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Effect of intermediate rolling on the microstructure and J_e performance of Ag-sheathed Bi-2223 tapes

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Abstract

The effect of intermediate rolling (IR) deformation on the microstructure and engineering critical current density, J_e (77 K, 0 T), has been investigated on Ag-sheathed Bi-2223 multifilamentary tapes. The experiment results showed that intermediate rolling reduces porosity, breaks down large deleterious secondary phases, and improves the alignment of Bi-2223 grains. All these effects strengthen the grain connectivity and therefore enhance the J_e value of the tape. Degradation in J_e with a further increase in IR reduction is caused by the introduction of more cracks that cannot be completely healed in subsequent heat treatment. J_e reaches a maximum value with an optimum IR reduction, which is determined by a competition between grain connectivity improvement and introduction of cracks. As a function of IR reduction, the degree of texturing in HTS oxide core also shows a similar peak value. XRD analysis demonstrates that the optimum IR reduction depends also on the volume fraction of Bi-2212 after the initial heat treatment.

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Keywords: Ag-sheathed Bi-2223 tape; Intermediate rolling; Engineering critical current density; Degree of texturing

1. Introduction

As the most promising material at the moment for superconducting electrical engineering applications, Ag-sheathed Bi-2223 tapes, widely fabricated by powder in-tube (PIT) process [1,2], face three major transport current-limiting factors: the

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Bi-2223 phase purity, grain alignment and grain connectivity [3]. To improve both the reproducibility and critical current of such tapes, a tremendous amount of effort has been made in optimizing important process parameters including sintering temperature, soaking time, oxygen partial pressure, heating and cooling rate, and precursor powder processing [4–8]. In contrast, the intermediate deformation, a vital step between heat treatments, is much less reported and understood [9].

The intermediate deformation improves the alignment of grains, increases the density of the HTS oxide core, breaks up the sintered structure, brings the reactants into more intimate contact, and facilitates the Bi-2212 to Bi-2223 phase conversion [9–13]. Too high a deformation reduction can, however, create more cracks that cannot be healed completely in the subsequent heat treatment [9]. Optimization of intermediate deformation process may also therefore depend on the interaction of other parameters [11]. A significant correlation between the rolling pressure and the critical current density (J_c) has been recently reported for Ag-sheathed Bi-2223 tapes [14,15].

Pressing usually results in higher J_c value (with a maximum of 73 kA/cm^2 reported at 77 K, 0 T [16]) than cold rolling (with a maximum of 40 kA/cm^2 reported at 77 K, 0 T [17]). Marti et al. demonstrated that by means of the new process called "Periodic Pressing", J_c values up to 35 kA/cm^2 were obtained for multifilamentary Bi-2223 tapes of several meters at 77 K, 0 T. However, rolling is scaleable to long lengths and so far the most common method of intermediate deformation in commercial production of Bi-2223 tapes.

In this study, the influence of intermediate rolling (IR) characteristics such as rolling force, roll diameter, rolling reduction, and lubrication condition, has been investigated together with the effect of initial heat treatment, on the microstructure and J_e of Ag-sheathed Bi-2223 tapes.

2. Experimental

2.1. Fabrication of superconducting tape

A 37-filament Bi-2223/Ag tape was made using standard powder in tube (PIT) processes. A pure Ag tube was filled with precursor of $Bi_{1.8}Pb_{0.33}Sr_{1.87}Ca_2Cu_3O_x$ composition, with a powder packing density of ~40%. After being drawn into round wires of 2 mm in diameter and cut into 37 monofilaments, which were then re-packed into a Ag alloy tube, the composite was subjected to the same drawing process to a final diameter of ∅ = 1.5 mm. The wire was then rolled to a flat tape 0.25 mm in thickness and 4 mm in width. A first heat treatment was performed at 830 °C, using a flowing 8.5% O_2-balance N_2 atmosphere. Subsequently further heat treatments were given, after an intermediate rolling step. Each intermediate rolling was performed in one pass. The force exerted on the tape was measured by means of a load cell fitted in the rolling shaft.

2.2. Sample preparation

To study the influence of IR characteristics on the microstructure and J_e of Bi-2223/Ag tape, a first group of samples were given an identical 20 h initial heat treatment. Intermediate rolling was then carried out using both 120 mm and 300 mm rolls, with rolling reductions varying from 5% to 30%. A lubricant was used during rolling on some samples, to make a comparison between rolling with and without lubrication. After the rolling deformation, the tapes were then given a final heat treatment of 100 h at 830 °C.

