

# High Field Performance of PbMo6S8 Superconductors

著者	渡辺和雄
journal or	IEEE Transactions on Magnetics
publication title	
volume	23
number	2
page range	1019-1021
year	1987
URL	http://hdl.handle.net/10097/47226

K.Hamasaki, \*K.Watanabe, T.Saito, T.Yamashita, T.Komata and \*K.Noto

Department of Electronics, Technological University of Nagaoka, Kamitomioka 1603, Nagaoka 940-21, Japan

\*The Research Institute for Iron, Steel and Other Metals, Tohoku University, Sendai 980, Japan

# ABSTRACT

Critical currents in Chevrel-phase lead molybdenum sulphide have been measured at two temperatures as a function of applied magnetic field up to 30 T. Improved critical current densities  $J_c$ 's were achieved with electroplating and diffusion processed PbMo<sub>6</sub>S<sub>8</sub> short-tapes. Increased  $J_c$  values were obtained for a short heat-treatment at 1050 °C. We find a  $J_c$  of  $\sim 5 \times 10^3$  A/cm<sup>2</sup> at 20 T and 4.2 K, and  $\sim 1 \times 10^4$  A/cm<sup>2</sup> at 20 T and 1.6 K. The dependence of  $J_c$  on grain size was also measured, and the correlation between flux pinning force and average grain size is discussed. The critical current density in the field range of < 27 T was strongly dependent on the grain size, and the inverse grain size.

# INTRODUCTION

Since the discovery of the extremely high critical field of Chevrel-phase PbHo<sub>g</sub>S<sub>a</sub>(PHS) superconducting compounds, much of the research directed toward the improvement of a critical current density  $J_c$  in this type of thin films, wires, tapes, and bulk samples.<sup>1-9</sup> For practical applications, the most important requirement is that it should carry a large dissipation-free transport current in a strong field. So far, the reported  $J_c$  values were not high enough, and also little is known of the metallurgy of this compound.

Very recently, high quality PMS wires were prepared with powder metallurgical technique based on molybdenum or tantalum matrix.<sup>10, 11</sup>The  $J_c$  values of these wires are relatively high compared with those reported in early wires? Thus application of the PMS materials for very high fields appears promising in the near future.

In the present paper we report on recent improvements of high field properties of the PMS short-tapes prepared by electroplating and diffusion techniques. We also present critical current density vs grain size measurement on PMS materials.

# EXPERIMENTAL RESULTS AND DISCUSSION

The measurement of the critical current was carried out in 16.5 T superconducting magnet, and three Hybrid Magnets (HM=1-3) at the High Field Laboratory for Superconducting Materials(HFLSM) at Tohoku University.<sup>12</sup> The typical examples of critical current densities are

The typical examples of critical current densities are shown in Fig.1 as a function of applied magnetic field in field up to 27 T, and for 4.2 and 1.6 K. The applied field direction was parallel to tape plane. The criterion for J<sub>c</sub> was 5  $\mu$ V/cm. Samples were prepared by an electro-plating and diffusion methods. Reaction times were 4 and 42 hrs, and reaction temperature was~1050 °C.<sup>13</sup>

The  $J_c$ 's of these samples are strongly dependent on the reaction time as shown in this figure. The  $J_c$ 's (at 4.2 K) for 4 hrs is relatively high and reaches to  $10^5$  A/cm<sup>2</sup> in a weak field, and is about two times as large as that for 42 hrs over most of the magnetic field range. In high fields the  $J_c$ 's were about 1x10<sup>4</sup> A/cm<sup>2</sup> at 15 T, and about 1x10<sup>3</sup> A/cm<sup>2</sup> at 27 T. As the temperature decreased to 1.6 K, the increased  $J_c$  values is about twice that at 4.2 K in a high field. The obtained  $J_c$  at 1.6 K is  $\sim$ 1x10<sup>4</sup> A/cm<sup>2</sup> at 20 T. The  $J_c$  at 20 T and 1.6 K is relatively high value.

Manuscript received September 30, 1986

#### $J_c$ 's in our samples show somewhat rapid decrease as the field increases. This may be due to a difference in the upper critical field $B_{c2}$ of each grain, because the $T_c$ 's of the present PMS samples were slightly broad as shown in reference[13].







