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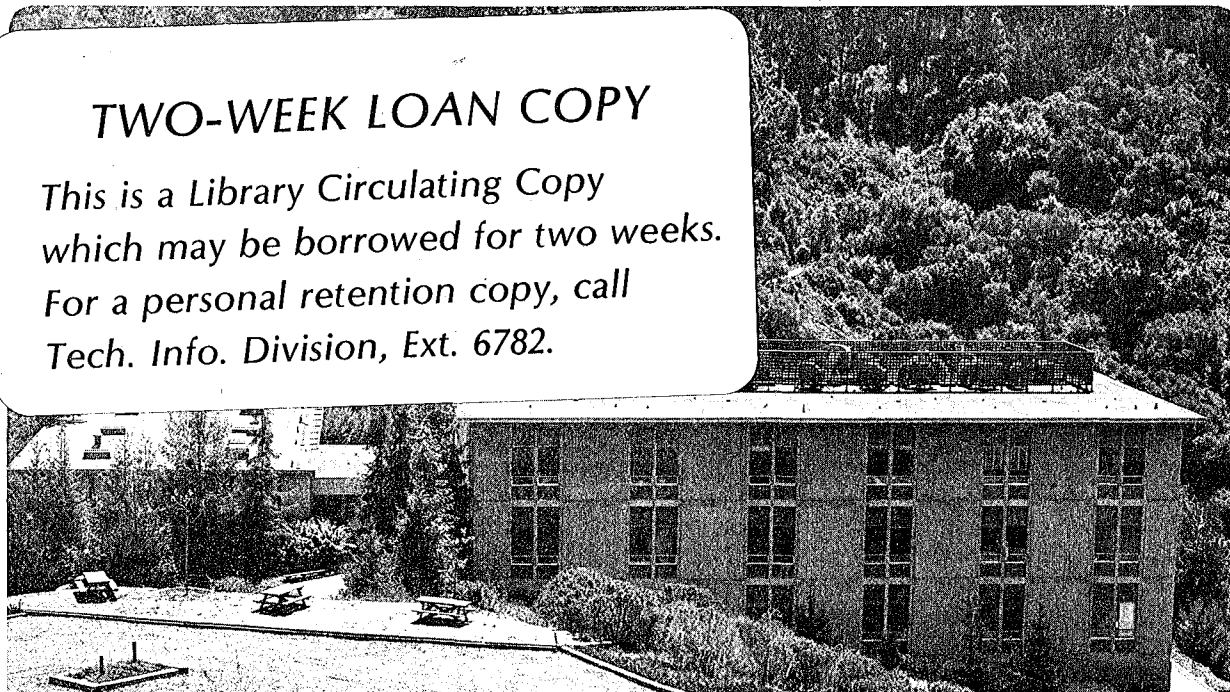
M. Hong and J.W. Morris, Jr.

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DIRECT SOLID-STATE PRECIPITATION PROCESSED

A-15 (Nb$_3$Al) SUPERCONDUCTING MATERIAL

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ABSTRACT

A "solid-state precipitation" process was used to prepare supercon-
ducting tapes containing Nb$_3$Al in a niobium matrix. Small ingots of
Nb-(17-19 at.%)Al were prepared by arc-melting, homogenized, quenched,
warm-rolled into tape, and aged at 750°C-900°C to precipitate the A15
phase. Transmission electron microscopy studies revealed Nb$_3$Al precipi-
tation in fine particles which formed as semi-continuous network over
sub-grain boundaries formed by the recovery of deformation-induced
dislocations. Promising high-field critical currents were obtained
($J_C \sim 10^4 A/cm^2$ in a field of 14T at 4.2K).

We have recently reported[1,2] that a solid-state precipitation process can be used to produce superconducting materials from the V-Ga system which have high critical transition temperatures and reasonable overall critical current densities. The purpose of this letter is to report initial results from similar research on the Nb-Al system.

The A15 compound Nb_3Al is difficult to synthesize in its stoichiometric composition because of competition from the more stable Nb_2Al phase,[3] but appears to be one of the best of the A15 superconducting compounds. Its critical temperature has been reported to be as high as 18.8K,[4] and its upper critical field exceeds 30T.[5] Woollam, et al.[6] have recently measured critical current densities in small samples of Nb_3Al which are near 10^5 A/cm^2 in 20 tesla fields at 4.2K. The research materials exhibiting these exceptional properties were, however, made using processes which cannot be scaled up to produce superconducting wires in useful lengths. Since the "bronze" and "in-situ" processes used in the manufacture of multifilamentary Nb_3Sn and V_3Ga superconducting wires are inapplicable to Nb_3Al for thermodynamic reasons,[7] the practical utility of Nb_3Al depends on the identification of new and suitable wire manufacturing techniques. Relevant research is hence underway in a number of laboratories.[8,9]

The "solid-state precipitation" process[1] is a monolithic manufacturing technique which involves arc-melting and homogenization to create a ductile metallic solid solution of the A15-forming elements, cold or warm deformation to form the product into wire or tape, and subsequent aging at intermediate temperature to precipitate the A15 phase.

