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Development of Ag-sheathed Bi2223 superconducting wires

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At present only one candidate of High-Tc superconducting (HTS) wire which we expect for practical use is $(Bi,Pb)_2Sr_2Ca_2Cu_3O_X$ (Bi2223) superconducting wires. we have developed semi-mass producing long wires (in the kilometer class) with high J_c. But it is still necessary for practical use to improve performance of Bi2223 wires. On the sintering process, there are many problems which degraded the performance by decreasing density, interfering with reactivity and so on. So our efforts were focused on sintering process and applied pressure sintering method. The effects of pressure sintering method are (1) almost 100% relative density, (2) Bi2223 superconducting phase homogeneity, (3) no pores and no defects in filaments (good connectivity). Pressure sintering method is promising method to improve performance of Bi2223 superconducting wires.

Key words: Bi2223 wires, Pressure sintering, Relative density, Bi2223 phase homogeneity, Connectivity,

1. INTRODUCTION

Sumitomo Electric Industries (SEI) has participated in numerous projects, which aiming at power transmission cables, cryocooler-cooled magnets for research and industrial use, using Bi2223 wires. Many have already been successfully achieved. The main applications in which SEI was engaged were a 7 Tesla cryocooler-cooled magnet [1], 1MVA-class power transformer [2], a cryocooler-cooled magnet for silicon single-crystal growth [3], a 100m 3-phase power transmission cable prototype [4], and so on. Recently 1 year long verification test of 100m 3-pahse cable was succeeded. And we have developed long length Bi2223 wires and the Ic is 110A and engineering current density Je is $12.3kA/cm^2$ at 77K in a self-magnetic field. The unit length is about 550m [5].We have improved the performance of long length silver sheathed multi-filamentary Bi2223 superconducting wires and established a method of evaluation.

It is still necessary for practical use to improve property of Bi2223 wires. Jc value is confirmed 300,000 A/cm2 at local regions in Bi2223 monofilamentary tape [6]. So we could achieve improvement of performance of Bi2223 wires if high Jc local regions could increase. Basic manufacturing process of Bi2223 wires is established (powder, drawing, rolling, and sintering process: PIT method) and we have to optimize each process for improvement of performance. There are three factors mainly affected for performance of Bi2223 wires, Bi2223 phase homogeneity, grain alignment and connectivity. In the case of homogeneity and alignment, we showed a strong correlation between critical current and homogeneity [7] or grain alignment [8, 9] and we reported the highest Jc was obtained for the largest Bi2223 content case or the smallest misalignment angle case, respectively. Connectivity is the most important factor for Jc. Pores cause poor connectivity in filaments because supercurrent can not flow through pores. These pores are formed by growing Bi2223 phase during sintering process.

In this paper, we focus on sintering process. There are problems on the sintering process that (1) density is decreased by growing Bi2223, (2) reactivity of Bi2223 phase is interfered by decreasing density, (3) defects by rolling can not be recovered perfectly and so on. So we apply pressures sintering method for solution of issues concerning sintering process. Pressure sintering method is curried out under gas pressure.

2. Experiment

Ag-sheathed Bi2223 superconducting wires are produced using PIT method. Bi2223 multifallamentry wires used in this paper were samples after first heat treatment and intermediate rolling process. Silver ratio is

