

## Growth of Bi-based whiskers from the glassy quenched platelets including SiO<sub>2</sub>, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>

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### Abstract

We grew the Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n</sub>O<sub>y</sub> (Bi-based) whiskers from the glassy quenched platelets scattering SiO<sub>2</sub>, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> powders. The lengths of the whisker grown using SiO<sub>2</sub>- and TiO<sub>2</sub>-scattered glassy quenched platelets were from 300 to 1200 μm and from several 10 to 500 μm, respectively. By using Al<sub>2</sub>O<sub>3</sub>-scattered glassy quenched platelets, by the whisker of about 8mm was obtained. Therefore, the Al<sub>2</sub>O<sub>3</sub> powders are thought to be more useful for the growth of the Bi-based whiskers than SiO<sub>2</sub> and TiO<sub>2</sub> powders.

Key words: Bi-based superconductor; Whiskers; Growth mechanism

### 1. INTRODUCTION

The Josephson junction is a basic technology for superconducting devices, and the layered structure of the high temperature copper oxide superconductor, especially Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n</sub>O<sub>y</sub> (Bi-based) provides for the construction of a stacked intrinsic Josephson junction. Bi-based whiskers are known to have high crystallinity and their size is suitable for the fabrication of superconducting devices. Recently, developments of such a device which are used into high frequency applications have been carried out by using the whiskers [1-4]. However, since these performance of devices are dependent on the quality of the whisker, it is necessary to get the high quality Bi-based whiskers.

Many growth methods of the Bi-based whiskers, for example, melt-quenched method [5-10], Al<sub>2</sub>O<sub>3</sub>-seeded glassy quenched platelets (ASGQP) method [11-13] and polycrystalline precursor pellets method [14,15], have been reported so far. However, it is not easy to obtain high quality Bi-based whiskers.

In this study, we grew the Bi-based whiskers from SiO<sub>2</sub>, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>-scattered glassy quenched

platelets, and investigated characteristics of the Bi-based whiskers.

### 2. Experimental

The starting materials with the composition of Bi:Sr:Ca:Cu=1:1:1:2 were prepared from chemical powders of Bi<sub>2</sub>O<sub>3</sub>, SrCO<sub>3</sub>, CaCO<sub>3</sub> and CuO, and were weighed, mixed and ground. They were transferred into an alumina crucible and melted at about 1100°C for 0.5h in air. Then, the melting were poured onto a polished iron plate, where SiO<sub>2</sub>, TiO<sub>2</sub>, or Al<sub>2</sub>O<sub>3</sub> powders were scattered as a seed, and quickly pressed with another iron plate. Thus, glassy quenched platelets were produced. The glassy quenched platelet were set on alumina boats, and the whiskers were grown in an oxygen gas flowing rate of 120 ml/min at the temperature from 850 to 900°C for 48 hours.

Surface morphology, composition, critical temperature ( $T_c$ ), critical current ( $I_c$ ), and crystal structure of the Bi-based whisker were estimated by scanning electron microscope (SEM), electron-probe micro-analysis (EPMA), resistance-temperature ( $R-T$ ),

current-voltage ( $I$ - $V$ ) and X-ray diffraction (XRD). The  $R$ - $T$  and  $I$ - $V$  characteristic were measured using a four probe method.

### 3. Results and discussion

Fig.1 shows the composition from the surface area without  $\text{SiO}_2$  and  $\text{TiO}_2$  powders in  $\text{SiO}_2$ - and  $\text{TiO}_2$ -scattered glassy quenched platelets. As shown in the figure, the composition of  $\text{SiO}_2$ - and  $\text{TiO}_2$ -scattered glassy quenched platelets was approximately equal to that of  $\text{Bi}:\text{Sr}:\text{Ca}:\text{Cu}=1:1:1:2$ . However, the  $\text{SiO}_2$ - and  $\text{TiO}_2$ -scattered glassy quenched platelets included Al. Al may be doped into the glassy quenched platelets from  $\text{Al}_2\text{O}_3$  crucibles.

