

## **DEVELOPMENT OF UNREACTED NbAl MULTIFILAMENTARY STRANDS FOR FABRICATION OF Nb<sub>3</sub>Al SUPERCONDUCTING CONDUCTORS FOR HIGH-FIELD APPLICATIONS**

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### **1. Introduction**

The superconducting Nb<sub>3</sub>Al compound has a great potential for high-field and large-scale applications. In comparison with the most widely used Nb<sub>3</sub>Sn, it exhibits intrinsic higher T<sub>c</sub> and H<sub>c2</sub>, larger J<sub>c</sub>, and a very good strain tolerance. The fabrication of Nb<sub>3</sub>Al superconducting strand is a two-step process. The first step consists in obtaining of a NbAl final-size strand as a microscale assembly of the Nb/Al constituents, and the second step is the heat treatment of the strand in order to obtain the desired A15 Nb<sub>3</sub>Al superconducting phase.

### **2. The fabrication of Nb-Al precursor cable by jelly-roll and powder-in-tube technique**

The Nb/Al precursor strands have been fabricated both by jelly-roll (JR) and powder-in-tube (PIT) technique. In the JR process, first thin foils of Nb and Al were superimposed and then wrapped around a 9 mm Nb cylindrical core. The thicknesses of the Nb and Al sheets were 104 μm and 36 μm, respectively. The as-obtained composites have been inserted in a Fe tube, and then rod rolled, swaged and drawn up to 0.60 mm diameter. The Fe sheath has been removed using a solution containing 40% HCl and 40% HNO<sub>3</sub> volume fractions: the final wire diameter was 0.43 mm, with a Nb/Nb<sub>3</sub>Al ratio = 0.14.

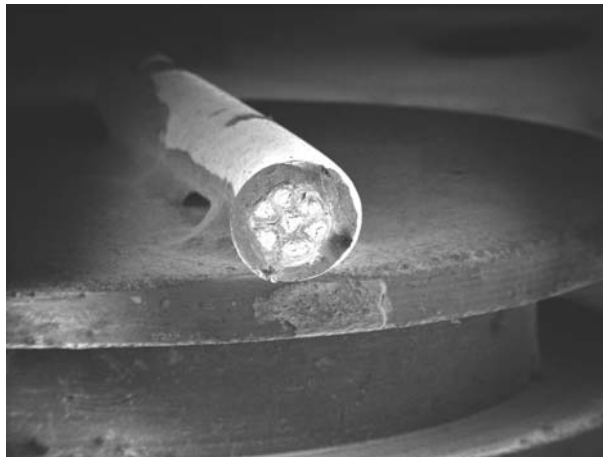
In the PIT technique, the Nb and Al powders were mixed to obtain the stoichiometric Nb<sub>3</sub>Al nanostructured powder with reduced diffusion spacing between Nb and Al. The powder grains mean size was of about 5 μm. To obtain the nanofilaments billets, the Nb<sub>3</sub>Al composite is pressed into a Nb or Cu tube. The monofilamentary billet is assembled into a Nb sleeve and then the multifilamentary is drawn down to the final size.

A good understanding of the phase formation and the microstructure evolution is very important step in order to optimise the superconducting properties of the Nb-Al multifilamentary wires. There are two thermal processing methods to obtain the Nb<sub>3</sub>Al superconducting filaments starting from the unreacted Nb-Al composites: by heat treatment at temperature below 1000°C or by high temperature treatment. The high temperature treatment is a two-step process. First the sample is heated for a short time at 2000°C and then is rapid cooled in order to retain the bcc phase. A second step consists in a low temperature heat treatment (800 °C for 12 hours) to transform the bcc in A15 superconducting phase. This two-step process is now named Rapid Heating Quenching and Transformation (RHQT). In the present study the low temperature treatment has been used for the PIT strand while the RHQT process has been used for the JR strand.

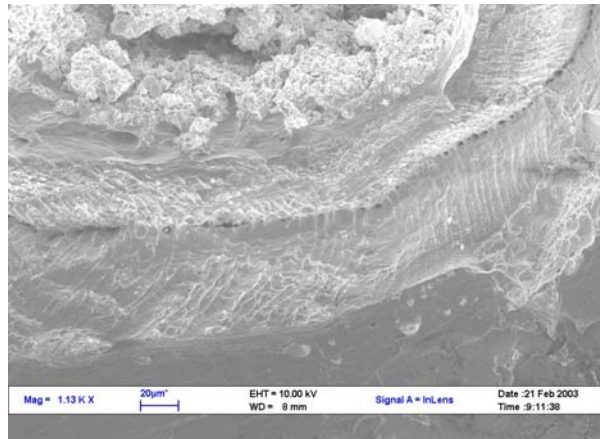
### 3. Results

#### 3.1 Cross-section morphology of the precursor wires

A typical structure of the Nb-Al multifilamentary wires manufactured by the PIT technique is presented in Figure 1. The cross-section of a Nb-Al strand after heat treatment at 950°C for 3 days is shown in Figure 2. It is important to note that during the heat treatment, the Al diffuses from the Nb-Al core into Nb sleeves. The diffusion front is clearly visible. A typical cross-section of a jelly-roll wire is presented in Figure 3. As it can be seen, the Nb and Al layers form a compact structure and have a thickness of about 300 nm and 800 nm, respectively.



