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Introduction

The Beta-tungsten intermetallic compounds have proven to be the best superconducting materials developed thus far, from the standpoint of critical temperature and critical current carrying capacity. The use of these materials for such applications as high field magnets, magnetic suspension devices and power transmission, to name a few, holds great promise for the future. One of the most interesting of the Beta tungsten compounds is Nb_3Al. The basic binary compound and ternary modifications of the compound have exhibited remarkable properties. Critical temperature in the range of 21°K have been reported.(1,2) Critical fields of 400 KG at 4.2°K have been measured(3) along with critical currents of 5×10^5 amps/cm^2 at 100 KG(4). Despite these very exceptional properties, the material has not been successfully fabricated into useful conductor. The work presented in this paper describes a study aimed at fabricating stabilized conductor containing both binary and ternary intermetallics of the Nb-Al system.

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Fabricating Nb_3Al Conductors

One of the primary difficulties encountered in fabricating conductors of Nb_3Al is the high temperature required to form the compound. A binary phase diagram of the Nb-Al system is shown in Fig. 1. It can be seen that Nb_3Al forms from the liquid at 1960°C. Consequently, most investigations have utilized the arc melting process for preparing the intermetallic compound.(5) This method however is not amenable for use in conductor fabrication.

Several investigators have prepared the binary compound by a process that could be utilized for conductor fabrication. Kohr(6) has described results of a dipping experiment and metalliding experiments, where small amounts of aluminum are deposited on a niobium substrate. This composite was then reacted at an elevated temperature. Looking at the phase diagram in Fig. 1, the problems associated with this process become apparent. In order to obtain only the compound Nb_3Al, a temperature in excess of 1870°C is required. Maintaining and controlling this temperature while reacting long lengths of superconductor is very difficult. Furthermore, because this reacting process must be performed in vacuum (to avoid oxidation), aluminum loss by vaporization is very rapid. Reaction time must be limited to periods of 10 to 20 seconds, and the resulting layer thickness is very small.

In the work reported here, the primary goal was the development of a fabrication technique whereby a low reaction temperature could be used.

If such a technique were developed, it would be possible to fabricate a fully stabilized conductor sheathed with either aluminum or copper.

Experiment Procedure

Several fabrication methods were investigated. In the first, niobium wires were coextruded with high purity aluminum. The composite, containing 2 strands of niobium (0.25 mm in diameter) was reacted in a radio-frequency induction unit (toccotron) using the floating zone technique. The induction coil was moved at a rate of 3 cm/hr. The temperature of the floating zone was kept slightly above the melting point of aluminum (700-750°C). Metallographic analysis indicated that significant diffusion had occurred. However, instead of forming a continuous layer of Nb_3Al, it was found that the interface consisted of a mixture of niobium solid solution and several intermetallic phases other than Nb_3Al. This result was attributed to the excessive amount of aluminum available for reaction, and the necessarily low reaction temperature.

In order to circumvent these difficulties, a second fabrication process was investigated. This consisted of preparing composites having a niobium core and a sheath of copper - 4% aluminum. As an addition parameter, composites were prepared with sheaths of the ternary alloy: copper-3% aluminum - 3% germanium. Ingots were melted in a graphite crucible in argon, and swaged to a diameter of 8 mm. Holes were drilled and a niobium rod (3 mm in diameter) was inserted into each ingot. The composites were then swaged to approximately 5 mm and reacted by means of the floating zone process. Because of the presence of a copper alloy sheath, the zone temperature could be raised to 1000°C. As will be discussed below a layer of intermetallic compound was formed under these conditions, but it was quite thin and somewhat irregular.

A third process was devised whereby an increase in layer thickness could be obtained by providing additional aluminum at the interface. This process consisted of drawing a composite, made up of a niobium core (2 mm in diameter) surrounded by a thin aluminum sheath (4 mm in O.D.), which in turn was surrounded by a copper sheath (8 mm O.D.)*. The composite was cold drawn to a final O.D. of 1.7 mm. Specimens were subsequently reacted at temperatures ranging from 1000°C to 1050°C for times varying between 3 minutes and 30 minutes.

Optical microscopy was used to study the nature of the reaction products formed by each fabrication process. Specimens were cut, mounted and polished using conventional metallographic techniques. An anodizing solution was used to etch grain boundaries and delineate structure.