Fig.2 shows maximum length of Bi-based whiskers as a function of growth temperature, where  $\text{TiO}_2$ - and  $\text{SiO}_2$ -scattered glassy quenched platelets were used and the growth period was 48h. As shown in the figure, the length of the Bi-based whiskers grown at the temperatures from 870 to 893°C using the  $\text{TiO}_2$ -scattered glassy quenched platelets was from several  $10\mu\text{m}$  to  $500\mu\text{m}$ . The glassy quenched platelets were melt at the temperatures more than  $895^\circ\text{C}$ , consequently any whiskers did not grow. When the  $\text{SiO}_2$ -scattered glassy quenched platelets were used, the length of the whiskers was from 300 to  $1200\mu\text{m}$ . The whisker with a maximum length of  $1200\mu\text{m}$  was obtained at  $888^\circ\text{C}$ . The length of the whiskers grown using the  $\text{SiO}_2$ -scattered glassy quenched platelets was longer than that using the  $\text{TiO}_2$ -scattered glassy quenched platelets. This indicates that the  $\text{SiO}_2$  powders are more useful for long Bi-based whiskers than the  $\text{TiO}_2$  powders.

Fig. 3 shows XRD patterns of the Bi-based whisker grown using the  $\text{SiO}_2$ -scattered glassy quenched platelets. As shown the figure, XRD peaks were observed at about  $23$ ,  $28$  and  $35^\circ$ , which were assigned to  $(008)$ ,  $(0010)$  and  $(0012)$  from a Bi-2212 phase superconductor. From the results, we found that the whiskers were Bi-2212 single-phase and  $c$ -axis-oriented. The FWHM (Full-Width-Half-Maximum) value of the

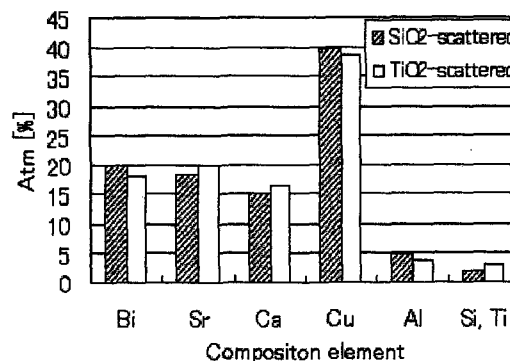


Fig.1. The composition of  $\text{SiO}_2$ - and  $\text{TiO}_2$ -scattered glassy quenched platelets.

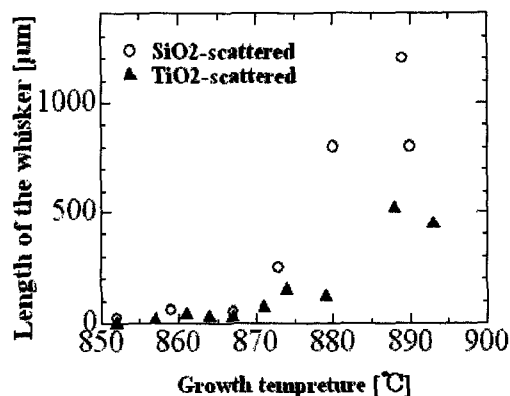


Fig.2. Maximum length of Bi-based whiskers as a function of temperature, where  $\text{TiO}_2$ - and  $\text{SiO}_2$ -scattered glassy quenched platelets were used and the growth period was 48h.

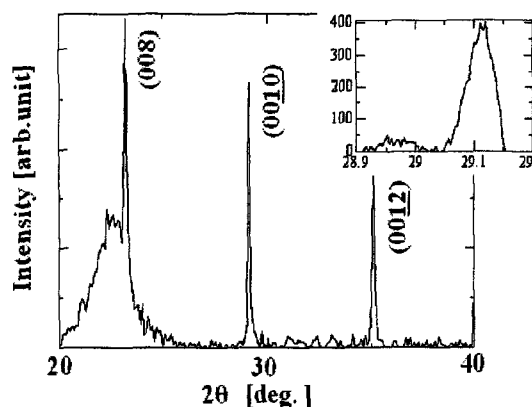


Fig.3. XRD patterns of the Bi-based whisker grown using  $\text{SiO}_2$ -scattered glassy quenched platelets

$(0010)$  peak was less than  $0.06^\circ$ . This indicates that the whiskers have high crystallinity.