For the second group of samples, the initial heat treatment was carried out for three different soaking times (10 h, 20 h and 30 h), which converted approximately 47%, 84% and 95% respectively of the Bi-2212 to Bi-2223 phase. These tapes were then given an intermediate rolling, using 300 mm rolls, with the same range of reductions, from 5% to 30%, as used in the first group of samples. Finally an additional 110, 100 or 90 h heat treatment was performed to make up a total heat treatment time of 120 h for each sample.

2.3. Characterization

The critical current I_c was measured at 77 K (0 T) using a standard four-terminal technique and defined at a value of 1 μV cm^{-1}. The engineering critical current density J_e was obtained by dividing I_c by the overall cross-sectional area of the tape.

XRD analysis was performed using Cu Kα radiation. To determine the relative amounts of Bi-2223 and Bi-2212, we took the ratio of the maximum intensity of the Bi-2223 (0 0 10) peak to the Bi-2212 (0 0 8) peak. To study the development of *c*-axis texturing after final heat treatment, the FWHM of the (0 0 12) peak rocking curve was used as a measure of the degree of texturing. Polished transverse sections of the tapes were studied using LEO-1530 scanning electron microscope.

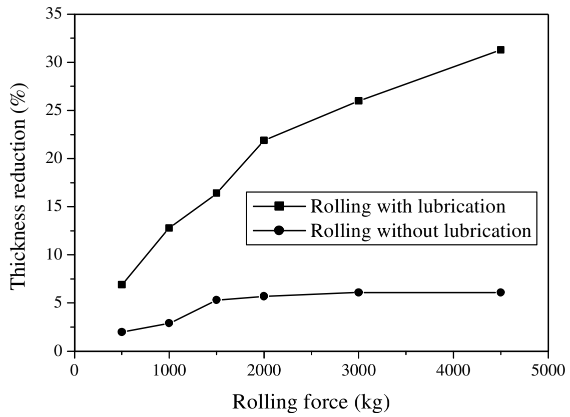

Fig. 2. Effect of lubrication on intermediate rolling reduction.

3. Results and discussion

3.1. Effect of IR characteristics on J_e

Fig. 1 shows the effect of intermediate rolling force on the engineering critical current density (J_e of the tape. The deformations have been carried out without lubrication. With increasing rolling force, J_e first increases but then remains almost constant as the rolling force further increases.

Fig. 2 compares the effect on thickness reduction of intermediate rolling with and without lubrication. For IR without lubrication, as the rolling force increases, the thickness reduction initially increases but eventually saturates. In contrast, for IR with lubrication, thickness reduction increases continuously with increased rolling force. Introduction of lubrication reduces the friction coefficient between the rolls and the tape, hence increases the thickness reduction.

From both Figs. 1 and 2 we can see that, in the case of deformation without deformation, after the rolling force reaches a critical value, the tape thickness is no longer reduced and no further changes is seen in J_e. Hence we can infer that the change in J_e does not correspond directly to the change in rolling force, but to the change in rolling reduction. The validity of this inference can be seen from the graph shown in Fig. 3, which plots J_e as a function of IR (with lubrication) reduction for different roll diameters. For 300 mm rolls, J_e increased from

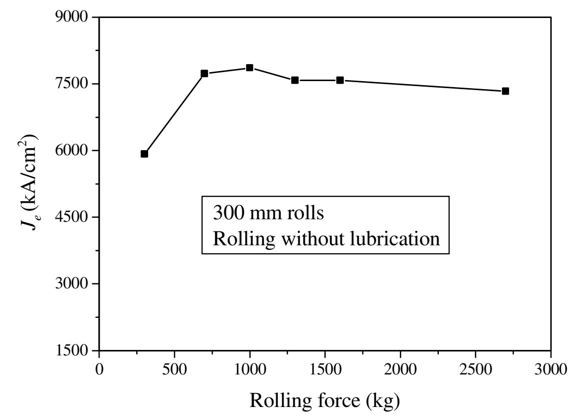

Fig. 1. Engineering critical current density versus intermediate rolling force with 300 mm rolls.

