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Journal of Nuclear Materials 271&272 (1999) 508–511

journal of nuclear materials

Manufacturing technique of Nb_3Al super-conductive sheet by electrically heated powder rolling

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Abstract

The conventional manufacturing processes of superconductive wires or sheets are complex. Nb_3Al is a well-known superconductive material. Nb_3Al could be produced from powder directly by an advanced powder rolling technique. The experimental apparatus is composed of a powder rolling mill within a vacuum chamber and a direct current power supply unit. Powder is supplied into the roll gap and heated by applying electricity during rolling. The method is called 'electrically heated powder rolling'. Nb–25.4Al (mol%) powder of which particle size was under 0.15 mm was prepared by plasma melt gas atomizing (PMGA) technique. The powder consists of super-saturated solid solution of Nb and Al. But after electrically heated powder rolling, the existence of Nb_3Al (A15) phase was confirmed by X-ray diffraction. The sheet was porous and the thickness was about 0.7 mm. The sheet was re-rolled to the thickness of about 0.5 mm, then pore of the sheet was eliminated. The resistivity of the re-rolled sheet was measured at lower temperature than RT and $Tc_{(0)}$ of 15 K was confirmed. It is believed that electrically heated powder rolling is effective manufacturing technique for superconductive sheet such as intermetallic compounds.

1. Introduction

Superconductive wires or sheets have been produced by various methods [1,2]. However, it is considered that the conventional manufacturing processes of superconductive wires or sheets are complex. It is due to the difficulty of plastic deformation work for the intermetallic compound superconductive materials such as Nb_3Al having A15 phase.

The experimental apparatus [3] was made for producing Nb_3Al sheets. The producing method was an advanced powder rolling. Powder could be heated by electrical resistance heating during powder rolling, because electricity was applied between the two work rolls. Nb_3Al is a brittle material at RT, so it was thought that the rolling temperature should be increased over the range of the ductility. However, the sticking of the rolls and the powder often happened when rolling temperature was increasing with increasing current. It was supposed that the sticking was caused by the interaction of high rolling temperature, high rolling pressure, vacuum atmosphere and that the rolls were metals.

In the present work, the experimental apparatus has been reconstructed to prevent the sticking. Therefore, a high quality graphite was selected as the roll material, then Nb_3Al sheets could be continuously produced from powder directly not to happen the sticking. The producing method is called 'electrically heated powder rolling'.

2. Experimental apparatus

The concept of electrically heated powder rolling is shown in Fig. 1. The apparatus is composed of a powder rolling mill within a vacuum chamber and a direct current power supply unit. The diameter of each roll is 200 mm and the width is 25 mm. A hopper for supplying powder into the roll gap formed between the rolls is installed on the rolls. The powder is naturally supplied into the roll gap by gravity. During powder rolling, the powder is directly heated by applying electricity between

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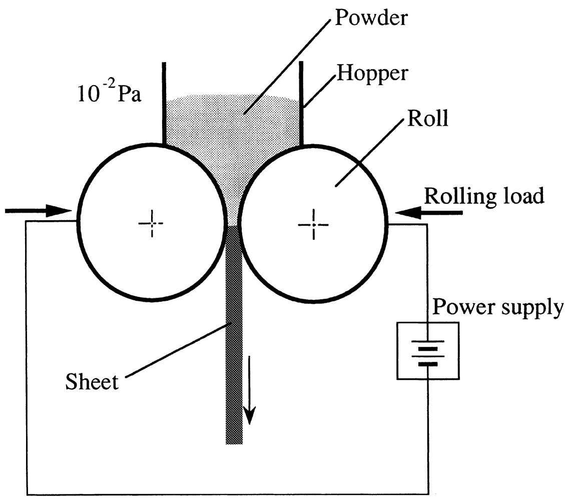

Fig. 1. Concept of electrically heated powder rolling.

the work rolls which are connected to a direct current source. The core of the roll shaft is cooled by water to prevent excessive heating. The process is carried out in a vacuum for preventing oxidation of powder and roll. Because it was considered that the rolling temperature required was above 1873 K, in the ductile temperature range of Nb_3Al. The roll material was the graphite of which the strength is higher than Nb_3Al above 1873 K.

3. Experimental method

Nb–25.4Al (mol%) powder produced by plasma melt gas atomizing (PMGA) technique [4] was prepared for producing Nb_3Al sheet. Fig. 2 shows a SEM image of the PMGA'd Nb–Al powder. The powder is a spherical shape. The particle size of the powder was less than 0.15 mm.