Results

As indicated previously, the coextruded niobium-aluminum specimens exhibited no evidence of a continuous intermetallic layer. The specimens sheathed with bronze (Nb-Al and Nb-Al-Ge alloys), on the other hand,

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*The role of the copper sheath is two-fold. First, it provides a means of raising the aluminum temperature considerably above its melting point while maintaining contact with the niobium surface. Secondly, since aluminum is soluble in copper, the outer sheath provides a sink for removal of excess aluminum. A similar process has been used by K. Tachikawa to produce V_3Ga tape(7).

did contain a thin layer of the intermetallic compound. A representative micrograph of the interface is shown in Fig. 2. The intermetallic layer has formed by diffusion of aluminum and germanium from the bronze to the interface. Detailed metallographic examination indicated that the layer thickness was somewhat inconsistant, varying between 1 and 5 microns. Longer annealing times did not result in the formation of a thicker or more uniform layer.

Specimens containing the niobium core with sheaths of aluminum and copper also exhibited the presence of an intermetallic layer. This layer was found to be quite uniform as seen in Fig. 3. The thickness appeared to be dependent on both reaction temperature and time. Optimum reaction temperature was found to be 1010°C ± 10°C. At higher temperatures the intermetallic compound growth was very discontinuous, while at lower temperatures, layer growth rates were found to be slow. In the optimum temperature range, formation of the intermetallic layer occurred within the first ten minutes of reaction time, then decreased in size. This can be seen by comparing Figures 3 and 4, representing reaction times of 10 and 30 minutes respectively. Longer reaction times resulted in the dissolution of the intermetallic layers. The maximum layer achieved after a 10 minute reaction time was in the range of 10-12 microns.

In order to determine the nature of the intermetallic layer, x-ray analysis was performed. This was achieved by etching away the copper and copper aluminum phases with nitric acid. X-ray peaks representative of both Nb_3Al and niobium were observed indicating that the layer formed was indeed the desired intermetallic compound Nb_3Al.

Discussion

The results of these studies point out a number of difficulties associated with the fabrication of stabilized Nb_3Al conductor stabilized with copper. Processes utilizing temperature in excess of 1060° C are not feasible, because the sheath material, copper, becomes molten. While the intermetallic compound Nb_3Al can be formed at reaction temperatures below 1060° C, layer growth occurs only under very special conditions. If there is an excessive amount of liquid aluminum present at the niobium surface, a distinguishable layer of Nb_3Al will not form. Instead, the intermetallic compounds richer in aluminum (Nb_2Al and $NbAl_3$) form, in a very discontinuous manner. These compounds can be kept from forming by limiting the amount of aluminum available for reaction. This was the case for specimens fabricated with bronze sheaths. Here, the aluminum concentration is controlled by the amount of alloy added to the copper sheath. The maximum amount of aluminum or ternary element that can be added is limited, however, because of fabricability problems. The addition of alloying elements in excess of 6-8% causes the bronze sheath to work harden severely, making large reductions difficult. With this limit placed on alloy addition, the intermetallic layer grows very slowly. All reactions occur in the solid state, where nucleation and growth kinetics are slow and orientation dependent. Consequently, the layer is thin and irregular.

Our third fabrication process represents a metallurgical condition which is more conducive to rapid layer growth. The outer sheath of copper allows the aluminum to be heated considerably above its melting point. Hence, the intermetallic layer grows from the niobium substrate into liquid aluminum. The growth rate is apparently enhanced, as is the tendency to grow as a planar interface. The presence of copper serves another important purpose. Because aluminum is soluble in copper, excess aluminum is removed from the interface during the reacting anneal, preventing the formation of the compounds Nb_2Al + $NbAl_3$.

Because the aluminum zone thickness is so small, and the temperature so near the melting point of copper, the reaction times and temperatures are very critical. If the temperature is too high, aluminum diffuses away from the interface before an adequate Nb_3Al layer thickness can be achieved. If time at temperature is long, aluminum diffuses into the copper, causing dissolution of the existing intermetallic compound. If the reaction temperature is lowered, the freezing point of aluminum is approached and less liquid is available for reacting with Nb. Hence growth rate is decreased.

Conclusions

While much work of a metallurgical nature remains to be done, in fully understanding the nucleation and growth mechanisms involved, the processes described are feasible techniques for fabricating long lengths of stabilized Nb_3Al superconductor. Once the parameters have been optimized, it is conceivable that multistrand twisted composites of both the binary and the ternary intermetallic can be made.

Critical temperature, fields, and currents for these alloys will be reported subsequently.