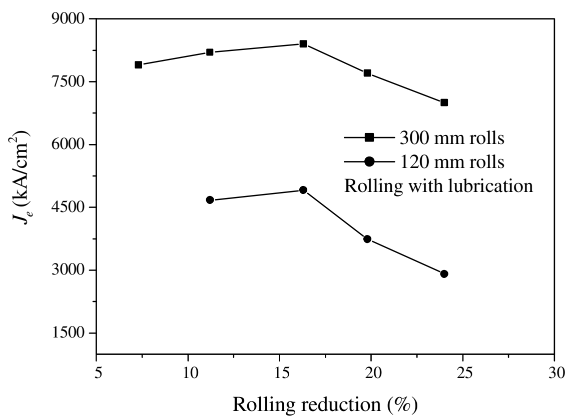

Fig. 3. Final engineering critical current density as a function of intermediate rolling reduction for tapes rolled with different rolls.

~7.5 kA cm^{-2} at a 7% reduction to a maximum of ~8.5 kA cm^{-2} at a 16% reduction. The J_e value decreased with further increase in reduction, due to the introduction of cracks. For intermediate rolling using 120 mm rolls, J_e was significantly lower at all reduction, the value being about two-thirds of that obtained using 300 mm rolls. One reason for the beneficial influence of larger rolls is that the contact area between the rolls and tape is larger, which causes the deformation to approximate more closely to uniaxial pressing, and causes more of the cracks to form to nearly parallel to the tape axis [9,18].

3.2. Effect of IR reduction on the tape microstructure

Fig. 4 shows the Bi-2223 phase volume fraction and degree of texturing evolution after the final heat treatment as a function of IR (with lubrication) reduction. The degree of texturing was determined from the FWHM of the (0 0 12) peak rocking curve, chosen because the (0 0 12) peak does not overlap with other peaks. As the rolling reduction increases, the final Bi-2223 volume fraction first increases, but then saturates. Fig. 4 also shows that a higher rolling reduction leads to improvement of texture. Too high a rolling reduction results, however, in a pronounced sausaging effect [18] and hence deteriorates the alignment of grains. The trend of texture change with IR reduction is similar to that of J_e versus IR reduction.

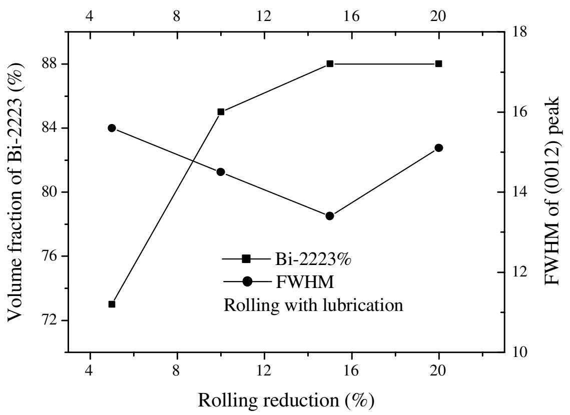

Fig. 4. The Bi-2223 phase volume fraction and degree of texturing after final heat treatment tapes rolled with different rolls.

Fig. 5(a) is a backscattered electron micrograph of as intermediate rolled tape with 5% reduction. The deformations have been carried out with lubrication. A large amount of porosity and intact AEC second phases are present in the section. However, very few cracks are seen. For as intermediate rolled tape with 20% reduction (Fig. 5(b)), although the degree of porosity is significantly reduced and most of AEC phases are crushed, severe cracks are formed. Even after being given the final heat treatment, there are still a number of residual or unhealed cracks existed in tape with 20% reduction (Fig. 5(d)), whereas for the 5% IR tape most of cracks have healed.

In summary, IR enhances the density of the HTS oxide core, disrupts large deleterious secondary phases, improves the texture of the tape and facilitates the Bi-2212 to Bi-2223 phase conversion. However, very large IR reduction results in large quantity of cracks that cannot be completely healed by a subsequent heat treatment.

3.3. Determination of optimum IR reduction from Bi-2212 volume fraction measurements

As demonstrated in Fig. 3, there exists an optimum IR reduction. An initial enhancement of J_e with IR reduction is ascribed to improvement of the grain connectivity. Further increase in IR reduction may create more and larger cracks which will degrade J_e. The healing of these cracks depends on the volume of liquid phase obtained in the subsequent heat treatment step, which in turn is dependant on the residual Bi-2212 phase after initial heat treatment.