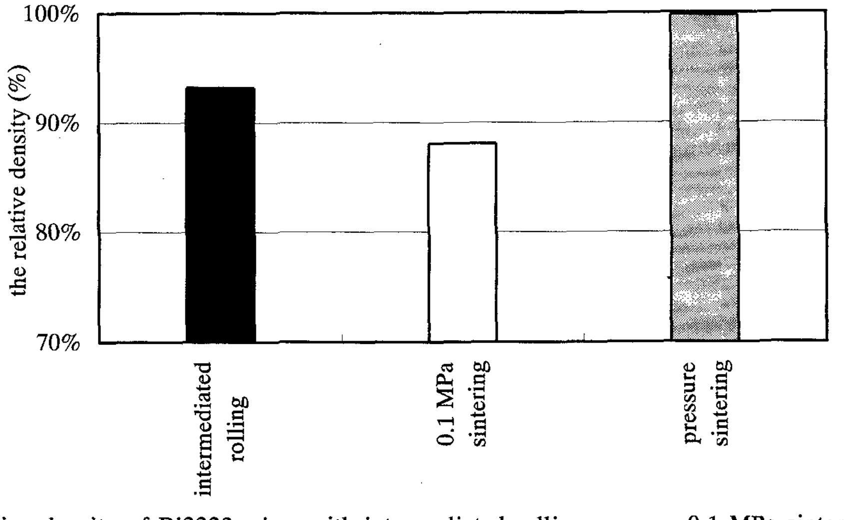

Fig.1. The relative density of Bi2223 wires with intermediated rolling process, 0.1 MPa sintering process and pressure sintering process.

1.5-2.0 and sample length is 1 m. Samples are not coated or in Ag foil like pressure sintering test by another group [10]. Our pressure sintering system is a flow system with a booster and uses a gas mixture of inert gas and O_2.In second heat treatment, processing total pressure and oxygen partial pressure are controlled independently.

The relative density was calculated by Archimedes method. The microstructure was observed with SEM. DC magnetization was measured with SQUID using crushed samples by press to examine Bi-based Superconducting contents (Bi2223, Bi2212 and Bi2201 contents) in filaments [11,12]. Defects in filaments were observed with Magneto-Optical imaging (MO) method using polished samples to image filament structure. I_c was determined from self-field 77 K I-V curves and I_c criteria was 1 V cm^{-1}. And tensile stress tolerance test was curried out. Ic was measured at 77 K using stressed samples at room temperature.

3. Results and discussion

3.1 Relative density

Figure 1 shows the relative mass density of Bi2223 wires of intermediated rolling, 0.1 MPa sintering and pressure sintering samples. 0.1 MPa sintering means standard sintering process. The ideal mass density of the Bi2223 phase in this study is 6.3 g cm^{-3} [13]. The relative density of Bi2223 wires with 0.1 MPa sintering and pressure sintering were about 88 % and almost 100 %, respectively. This means that the problem which density decrease on the sintering process was improved. Then considering that the relative density of intermediated rolling was about 93 %, the effect of pressure sintering not only prevent decreasing of density by growing Bi2223 phase but also increase density compared to density of intermediated rolled sample.

3.2 Bi2223 phase homogeneity

Figure 2 shows SEM image of (a) 0.1 MPa sintered sample and (b) pressure sintered sample. Gray regions in SEM show Bi2223 phase. Fig. 2 (a) shows many non-superconducting phases and pores which degrade performance in filaments, and fig. 2 (b) shows large gray regions and no pores in filaments. This means Bi2223 phase tends to become homogeneous by pressure sintering method. Increase of density in filaments accelerates the reactivity of Bi2223 phase. And there are no pores in fig. 2 (b) showing the relative density is almost 100%.

Furthermore fig. 4 shows Bi2212 content calculated from SQUID magnetization curves in fig. 3. SQUID characterization method was suggested by Huang *et, al.* [14]. They showed a strong correlation between critical current density and residual Bi2212 phase in filaments and suggested that higher levels of performance would be achieved by eliminating residual Bi2212 phase. As can be seen in Fig. 4, Bi2212 content of Bi2223 wires with pressure sintering process decreased compared to 0.1 MPa sintered sample. And in fig. 3, signal of Bi2201 phase (T_c ~ 10 K) can not be seen. Pressure sintering method tends to accelerates Bi2223 phase homogeneity from the results of fig. 3 and fig. 4. But, a few non- superconducting phase (fig. 2) and residual Bi2212 content (fig. 3) are observed in filaments. It is necessary for improvement of performance to optimize pressure sintering method as Bi2223 phase will become homogenous.