In the present work ingots were prepared by arc-melting a powder mixture of Nb and Al under an argon atmosphere using a non-consumable tungsten electrode. During melting the samples were inverted on a copper hearth and re-melted at least four times to achieve complete alloying between Nb and Al. There is, of course, some loss of Al through vaporization during melting, and the starting composition of the mixture was hence adjusted to achieve the desired final Al content. The arc-cast samples were rod-shaped ingots weighing ∿30 gms. with nominal composition (in atom percent) Nb-17.7 Al or Nb-18.8 Al.

After casting the ingots were homogenized under an argon atmosphere for one hour at 1930°C, and were then rapidly quenched by dropping them from the homogenizing furnace into an ice-brine solution. While recent determinations of the Nb-Al phase diagram[10,11] disagree on the high temperature range of the A15 phase, there is agreement that temperatures between 1850°C and 2000°C fall in the BCC solid solution field for Nb-(17-19)Al. The as-quenched samples were, in agreement, of BCC structure; no evidence of second phases appeared in either x-ray diffraction patterns or optical micrographs, and no quench cracks or micro-fractures were found.

Given the integrity of the as-quenched specimens they could be deformed severely at room temperature without catastropic failure. However, some cracking did occur during cold deformation. A scanning electron microscopic examination of these fractures revealed a mixed cleavage and dimpled fracture mode, which suggests that the cracking has its source in the relatively large grain size established

during homogenization. To avoid this problem, the samples were warm-rolled into tape at temperatures between 300°C and 400°C. At these temperatures deformations of up to 99% could be accomplished without fracture.

Following deformation the samples were annealed for various times at temperatures in the range 600°C to 900°C to precipitate the A15 phase. A systematic examination of the consequences of these aging treatments is now in progress. However, preliminary results show a marked dependence of the size and morphology of the A15 precipitates on the aging treatment and the prior deformation, a dependence which is due in part to the interaction between the precipitation process and the recovery of the deformation-induced defects. A transmission electron micrograph of a Nb-17.7Al sample which was deformed 99%, then aged for 3 hours at 900°C, shows almost all of the deformed BCC solution has transformed to the A15 phase. The grains of the A15 phase are large and elongated, having widths on the order of 1000 Å and lengths closer to 5000 Å. Both the A15 phase and the residual matrix are low in dislocation density. A similar sample aged 3 hrs at 750°C, however, has a very different microstructure. The dislocations present in the deformed BCC matrix have apparently recovered into sub-cells a few microns in size. Fine, equi-axed A15 particles of diameter near 300-1000 Å are precipitated along the cell walls in a continuous or semi-continuous network. Since a fine A15 grain size is expected to enhance the critical current density through strong flux pinning at the grain boundaries, the latter microstructure should be conducive to a high critical current, provided, of course, that the

A15 particles have good inherent superconducting properties and are present in a reasonable volume fraction.

Three ingots were processed to provide specimens for initial tests of superconducting properties. The specimen compositions and treatments were (1) Nb-17.7Al, deformed 90%, followed by aging for 3∿120 hrs at 750°C; (2) Nb-17.7Al, deformed 99%, followed by aging for 2 hrs at 750°C; (3) Nb-18.8Al, deformed 90%, followed by aging for 6 hrs at 750°C. The first two specimens were intended to show the effect of increased pre-aging deformation. The third was made to explore the effect of increased Al content. Critical temperature measurements were made using the inductive technique, and showed an onset critical temperature near 14K in all cases. Critical current densities were measured using a standard four-probe technique in transverse applied magnetic fields up to 19T in water-cooled bitter solenoids. The critical current was taken to be that current at which the potential difference across the voltage leads (spaced 5mm apart) exceeds $1\mu V$, and the current density, J_c, was taken to be the critical current divided by the total cross-sectional area of the specimen. The results are plotted in Figure 1.

An examination of the $J_c(H)$ curves shows that the overall critical current density is strongly influenced by both prior deformation and sample composition. Increasing the prior deformation by a factor of 10, from a 10:1 (90%) to a 100:1 (99%) thickness reduction in rolling, increases the critical current density of Nb-17.7Al in the standard heat treatment by roughly a factor of 5: From 10^3 A/cm^2 to 5×10^3 A/cm^2 at 10T and 4.2K. Increasing the Al content by 1%, to 18.8Al, causes a still more pronounced increase in critical current density: After a

90% area reduction followed by the standard heat treatment $J_c \sim 10^4$ A/cm^2 at 14T and 4.2K.