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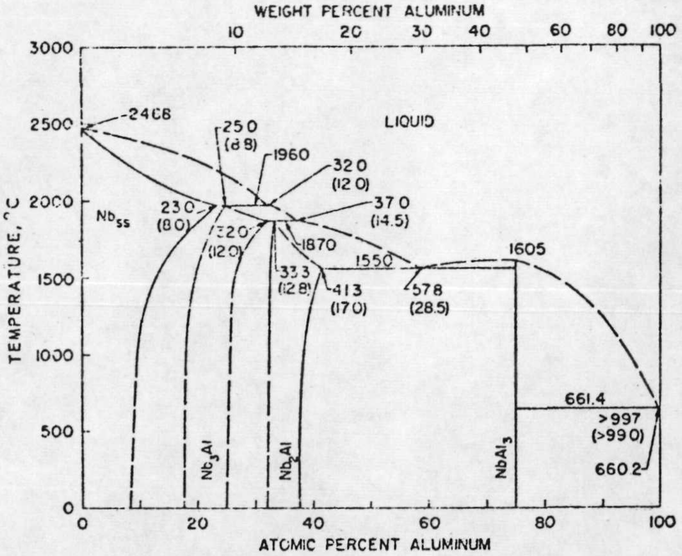

Fig. 1 - Binary phase diagram of the niobium-aluminum system.

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Fig. 2 - Typical intermetallic compound interface formed in specimens sheathed with bronze. Note irregular layer thickness. As Polished

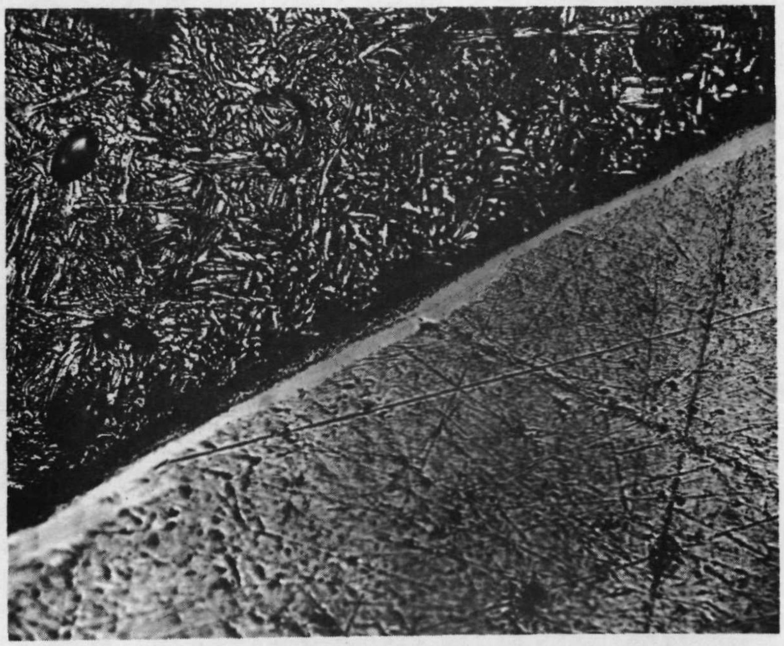

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Fig. 3 - Intermetallic layer formed after reacting at 1000° C 10 minutes. Anodized

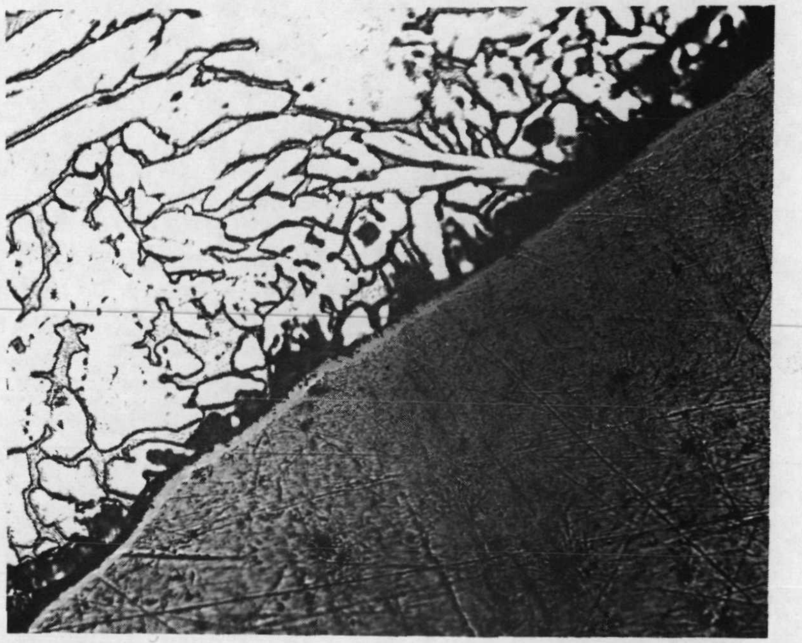

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Fig. 4 - Intermetallic layer formed after reacting at 1000° C for 30 minutes. Note diminishing thickness. Anodized