# APPLIED FIELD B (T)



#### 0018-9464/87/0300-1019\$01.00©1987 IEEE

Authorized licensed use limited to: TOHOKU UNIVERSITY. Downloaded on March 04,2010 at 02:35:58 EST from IEEE Xplore. Restrictions apply.



Fig.3 Pinning force density at 4.2 K as a function of applied field. Five curves are for PMS tapes reacted for 4-70 hrs at 1050 ℃.

APPLIED FIELD B(T)

30

The variation of pinning force density,  $F_P=J_cx B$ , with applied magnetic field is shown in Fig.2, and 3. Fig.2 shows the temperature dependence of the pinning force densities for two PMS tapes. Note that  $F_P$ 's substantially increase as the temperature decreases. In figure 3, five curves are for tapes reacted for 4-70 hrs. Note that  $F_P$  continues to increase as the reaction time decreases, and become maximum at about 5 T or thereabouts. Figure 4 shows the normalized pinning force density  $F_P(B)/F_P(15 T)$ . These values are essentially the same for all samples reacted for various periods. The only difference between the samples is the magnitude of the pinning mechanism is the same in all samples shown in Fig.4.

As a result of investigating some  $F_{\rm D}$  data in PHS tapes, it was not consistent with a major feature of Kramer equation, <sup>14</sup> i.e., that the pinning force maximum is  $b(B/B_{\rm C2})=0.2$  and moves to lower value of b as the pinning force increases. The measured  $T_{\rm C}$  value suggest that the  $B_{\rm C2}$  for these samples could be above 40 T. Consequently the  $F_{\rm P}$  maximum is seen to fall at  $b \leq 0.1$  for all samples shown in Fig.4. This may be due to inhomogeneities in these materials. In some samples, however, we observed the large b value of



Fig.4 Normalized pinning force density  $F_{p}(B)/F_{p}(15 T)$ vs applied field B for several PMS samples.

about 0.2.<sup>15</sup> Unfortunately, high quality samples with large b value can not be reproducibly prepared with the present techniques. For a more detailed comparison with flux pinning theories, homogeneous samples and data on B<sub>c2</sub> and J<sub>c</sub> as a function of temperature and field are necessary.

In all superconducting compounds, grain boundary is one of the most effective flux-pinning center to achieve high critical current density. We investigated the influence on the grain size to  $J_{\rm c}$  in our PMS tapes.

SEM observations were made on the fractured section of tapes etched for 1-10 sec in  $\sim$ 5 % HNO<sub>3</sub>/ 95 % water. Fig.7 shows SEM photographs of etched cross section of PMS reacted at 1050 °C for 4 or 70 hrs : (a), (b) reacted for 4 hr, and (c), (d) for 70 hr ; (a), (c) near center, and (b), (d) near surface in the fractured cross-section.

A finely divided structure is seen in Fig.5(a) and (b). The grain size is about 0.2 to  $1 \mu m$ . The pores (black areas) partially resulting from the chemical etching in nitric acid are surrounded by PMS compound. The material etched in nitric acid may be lead educed on cooling after the reaction at 1050 °C. On the other hand, we can see a large grain in Fig.5(c). and somewhat small grain in Fig.5(d). In a long reaction time, the grain generally grew to be nearly 1 to 10  $\mu m$  in size.



(a) near center [4 hrs] (b) near surface



(c) near center [70 hrs] (d) near surface

Fig.5 SEM photographs of etched cross section of PMS tapes : (a), (b)reacted for 4 hr, and (c), (d) reacted for 70 hr. The scale on these photographs is in micrometers.





The grain size as a function of reaction time over a range from 4 to 70 hrs is shown in Fig.6. The solid curve is for the near surface in the fractured cross-section. The dashed line is for the near center. The grain size at a given temperature(~1050°C) remarkably increased in the near center with reaction time. This observation would appear to explain the  $J_c$  - B result which the  $J_c$ 's show somewhat rapid decrease as the field increases(see Fig. 1).