*Figure 1. Typical cross-section of a PIT multifilamentary Nb-Al strand. The bright gray regions in the centre of filaments are the mixture of Nb and Al powders. The dark gray regions are the Nb sleeves.*



*Figure 2. Cross-section of a PIT multifilamentary Nb-Al strand after a heat treatment at 950°C for 3 days. The bright gray regions in the centre is the (NbAl)ss + Nb<sub>3</sub>Al. The line between the dark and bright region inside the Nb sleeve represents the diffusion front of the Al in the Nb sleeves.*

#### 3.2 Structural properties

Figure 4 presents a typical diffraction pattern both for the as-quenched and the annealed JR strand. The peaks observed in the XRD pattern of the as-quenched wire can be assigned to pure Nb and to Nb-Al supersaturated solid solution (Nb(Al)ss). As can be seen, the peaks corresponding to Nb(Al)ss have completely disappeared for the samples annealed at 850 °C for 10 h. Instead, the peaks of A<sub>15</sub> phase are present together with those of pure Nb. It is to be noted that no reflections corresponding to the Nb<sub>2</sub>Al phase were observed. This indicates that the (Nb(Al)ss) is mainly transformed into Nb<sub>3</sub>Al. Such a transformation implies only a change in

the crystalline structure, without any change in the local composition of the sample, leading to very fine grain structure of the stoichiometric composite. The peaks corresponding to pure Nb are mainly due to the Nb core, but can be attributed also to the unreacted Nb.

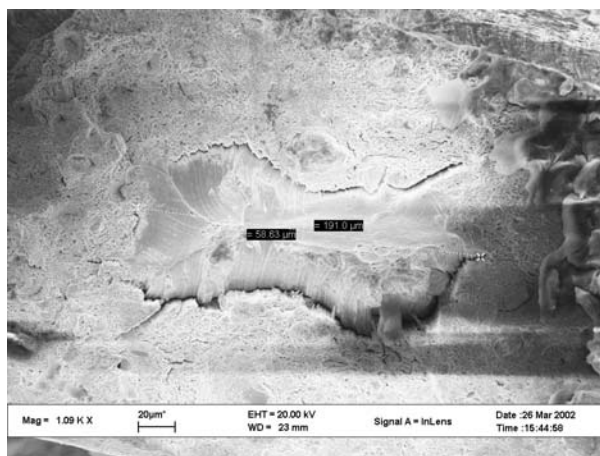


Figure 3. Typical cross-section of jelly-roll wire. The central region is the Nb core.

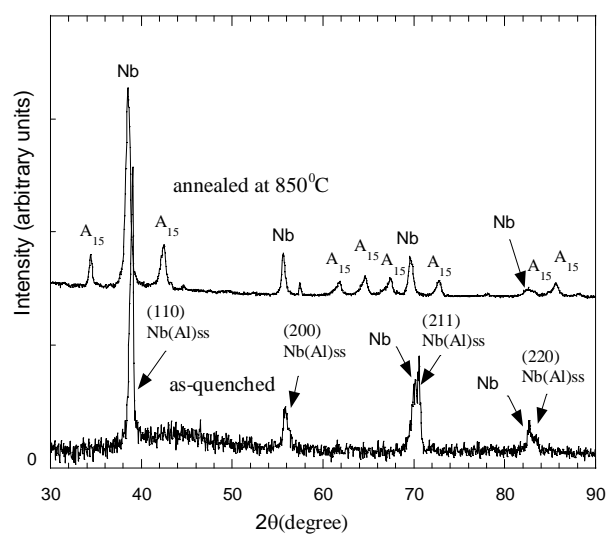


Figure 4. X-ray  $\theta - 2\theta$  scan both for the as quenched and for the samples annealed at 850 °C for 10 h.

A typical X-ray diffraction pattern of a multifilamentary cable manufactured by PIT technique and heat treated at 950 °C for 3 days is shown in Figure 5. The peaks observed in XRD are mainly due to Nb. Nevertheless, small intensity peaks corresponding to Nb<sub>3</sub>Al can also be noted. Taking into account the SEM analysis, one can conclude that the small amount of A15 phase is most probably due to the Al diffusion into Nb sleeves. Therefore, to overcome this problem, is necessary to start from Al rich powders.