Fig. 6 shows that the optimum IR (with lubrication) reduction increases with increasing residual Bi-2212 volume fraction after initial heat treatment. XRD analysis indicated that there was about 53%, 16% and 5% residual Bi-2212 phase for tapes given 10 h, 20 h and 30 h respectively of initial heat treatment. Correspondently, the optimum IR reduction values for these tapes are 25%, 15% and 10%, respectively. This behavior results from the fact that the more the unreacted Bi-2212 after initial heat treatment, the more the liquid phase

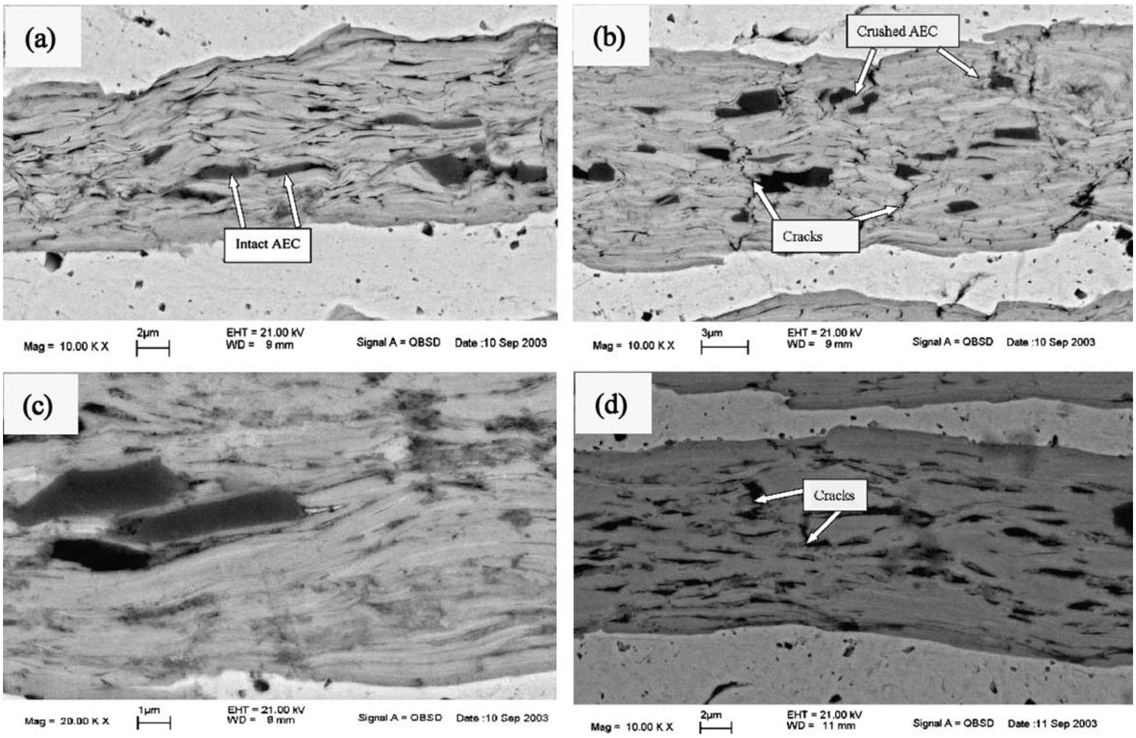

Fig. 5. Backscattered electron micrographs of (a) as intermediate rolled tape with 5% reduction, (b) as intermediate rolled tape with 20% reduction, (c) a tape with 5% IR reduction, after the final heat treatment, and (d) a tape with 20% IR reduction, after the final heat treatment.

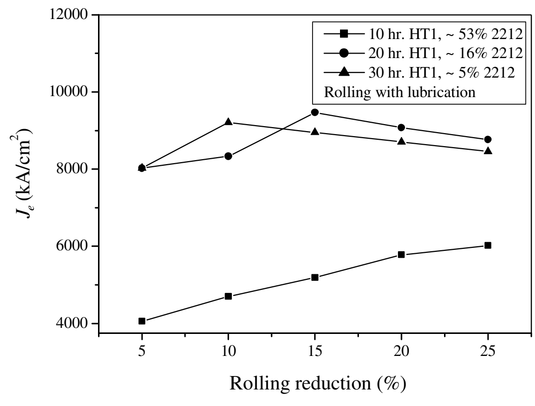

Fig. 6. Plots of final J_e versus IR reduction for tapes given different initial heat treatments (10 h, 20 h and 30 h). Optimum IR reduction increases with increasing residual Bi-2212 after initial heat treatment.

available during subsequent heat treatment, which leads to higher ability for crack healing, and therefore allows a larger optimum IR reduction.