The dependence of critical current density on inverse of average grain size  $G^{-1}$  is shown in Fig.7. The J<sub>c</sub>'s at 14 T vary roughly linearly with  $G^{-1}$ , i.e. it can be scaled by the average grain size. The result suggests that the grain boundary is the most effective pinning-center in PMS as well as most of other superconducting compounds. At present our smallest average grain size was about 0.5  $\mu$ m or thereabouts. If we require a significantimprovement of  $J_c$  above 10^4 A/cm² at 30 T, the further reduction of grain size (<0.1  $\mu m)$  should be achieved without the reduction in its critical temperature.





## CONCLUSION

J<sub>c</sub>'s of PbMo<sub>6</sub>S<sub>e</sub>short tapes have been measured as a function of applied magnetic field up to 30 T at 4.2 K and 1.6 K. Improved  $J_c$  values were obtained for a short heat-treatment at 1050 °C. Jc's at 20 T were  $\sim$ 5x10<sup>3</sup> A/cm<sup>2</sup> at 4.2 K, and  $\sim$ 1x10<sup>4</sup> A/cm<sup>2</sup> at 1.6 K. The Jc's at 14 T vary roughly linearly with the inverse of average grain size. Optimal reaction conditions improved Jc's in low fields. J<sub>c</sub>'s in high fields above 20 1, numerol, and containing ficient for practical application. We note that much work 's in high fields above 20 T, however, are still insufhas still to be done in order to elucidate the flux pinning mechanism in this compound.

# ACKNOELEDGEMENT

This work was financially supported by the Grant-in-Aid for Fusion Research from the Ministry of Education of Japan. The authors would like to thank K.Egawa for his assistance in experiments.

# REFERENCES

- [1] N.E. Alekseevskii, N.H. Dobrovolskii, D. Eckert and V.I. Tsevro ; J.Low Temp., Vol.29, 565 (1977)
- T. Luhman and Dew-Hughes ; J. Appl. Phys., Vol. 49, [2] 336 (1978)
- [3] S. A. Alterovitz, J. A. Woolan, L. Kammerdiner; Phyloso.
- Magazine B, Vol. 38, 619 (1978) B. Seeber, C. Rossel and O. Fisher ; "Ternary Super-
- conductors", New York, North Holland, 119 (1981) C. Rossel ; Ph. D Thesis No. 2019, University of [5] Geneva (1981)
- [6] K. Hamasaki, T. Inoue, T. Yamashita, T. Komata and
- T.Sasaki ; Appl. Phys. Helt , Vol.41, 667 (1982) [7] B. Seeber, C. Rossel, Ø. Fisher and W. Glaetzle; IEEE
- Trans. Magn., <u>MAG-19</u>, 402 (1983) C. Rossel and <del>D. Fisher</del>; J. Phy. F : Met. Phy.,
- Vol. 14, 455(1984) K. Watanabe
- [9] K. Hamasaki, T. Yamashita, T. Komata, K. Noto, K. Wata;
  Adv. Cryoge. Eng. Mate., Vol. 30, 715 (1985)
  [10] D. Cattanni, R. Baillif, B. Seeber and B. Fisher ;'85
- MT-9, 2PE-36
- [11] Y.Kubo, K.Yoshizaki, F.Fujiwara and Y.Hashimoto ; 85 ICMC at Boston, U.S. (Adv. Cryog. Eng. Hate., Vol. 32, to be published)
- [12] K. Noto, A. Hoshi, K. Watanabe and Y. Muto ; '85 ICMC at Boston, U.S. (Adv. Cryo. Eng. Mate., Vol.32, to be published)
- [13] K. Noto, K. Hamasaki, T. Komata, T. Saito, T. Yamashita and K. Watanabe; '85 ICHC at Boston, U.S. (Adv. Cryog. Eng. Hate., Vol. 32, to be published)
- [14] E.J. Kramer; J. App. Phys. Vol. 44, 1360 (1973)
  [15] K.Hamasaki, K.Hirata, T. Vamashita, T. Komata, K. Noto and K.Watanabe; IEEE Trans. Magn.<u>, MAG-21</u>, 471 (1985)