The metallographic studies needed to complete the interpretation of these results are in progress, but initial metallographic observations suggest that the increase in J_c with prior deformation is associated with a finer grain size and distribution of the A15 phase, due to increased dislocation density and a finer cell structure. The principal effect of increasing Al content appears to be an increase in the volume fraction of the A15 phase, which improves both the connectivity of the superconducting phase and its areal fraction in the sample cross-section. Research toward further improvement of the critical current of samples made by solid-state precipitation is in progress.

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FIGURE CAPTIONS

Fig. 1. The overall critical-current density (J_c) versus applied

transverse magnetic field for Nb-17.7 at.% Al, deformed 90%,

followed by aging for 3-120 hrs (A), Nb-17.7 at.% Al, de-

formed 99%, followed by aging for 2 hrs at 750°C (B), and

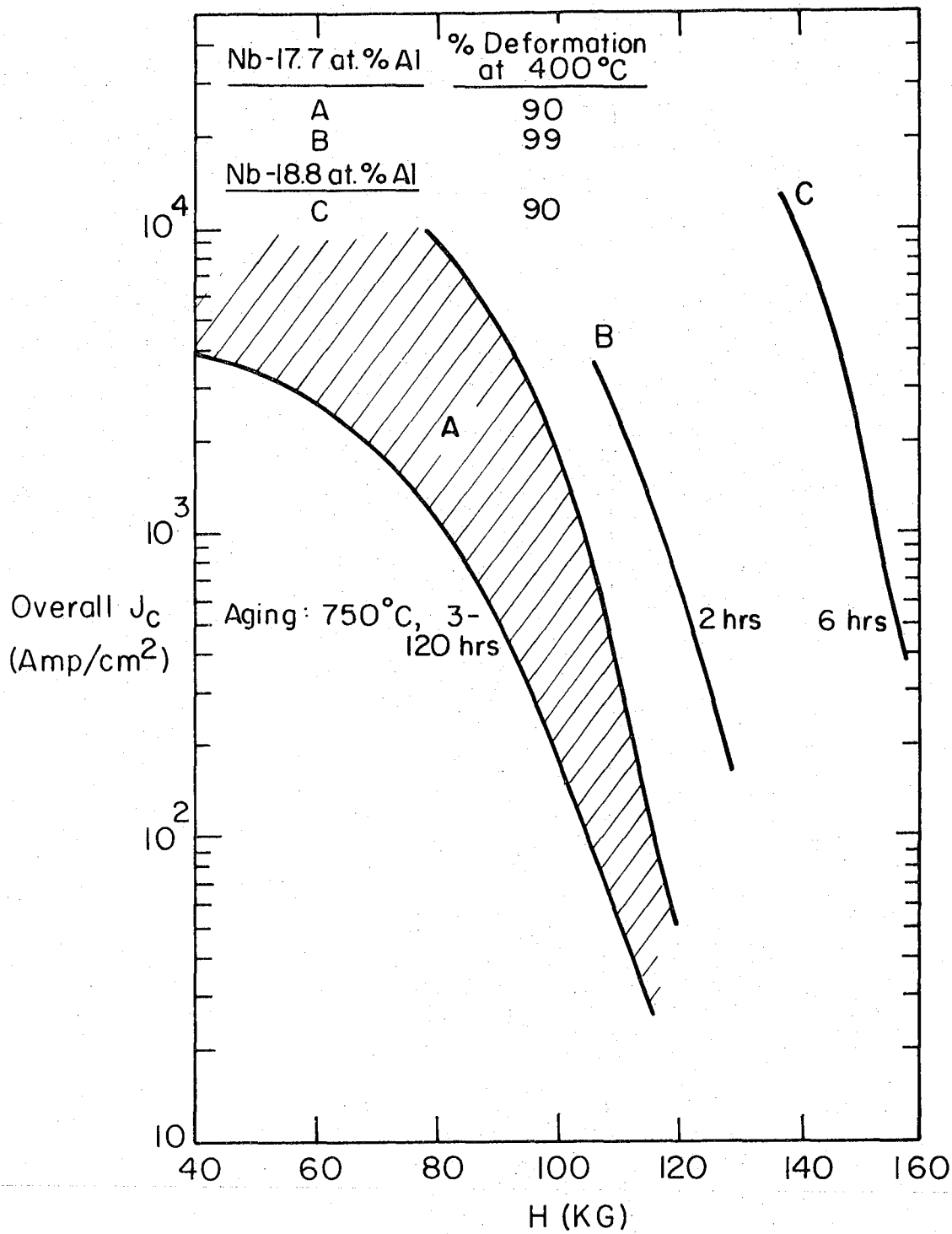Nb-18.8 at.% Al, deformed 90%, followed by aging at 6 hrs at

750°C (C).

Figure 1.

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