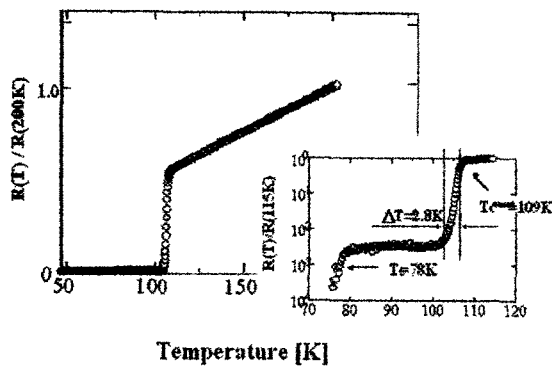


Fig.4. *R-T* characteristics in *a-b* plane of the Bi-based whisker grown using the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets.

Fig.4 shows the *R-T* characteristics of the Bi-based whisker grown using the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets. As shown in the figure, the whisker showed a metallic temperature dependence of resistance in the normal and two superconducting critical temperatures, 109K and 78K, which are assigned to Bi-2223 and Bi-2212 phases. The transition temperature width of the Bi-2223 and the Bi-2212 phases in the *R-T* characteristics of the whisker were about 3K and 4K, respectively. This indicated that the Bi-based whisker was dominantly Bi-2223 phase including a little Bi-2212 phase in the current path of *R-T* characteristics.

Although the figures were not shown, we carried out XRD of the whiskers grown using the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets. From the results, we found that the whiskers were Bi-2212 single-phase, as well as the whiskers grown using  $\text{SiO}_2$  and  $\text{TiO}_2$ -scattered glassy quenched platelets. In addition, the FWHM value of (0010) peak in the whisker was approximately equal to that in the whisker grown using the  $\text{SiO}_2$ -scattered glassy quenched platelets. This indicates that the whiskers have high crystallinity.

The growth rate of the whiskers grown using the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets were very large in comparison with those of the whiskers using the  $\text{SiO}_2$  and the  $\text{TiO}_2$ -scattered glassy quenched platelets. As a result, the whisker with a length of 8mm was obtained. The results of *I-V* characteristics indicated that the critical current of the whisker was about 150mA at 40K, which led to the critical current density ( $J_c$ ) of

about 2.2

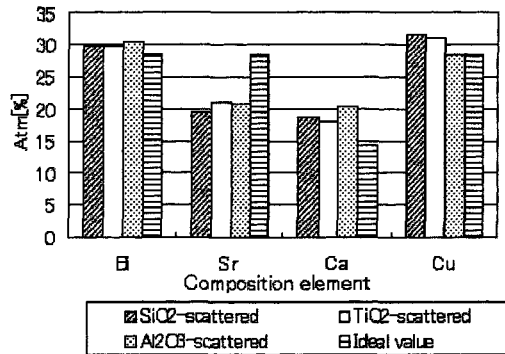


Fig.5. The compositions of the Bi-based whiskers grown using the  $\text{SiO}_2$ -,  $\text{TiO}_2$ -, and  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets, and a Bi-2212 superconductor.

$\times 10^5 \text{A/cm}^2$ .

Fig.5 shows the composition of the whiskers grown using the  $\text{SiO}_2$ -, the  $\text{TiO}_2$ - and the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets, which are estimated by EPMA results. In the figure, the ideal composition of a Bi-2212 superconductor was also shown. The composition of the whisker grown using the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelet was approximately equal to those grown using the  $\text{SiO}_2$  and  $\text{TiO}_2$ -scattered glassy quenched platelets. Comparing the composition of the whisker with an ideal composition of Bi-2212 phase, Sr content in the whisker was deficient, whereas Ca content was excess. In addition, the whiskers never included any Si, Ti and Al within an error of our EPMA apparatus.

#### 4. Conclusions

We grew the Bi-based whiskers using the  $\text{TiO}_2$ -, the  $\text{SiO}_2$ -, and the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets, and investigated the characteristic of the whiskers. From the results, we found that the Bi-based whiskers were obtained from the  $\text{TiO}_2$ -, the  $\text{SiO}_2$ - and the  $\text{Al}_2\text{O}_3$ -scattered glassy quenched platelets. The characteristics of the whiskers were the same and were independent of scattered materials,  $\text{TiO}_2$ -,  $\text{SiO}_2$ -, and  $\text{Al}_2\text{O}_3$ -, whereas the growth rates of the whiskers were dependent on kinds of glassy quenched platelets.

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