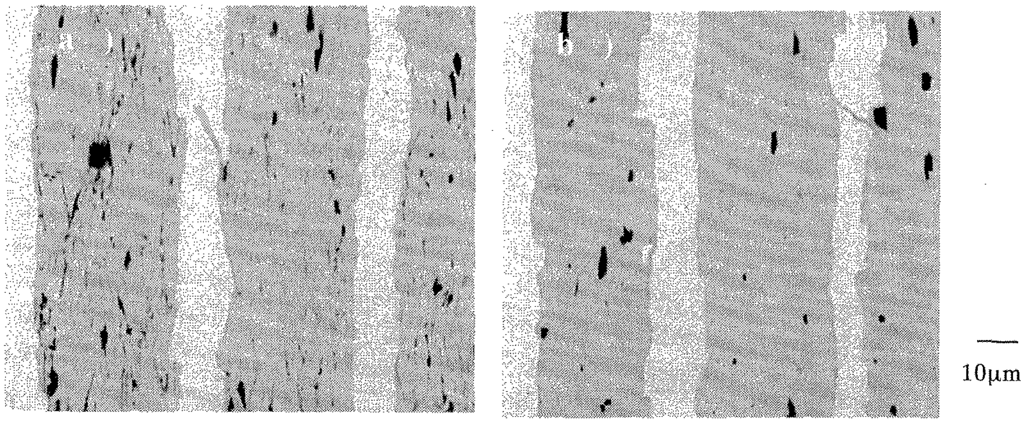

Figure 2. SEM image of (a) 0.1 MPa sintered sample and (b) pressure sintered sample. The gray regions, black regions, white regions and tiny irregular black regions are Bi2223, alkaline earth cuprates (AEC), Ca-Pb-O and pores, respectively.

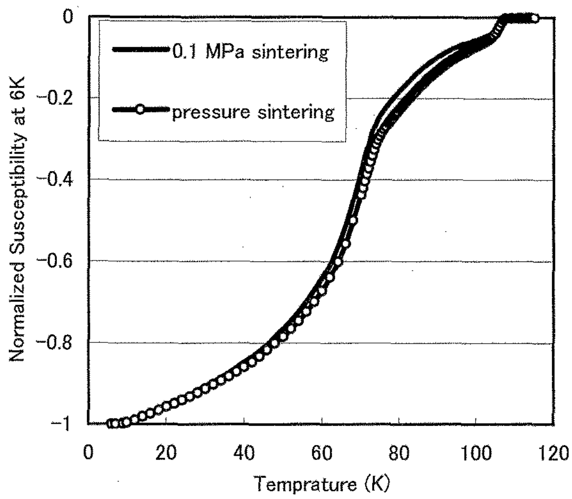

Figure 3. DC magnetization curves of 0.1 MPa sintered sample and pressure sintered sample using SQUID. Magnetic field of 10G applied parallel to the wire surface.

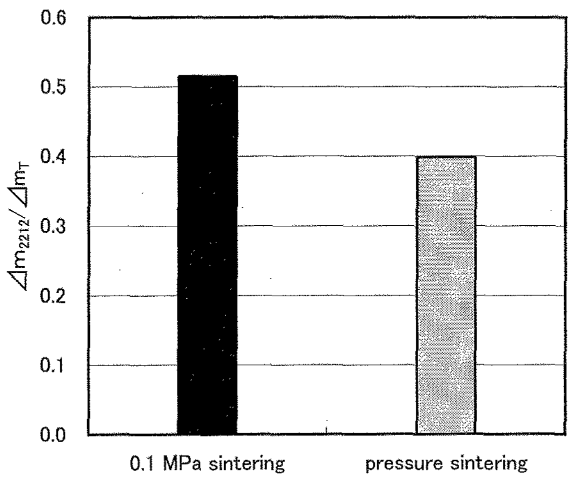

Figure 4. Bi2212 content in filaments from DC magnetization curves in fig. 3.

3.3 Connectivity

Figure 5 shows Magneto-Optical image of (a) 0.1 MPa sintered sample and (b) pressure sintered sample. Magnetic field was applied 800 G perpendicular to wire surface at10 K after zero field cooling (ZFC). There are many defects indicated by arrows in filaments of 0.1 MPa sintered sample. As supercurrent flows through defects as can be seen in fig. 5 (a), these defects concern to poor connectivity and high Jc can be achieved only in the non defect part of the filament.

Pressure sintering method is able to suppress such defects in filaments as can be seen in fig. 5 (b). Contrast in filaments of pressure sintered sample is darker than that of 0.1 MPa sintered sample. This means that filaments shielded magnetic field and the phase in filaments becomes strong connected superconducting phases.

Pressure sintering method is effective method for improvement of performance from results of this content and then critical current density is 30,000 A cm^{-2} on 0.1 MPa sintering process and 40,000 A cm^{-2} on pressure sintering process. So critical current density becomes 130 % and more by pressure sintering method.