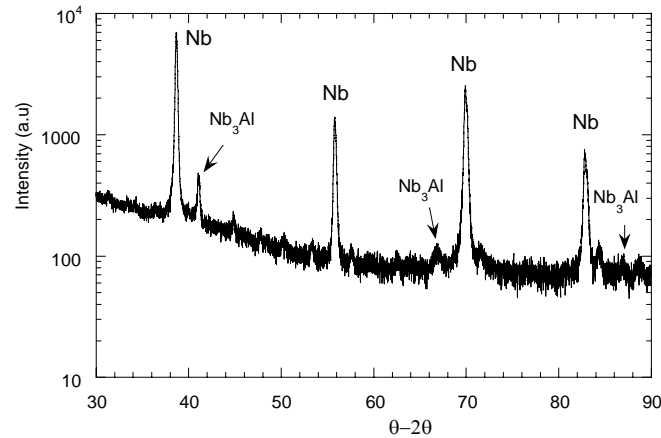
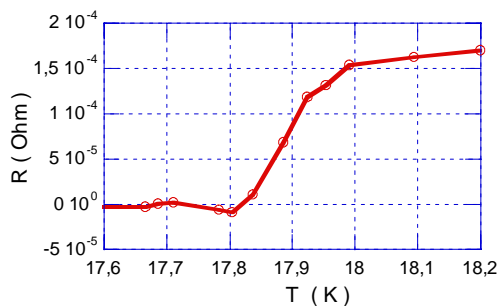


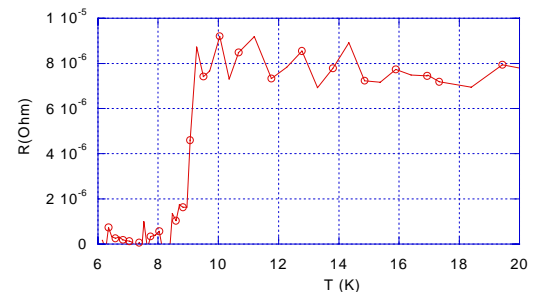
Figure 5. X-ray  $\theta - 2\theta$  scan for a short PIT multifilamentary wire annealed at 950 °C for 3 days.

### 3.3 The superconducting properties

The Figure 6 shows the resistance vs. temperature curve  $R(T)$  both for JR after the RQHT thermal process (Figure 6a) and for a PIT cable annealed at 950 °C for 3 days (Figure 6b). The onset of the transition for JR cable is at about 18 K, with a zero resistance temperature of 17.8 K, indicating the presence of the near stoichiometric  $Nb_3Al$  phase. The PIT cable annealed at 950 °C for 3 days exhibits a zero resistance temperature of about 9 K, indicating a non-stoichiometric A15.



(a)



(b)

Figure 6. The temperature dependence of the electrical resistance for (a) JR and (b) PIT wire.

For the JR sample the magnetisation cycles have also been measured by using a Vibrating Sample Magnetometer (VSM). At fields lower than 3 T and at  $T=4.2$  K, cycles are fragmented with the saw tooth shape typical of flux pinning jumps. In these conditions, cycles are not closed and losses cannot be calculated. From the magnetisation loop acritical current have been calculated. The obtained values are 1100 A/mm<sup>2</sup> at 6 T and 4.2 K, 400 A/mm<sup>2</sup> at 10 T and 4.2 K, 150 A/mm<sup>2</sup> at 1 T and 15 K.

These values are about a factor of two less than best results published in the literature, which could be ascribed to the low accuracy of the indirect method of measurement, however the lack of optimisation of the procedure could not be ruled out.

#### 4. Conclusions

Finally, we can conclude the following:

1. The Nb/Al precursor strands have been fabricated both by the jelly-roll (JR) and the powder-in-tube (PIT) technique.
2. The low temperature treatment has been used for the PIT strand, while the RHQT process has been used for the JR strand. The RHQT is a two-step process. First, the sample is heated for a short time at 2000°C and then is rapidly cooled in order to retain the bcc phase. A second step consists in a low temperature heat treatment (800 °C for 12 hours) to transform the bcc into the A15 superconducting phase.
3. For the JR strand rapid heated at 2000°C the (Nb(Al)ss) is mainly transformed into Nb<sub>3</sub>Al after a thermal treatment at 800 °C for 12 hours.
4. The X-Ray diffraction measurements have revealed that for the multifilamentary cable manufactured by the PIT technique and heat-treated at 950 °C for 3 days, only a small quantity of A15 superconducting phase is formed.
5. The onset of the transition for JR cable is at about 18 K, with a zero resistance temperature of 17.8 K, indicating the presence of the near stoichiometric Nb<sub>3</sub>Al phase. The PIT cable annealed at 950 °C for 3 days exhibits a zero resistance temperature of about 9 K, indicating a non-stoichiometric A15.