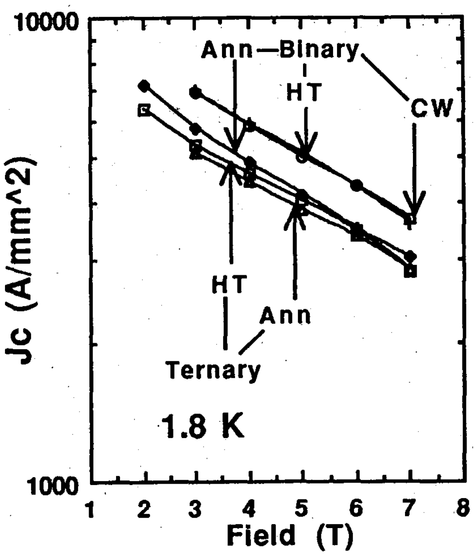

Fig. 2. J_c's vs. Field (1.8 K) for binary and tenary alloys

B. α-Ti precipitate Vol. % and particle size

While the 25 wt.% Ta alloy did not yield the hoped for J_c's after 4 HT's of 40 h. at 375°C, it was not known whether this was due to a lower H_{c2}, lower T_c, or a difference in the size or quantity of the α-phase precipitate. It was decided therefore, to measure the amount and size of the α-phase precipitate obtained in the materials which are in the various conditions. As the time available was short, we elected to use the recently perfected SEM-BEI technique rather than the more precise TEM approach [3]. The work was carried out at the University of Wisconsin. In order to ensure that the sizes to be measured were within the resolution of the SEM technique, the measurements were made immediately after last heat treatment and not at the wire size where the J_c peaked. At this heat treatment size, the α-phase precipitates are approximately equiaxed and 100-150 nm in diameter (Table II). During the drawing of the wire, the precipitates are transformed into ribbons whose thickness is assumed to obey the $d^{1.6}$ relationship outlined by Meingast et al [9]. This predicts that, at the peak J_c wire size, the precipitate thickness is approximately 6 nm, i.e. the same order as that of the coherence length (ξ[4.2 K] ~5 nm). Again, from Ref. 9, it is assumed that the H_{c2} and T_c of the material with this size of precipitate can be predicted from the bulk composition. It is not expected that H_{c2} and T_c would change significantly as a result of the precipitation of a high Ti α-phase.

The results in Table II show that the ternary alloy produces more precipitate than does the binary despite its lower J_c. This is in accordance with the fact that the Ta is a weaker β-phase stabilizer than Nb. The particle size of the ternary appears to be somewhat greater than that of the binary, although care has to be taken in the interpretation of the SEM-BEI photographs. This size difference may help to explain some of the lower J_c of the ternary.

Table II. Vol. % and average diameters of the particles

	Vol % α-Ti	Average α-Ti diameter (nm.)
Ternary		
Ann	25.2	123
HT	23.3	112
CW	24.8	127
Binary		
HT	-	-
CW	18.6	114
Ann	17.8	107

C. Comparison of the J_c results with those obtained previously on 15 wt.% Ta alloy.

Figures 3 and 4 show a comparison of the J_c data obtained on the annealed materials in this work, with that on 15 wt.% Ta, 44 wt.% Ti materials in the same annealed condition, obtained in the earlier work [1]. Unfortunately, in the recent work, we only obtained data up to 8T at 4.2 K and to 7T at 1.8 K. It is unlikely, however, that, had the higher field data been obtained, the conclusions to be drawn would have been significantly different. While the binary data obtained in the recent work was slightly lower than that obtained earlier [1], considering the many variables involved, the differences are not too great. It appears that the 15 wt.% Ta alloy shows somewhat better properties than the binary at both 1.8 K and 4.2 K. The 25 wt.% Ta alloy, even in its best condition (Ann) at 1.8 K, shows only similar properties to those of the annealed binary. At 4.2 K, its J_c values under all conditions are inferior.

A comparison of particle size and vol.% in the two different ternary alloys cannot be made as no information of this type was obtained on the 15 wt.% Ta alloy.