It is clear that the effect of IR reduction on grain connectivity and hence J_e performance is in a competing manner. High IR reduction certainly increases the oxide core density, whereas excessive deformation will introduce large quantity of cracks. Thus, the maximum J_e performance should be correlated to an optimum IR reduction. Although such crack formation during IR deformation is inevitable, for further J_e improvement it is crucial to effectively heal these deformation induced cracks and understand the healing mechanisms.

4. Conclusion

Microstructure evolution and J_e performance of Ag-sheathed Bi-2223 tapes are thoroughly studied using different IR roll diameter, rolling force, rolling reduction, lubrication during IR deformation, and initial heat treatment conditions. The following results are obtained:

(1) J_e enhancement is closely correlated to rolling reduction during deformation, instead of rolling force.
(2) IR under lubrication conditions results in higher reductions than does IR without lubrication.
(3) Large rolls are more beneficial for J_e improvement than small ones.
(4) J_e reaches a maximum value with an optimum IR reduction, which has a competition fashion between improvement of the grain connectivity and introduction of cracking. As a function of IR reduction, the degree of texturing in Bi-2223 grains also shows a similar peak value.
(5) XRD analyses indicate that the optimum IR reduction increases as increasing the volume fraction of residual Bi-2212 after initial heat treatment.

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References

[1] H. Maeda, Y. Tanaka, M. Fukutomi, T. Asano, Jpn. J. Appl. Phys. 27 (1988) L209.
[2] K. Heine et al, Appl. Phys. Rev. Lett. 55 (1989) 441.
[3] Y. Yamada, J.Q. Xu, J. Kessler, R. Flükiger, Physica C 185 (1991) 2483.
[4] Y.L. Liu, W.G. Wang, H.F. Foulsen, P. Vase, Supercond. Sci. Technol. 12 (1999) 377.
[5] J.C. Grivel, R. Flükiger, J. Alloys Compds. 235 (1996) 53.
[6] J. Muller, O. Eibl, B. Fischer, P. Herzog, Supercond. Sci. Technol. 11 (1998) 239.
[7] D.C. Larbalestier, X.Y. Cai, Y. Feng, H. Edelman, A. Umezawa, G.N. Riley, W.L. Carter, Physica C 221 (1994) 299.
[8] L. Martini, Supercond. Sci. Technol. 11 (1998) 232.
[9] J.W. Anderson, X.Y. Cai, M. Feldmann, Supercond. Sci. Technol. 12 (1999) 617.
[10] J.A. Parrell, A.A. Polyanskii, D.C. Larbalestier, Supercond. Sci. Technol. 9 (1996) 393.
[11] R. Zhao, F. Darmann, G. MacCaughey, Supercond. Sci. Technol. 11 (1998) 1068.
[12] V. Beilin, A. Goldgirsh, E. Yashchin, Physica C 309 (1998) 56.
[13] P. Skov-Hansen, M.R. Koblischka, P. Vase, IEEE Trans. Appl. Supercond. 11 (2001) 3740.
[14] A. Oota, K. Kawano, IEEE Trans. Appl. Supercond. 9 (1999) 2529.
[15] G. Grasso, A. Jeremie, R. Flükiger, Supercond. Sci. Technol. 8 (1995) 827.
[16] M.W. Rupich, Q. Li, R.D. Parrella, M. Teplitsky, et al., The 1998 Int. Workshop on Supercond., Okinawa, 12–15 July 1998, p. 145.
[17] K. Sato, K. Ohkura, K. Hayashi, M. Ueyama, J. Fujikami, T. Kato, in: Proc. Int. Workshop on Advances in High Magnetic Fields, Physica B 216 (1996) 258.
[18] Z. Han, P. Skov-Hanson, T. Freltoft, Supercond. Sci. Technol. 10 (1997) 371.