3.4 Tensile stress

Figure 6 shows tensile stress tolerance of 0.1 MPa sintered sample and pressure sintered sample. Low Ag ratio sample was used. Tensile stress value which I_c/I_{c0} starts to decrease was 85 MPa and 135 MPa for 0.1 MPa sintered sample,

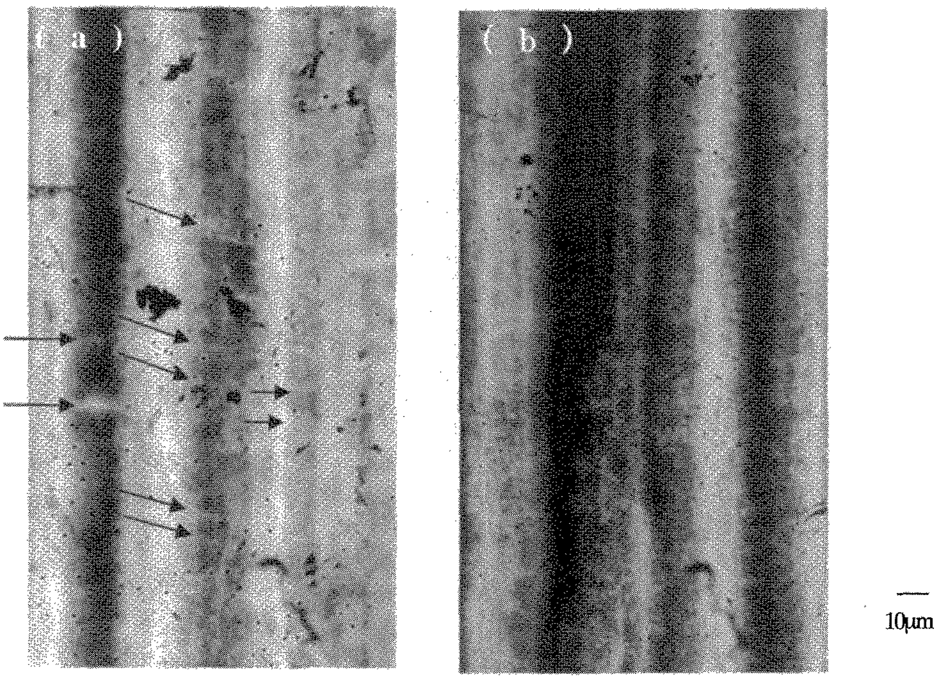

Figure 5. Magnet-Optical image of (a) 0.1 MPa sintered sample and (b) pressure sintered sample at T=10 K and H=800 G. The parts indicated by arrows are defects.

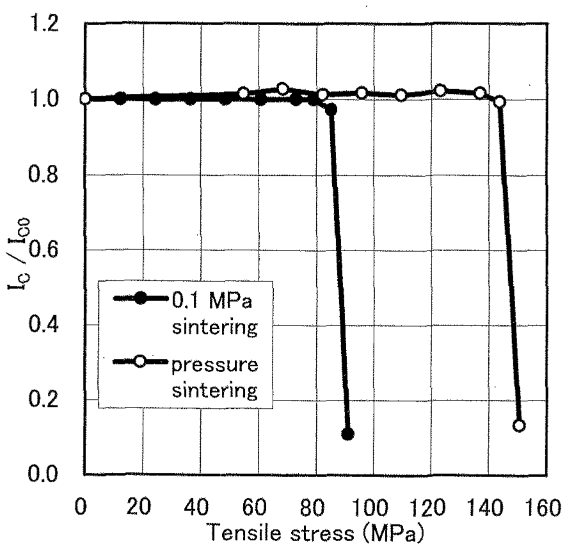

Figure 6. Tensile stress tolerance of 0.1 MPa sintered sample and pressure sintered sample. I_c and I_{c0} mean critical current after tensile stress and without tensile stress, respectively.

and pressure sintered sample, respectively. Tensile stress of Bi2223 wires increases because there are no pores and defects as can be seen in fig.2 (b) and fig. 5(b) by pressure sintering. Mechanical property is fundamental for application and mechanical property as is the case with homogeneity and connectivity is also improved by pressure sintering.