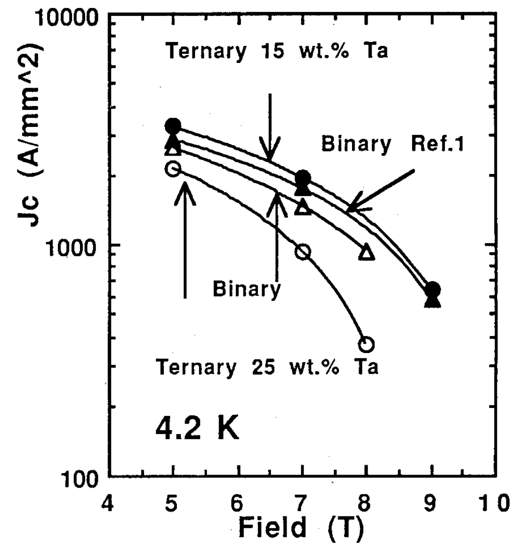

Fig. 3. Comparison of J_c vs. field curves between 15 wt.% Ta alloy and those of the 25 wt.% Ta alloy at 4.2 K

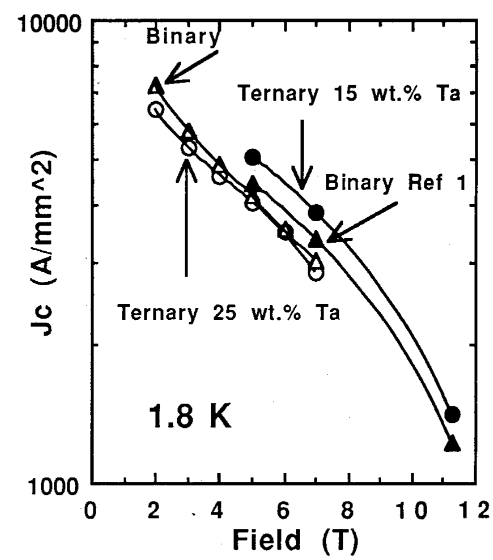

Fig. 4 Comparison of J_c vs. field curves between 15 wt.% Ta alloy and those of the 25 wt.% Ta alloy at 1.8 K.

SUMMARY

While more precipitate is obtained in the 25 wt.% Ta alloy, it is of a larger particle size and appears to be a less effective pinner than the α-Ti precipitate obtained from the binary. An examination of the T_c's and the H_{c2}'s of the two alloys whose J_c's are compared above, are shown in Table III below. The H_{c2} data were estimated from Reference 8 and the T_c data from Reference 5.

Table III. H_{c2}'s and T_c's

Values estimated from	Ref. 8 H_{c2} 4.2 (T)	Ref. 8 H_{c2} 2.0 (T)	Ref. 5 T_c (K)
25 wt.% Ta 45 wt.% Ti	10.9	14.8	8.2
15 wt.% Ta 44 wt.% Ti	11.6	15.0	8.85
Binary 46.5 wt.% Ti	11.3	14.1	8.9

An examination of the data in Refs. 8 & 5 shows that the 25 wt.% Ta alloy is in a region of the ternary diagrams where the values of H_{c2} change rapidly with composition, both at 2.0 K and 4.2 K and that the alloy has a relatively low T_c.

At 4.2 K, the 25 wt.% Ta alloy is unlikely to show better high field properties as it has both lower H_{c2} and T_c than the binary. Even at 2.0 K, the H_{c2} of the 25 wt.% Ta alloy is lower than that of the 15 wt.% Ta alloy and the actual value is much more critically dependent on titanium content. The results indicate that, while the heat treatment and cold work cycles may be capable of modification to take advantage of the larger volume percentage of precipitate available in the ternary, it does not appear worthwhile to carry out such an investigation on the 25 wt.% 45 wt.% Ti alloy.

The 15 wt. % Ta alloy appears to have better H_{c2} properties than the 25 wt.% ternary and the binary at both 4.2 K and 2.0 K. It's T_c is higher than that of the 25 wt.% Ta alloy and almost equal to that of the binary. While, of the two alloys, the 15 wt.% Ta appears to have more potential, an examination of the ternary diagrams suggest that other compositions have greater potential still. Assuming that a high H_{c2} is desirable, if it can be achieved without sacrificing T_c significantly, lowering the titanium content to around 40 wt.% would be advantageous for both 4.2 K and 1.8 K operation. An intermediate Ta level (19 wt.%) would appear optimum for 4.2 K operation and 30 wt.% Ta may be required for best operation at 1.8 K. These conclusions are similar to those reached in Ref.2. Earlier work, carried out on a lower Ti alloy [2] (41 wt.% Ti 15 wt.% Ta), and that on an intermediate Ta alloy [10] (44 wt.% Ti 19 wt.% Ta), did not employ the techniques developed for the SSC binary [11] conductor and, therefore, showed relatively low J_c's, presumably controlled by "extrinsic" effects.

From this and other work [3], it appears that the precipitation behavior, and perhaps the effectiveness of the precipitate as a pinner, is different in the ternaries from that of the α-Ti precipitate in the binary. For this reason, whatever ternary alloy containing tantalum is chosen for further work, an investigation of heat treatment, cold work and precipitation behavior will have to be carried out to ensure that optimum J_c properties are obtained.

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