The experiment was performed as follows. The powder was put into the hopper, and the pressure of inside the chamber was reduced to 10^{-2} Pa. Then, the rolls were rotated at 1.6 mm/s as a roll surface speed, and the rolling load per roll width was set at 2.7 kN/mm by controlling the roll gap level. The current per roll width of 160 A/mm was applied to the rolls, producing sheet from the powder. The sheet formed was re-rolled with applying a current per roll width of 176 A/mm. A roll surface speed and a rolling load per roll width were respectively 1.1 mm/s and 3.7 kN/mm during re-rolling.

The resistivity of the re-rolled sheet was measured below RT by a conventional 4 terminal method. A size of the specimen for the measurement was about 6 mm × 3 mm, and a thickness was about 0.5 mm. The applied current was 100 mA.

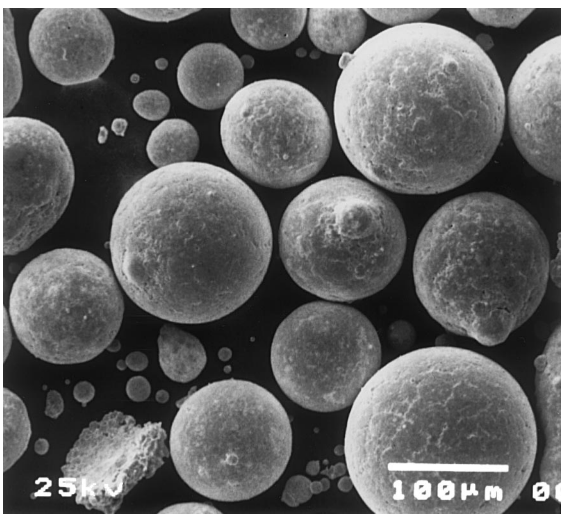

Fig. 2. SEM image of PMGA'd Nb–Al powder.

4. Results and discussion

The sheet produced by electrically heated powder rolling is shown in Fig. 3. The sheet was stably produced, and the thickness was about 0.7 mm. The width of the sheet was 25 mm. The thickness of the sheet formed by re-rolling was about 0.5 mm. The cross sections of the sheet formed by electrically heated powder rolling and re-rolled with applying electricity are shown in Fig. 4. The former was porous sheet, but pore of the sheet was eliminated in the latter. It was supposed that the current of direct heating was not enough for producing dense sheet. So, the flow stress of the material might be higher than the rolling pressure, because the rolling temperature was not enough high.

X-ray diffraction patterns of the Nb–25.4Al powder, the surface of the sheet produced by electrically heated powder rolling and the surface of the re-rolled sheet are shown in Fig. 5, respectively. The A15 phase of Nb_3Al was not observed in the powder, but it showed only bcc phase of Nb. It has been confirmed that PMGA'd Nb–Al powder consists of the supersaturated solid solution of Nb and Al because of quench effect [4]. In the sheet formed by electrically heated powder rolling, Nb_3Al phase and Nb phase were mainly observed. And a little Nb_2C phase was also observed, because graphite was used as the roll material. In the re-rolled sheet, it was confirmed that Nb phase decreased, because the quench

Fig. 3. A sheet formed by electrically heated powder rolling.

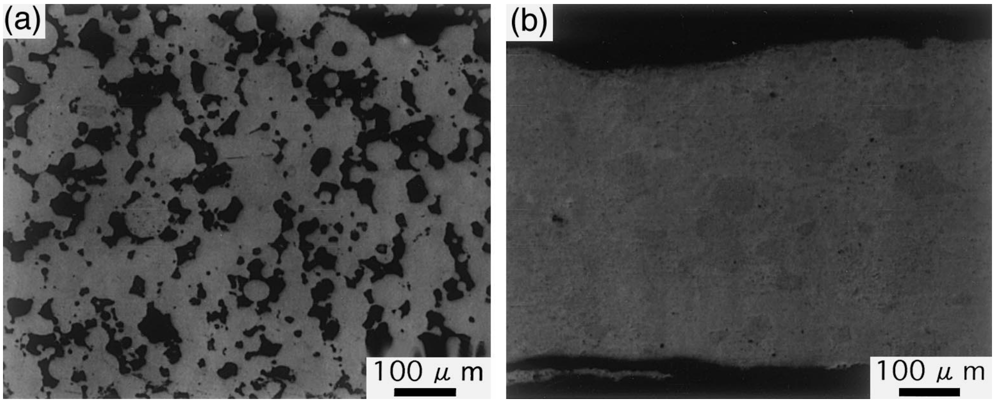

Fig. 4. Cross sections of Nb–25.4Al sheets.(a) as powder rolled at 160 A/mm, (b) as re-rolled at 176 A/mm.