4. Summary

Sumitomo Electric industries focused on the sintering process for improvement of performance of Bi2223 superconducting wires and then applied the pressure sintering method. The results of the pressure sintering method summarized as follows:(1) the relative density was almost 100 %, (2) Bi2223 phase tented to become homogeneous and (3) connectivity was improved by no pores and no defects. At present, critical current density of Bi2223 wires on the pressure sintering method was 130 % and more as high as that of the standard process. The pressure sintering method was effective method for improvement of mechanical property (tensile stress) and this improvement correlated closely with increasing density in filaments. Mechanical property is one of important performance for practical use of Bi2223 wires. Considering improvement of Bi2223 phase homogeneity, connectivity, critical current density and mechanical property, increasing relative density is effective for improvement of performance of Bi2223 wires and the pressure sintering method is promising for practical use of Bi2223 wires.

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References

[1] T. Kato, K. Ohkura, M. Ueyama, K. Ohmatsu, K. Hayashi, and K. Sato, in: Proceedings of Fifteenth International Conference on Magnet Technology, Science Press, p.793, (1998)

[2] K. Funaki, M. Iwakuma, K. Kajikawa, M. Hara, J. Suehiro, T. Ito, Y. Takata, T. Bohno, S. Nose, M. Konno, Y. Yagi, H. Maruyama, T. Ogata, S. Yoshida, K. Ohashi, H. Kimura, and K. Tsutsumi, *IEEE. Trans. Appl. Supercond.*, **11**, 1578, (2001).

[3] M. Ono, K. Tasaki, Y. Ohtani, T. Kuriyama, Y. Sumiyoshi, S. Nomura, M. Kyoto, T. Shimonosono, S. Hanai, M. Shojyu, N. Ayai, T. Kaneko, S. Kobayashi, K. Hayashi, H. Takei, K. Sato, T. Mizuishi, M. Kimura, and T. Masui, 17^{th} International Conference on Magnet Technology, (2001)

[4] T. Masuda, T. Kato, H. Yumura, M. Watanabe, Y. Ashibe, K Ohkura, C. Suzawa, M. Hirose, S. Isojima, K. Matsuo, S. Honjo, T. Mimura, T. Kuramochi, Y. Takahashi, H. Suzuki, and T. Okamoto, *Physica C*, **372-376**, 1580, (2002).

[5] J. Fujikami, T. Kaneko, N. Ayai, S. Kobayashi, K. Hayashi, H. Takei, and K. Sato, *Physica C*, **278-281**, 1061, (2002).

[6] E. E. Hellstrom, ISS2002, PL-2.

[7] S. Kobayashi, T. Kaneko, N. Ayai, K. Hayashi, H. Takei, and R. Hata, *Physica C*, **357-360**, 1115, (2001).

[8] T. Kaneko, S. Kobayashi, K. Hayashi, and K. Sato, *Advances in Superconductivity* (Tokyo: Springer), p907, (1997).

[9] S. Kobayashi, T. Kaneko, T. Sashida, M. Ueyama, K. Hayashi, and K. Sato, *Advances in Superconductivity* **X** (Tokyo Springer), p909, (1998).

[10] Y. Yuan, R. K. Williams, J. Jiang, D. C. Larbalestier, X. Y. Cai, M. O. Rikel, K. L. DeMoranville, Y. Huang, Q. Li, E. Thompson, G. N. Riley, and E. E. Hellstrom, *Physica C*, **372-376**, 883, (2002).

[11] T. Kaneko, S. Kobayashi, and K. Sato, *Advances in Superconductivity* (Tokyo Springer), p789, (1994).

[12] T. Kaneko, S. Kobayashi, and K. Sato, *Advances in Superconductivity* (Tokyo Springer), p807, (1995).

[13] Q. Y. Hu, H. K. Liu, and S. X. Dou, *Appl. Supercond.*, **4**, 17, (1996).

[14] Y. B. Huang, X. Y. Cai, G. N. Riley, Jr, D. Larbalestier, D. Yu, M. Teplitsky, A. Otto, S. Fleshler, and R. D. Parrella, CP614, *Advances in cryogenic Engineering*, **48**, 717, (2002).

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