A QUENCH-AGE METHOD FOR THE FABRICATION OF Nb(Al) SUPERCONDUCTORS*

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ABSTRACT

The possibility of taking advantage of the extended, high temperature solid solubility of Al in Nb in order to produce composite Nb-Nb₃Al superconductors was investigated. Rapid quenching after an elevated (1950°C) solution treatment makes it possible to retain at room temperature a metastable solid solution containing up to 21 at.% Al. The solid solution transforms into the A-15 phase in the course of a short and relatively low temperature (1000°C) aging treatment. The inductively measured onset of superconductivity was 17.6 K in a 100 μ m thick core of Nb₃Al embedded in a Nb matrix and prepared by the quench-age method.

INTRODUCTION

High values of the critical field and of the current carrying capacity have been reported for the A-15, Nb₃Al intermetallic compound. No viable, practical method of processing this material, however, has yet been developed. Most trials were based on a direct diffusion reaction between Al and Nb to yield the Nb₃Al phase. The composite material produced at elevated temperature had a low current carrying capacity¹ while the materials reacted at low temperatures displayed relatively low T_c values.²

The solid solubility of Al in Nb reaches 23 at.% at 1960°C and decreases rapidly with decreasing temperature.³ At room temperature, the range of stability of the Nb₃Al compound extends from 18 to 22 at.% Al. The objective of the present study was to investigate the possibility of taking advantage of these particular features of the Nb-Al phase diagram in order to develop a new method for producing composite Nb-Nb₃Al superconductors.

EXPERIMENTAL METHODS

Composite Nb-Nb₃Al superconductors were prepared by sheathing a 0.093 cm diameter, 99.99% pure Al rod in a 0.58 cm external diameter, 99.9% pure Nb tubing. This composite was first swaged to 0.188 cm diameter and then either flat rolled to a 0.037 cm thick tape or wire drawn to 0.05 cm diameter. Samples, approximately 8 cm long were heated to temperatures above 1900°C by passage of a low voltage electric current. The current settings necessary to attain a given temperature had been previously determined using an optical pyrometer. The samples were heated in a vacuum chamber (10⁻⁵ mm Hg). The Nb sheathing prevented the loss of Al by evaporation. As a result of the very high interdiffusion coefficients at the elevated temperatures attained in the sample, it was necessary to limit the duration of the anneals to approximately 30 sec. Lengthier anneals depressed the peak Al content in the core to below the solubility

limit of Al in the A2, Nb(Al) solid solution at 1900°C. A relatively rapid quench of the order of 500°C/sec was achieved by cutting off the current to the sample and simultaneously directing a jet of high pressure He gas to its surface. The aging heat treatments were carried out on vacuum sealed samples in the 800-1000°C temperature interval.

Samples were prepared for metallographic, electron microprobe and x-ray diffraction examination. Microhardness test were also made at various stages of the process. An inductive method was used to measure the superconductive critical temperatures.

RESULTS AND DISCUSSION

In the course of this investigation we have studied some of the mechanical properties of the quenched solid solution, the parameters of the aging heat treatment and the morphological features resulting from the transformation of the metastable solid solution into the A-15, Nb₃Al phase. The addition of Al, even at low concentrations significantly hardens the Nb matrix and practically excludes the possibility of submitting the solid solution to extended mechanical deformation. A deformation in excess of 6% results in the appearance of cracks in the sample.

Metallographic examination of the as-quenched samples revealed a central reaction zone about 100 μ m thick with a microhardness of 385 V_H. The microhardness value is similar to that observed in a Nb(Al) solid solution prepared by conventional alloying technique. The electron microprobe point analysis results indicate that the highest Al content in the central zone was 21 at.%. No traces of the presence of the A-15 phase at this stage were detectable either metallographic or X-ray examination. Following an aging treatment of one hour at 850, 900 or 1000°C no changes

Fig. 1. Central core of the A-15 phase embedded in the Nb matrix. The dark areas have a composition corresponding to the two phase, Nb(Al) and Nb₃Al, region of the Nb-Al phase diagram. They show a very fine dispersion of Nb₃Al precipitated at low temperatures within the metastable Nb(Al) solid solution.

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in composition or reaction zone width could be observed (Fig. 1). The microhardness increased, however, to 890 VH, a value characteristic of the intermetallic A-15 phase. The presence of this phase after the aging treatment was further confirmed by X-ray analysis. The formation of the A-15 phase at temperatures below 1000°C is highly significant. Previous investigations have shown that it was not possible to form the Nb₃Al compound by means of a diffusion reaction at temperatures below 1100°C even by starting with an extremely fine distribution (3000 Å) of the reaction components. Higher reaction temperatures lead to the formation of the A-15 phase having a relatively coarse grain size. A large grain size is known to impair severely the current carrying capacity of A-15 based superconductors.

Metallographic examination showed that mechanical twinning is an important mode of deformation of both the stable and the metastable Nb(Al) solid solution (Fig. 2). Depending on the Al concentration, the quenching rate and the aging temperature, the metastable solid solution, in the course of the aging treatment, transforms into the A-15 phase by two mechanisms.

Fig. 2. Mechanical twins in the metastable Nb(Al) solid solution.

Fig. 3. A-15 phase precipitates along the twin network, formed in the course of the aging treatment.

In agreement with Lundin and Yamamoto's findings³ we observed for high aging temperatures the morphological features characteristic of a massive transformation. For lower aging temperatures and for Al concentrations up to the maximum retainable 21 at.% Al, we also observed the precipitation of the A-15 phase which occurs preferentially along grain boundaries and the twin network. Actually, by a judicious choice of conditions, it is possible to prepare a sample in which the A-15 phase forms almost exclusively along the twin network (Fig. 3). In principle, therefore, a filamentary A-15 network can be achieved in this manner.

Several different aging temperatures were tried in order to establish a practical lower limit for this heat treatment. In theory, the lower the A-15 formation temperature, the higher will be the local atomic order and the smaller the grain size, leading to better superconducting properties. Aging occurs at temperatures as low as 850°C; at this temperature, however, the precipitation rate is very slow. Further work is necessary in order to determine a quantitative correlation between the current carrying capacity and the parameters of the aging treatment.

The critical temperature (defined as the onset of superconductivity) of samples aged at 900, 950 and 1000°C was 17.640.5 K. This value compares favorably with the values reported for equivalent Al contents by Kohr et al.⁴ for bulk materials formed at high temperatures and subsequently annealed at low temperatures.

CONCLUSIONS

The results of this study indicate that composite Nb-Al wires and tapes can be diffusion-solution treated, quenched and aged in order to produce a functionally shaped Nb-Al superconductor. This method is not limited by equilibrium phase boundary considerations; moreover, the formation temperature of the A-15 phase below 1000°C eliminates the grain growth inherent to high temperature reactions. Further development of this process may, in principle, lead to the fabrication of a high T_c , high J_c stabilized Nb₃Al superconductor.

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REFERENCES

1. T. W. Eager and M. R. Rose, IEEE Trans. Mag. MAG-11, p. 214, 1975.
2. S. Ceresara, M. V. Ricci, N. Sacchetti and G. Sacerdoti, IEEE Trans. Mag. MAG-11, p. 263, 1975.
3. C. E. Lundin and A. S. Yamamoto, Trans. AIME 236, p. 863, 1975.
4. J. G. Kohr, T. W. Eager and R. N. Rose, Met. Trans. 3, p. 1177, 1972.

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