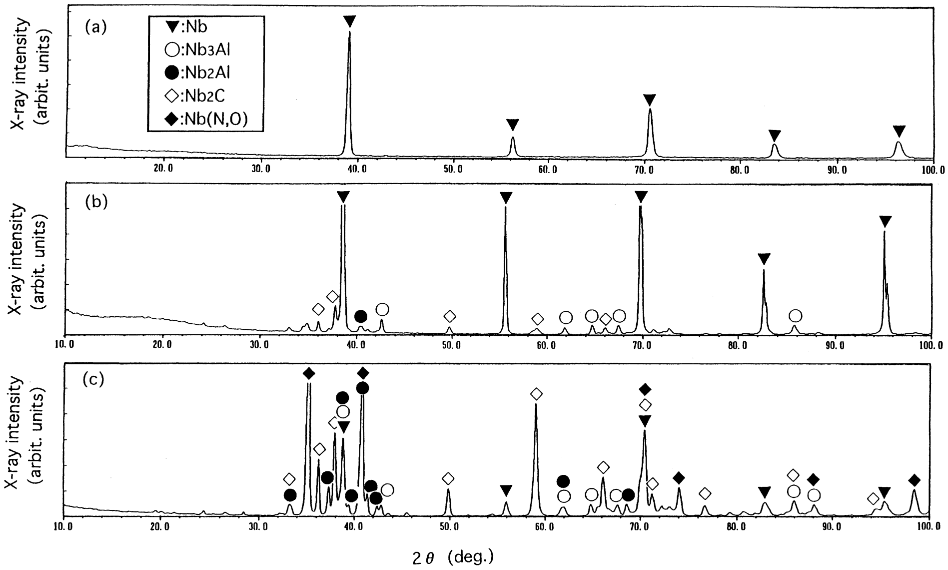

Fig. 5. X-ray diffraction patterns. (a) PMGA'd Nb–25.4Al powder. (b) Surface of Nb–25.4Al sheet formed by electrically heated powder rolling. (c) Surface of the re-rolled sheet.

effect of the powder could not be kept during re-rolling. The X-ray diffraction patterns of the sheets show the surface part of the sheet, therefore, it was considered that several phase appeared in the surface of the sheet by interaction of the powder and the graphite rolls. But it was supposed that the interaction occurred only surface part of the sheet.

The resistivity of the re-rolled sheet is shown in Fig. 6. The behavior of resistivity was dependent on temperature such as a metallic type superconductivity. $Tc_{(0)}$ was confirmed at 15 K, and the gradient of resistivity around $Tc_{(0)}$ was very sharp. Susceptibility of the sheet was also measured by SQUID measurement. It is shown in Fig. 7. The Meissner effect was observed by the change of the susceptibility less than 17 K. Therefore it was supposed that the major phase of the sheet showed

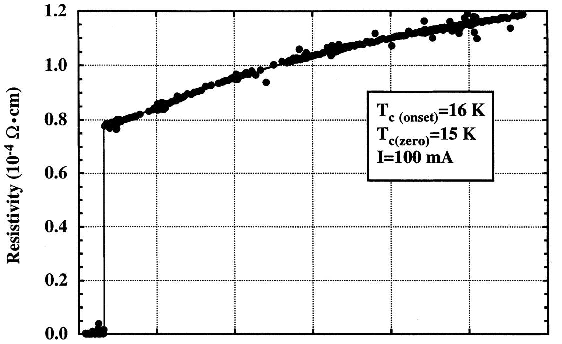

Fig. 6. Resistivity of Nb–25.4Al re-rolled sheet.

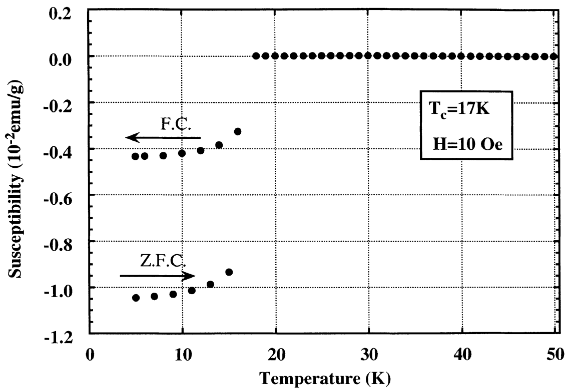

Fig. 7. Susceptibility of Nb–25.4Al re-rolled sheet.

superconductivity and it was A15 of Nb_3Al. It is believed that a long and a wide Nb_3Al sheet can be produced by electrically heated powder rolling.

5. Conclusions

1. Nb_3Al sheets could be produced using graphite rolls by electrically heated powder rolling.
2. The produced sheet of Nb–25.4Al showed superconductivity below 15 K.
3. It is believed that electrically heated powder rolling is effective for manufacturing superconductive sheet.

Acknowledgements

The authors are grateful to Professor J. Akimitsu and Mr Y. Zenitani of Aoyama-Gakuin University for supporting measurements. And we are also grateful to Dr M. Ayabe and Dr N. Kubota of Ishikawajima–Harima Heavy Industries Co., Ltd. for fruitful discussions.

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