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$TbFe_2$ giant magnetostrictive alloy prepared by drop-tube system

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Abstract

In this paper, we investigated the possibility of preparation of $TbFe_2$ giant magnetostrictive alloys (GMM) under a microgravity condition by 6 m drop-tube system. $TbFe_2$ droplets diameter which varied from 1.1 to 2.1 mm were obtained. The magnetostriction of 1.1 mm droplet shows the highest value of about 1600 ppm at the applied 15 kOe. The magnetostriction of $TbFe_2$ samples increased with decreasing diameter. It was suggested that magnetostriction of the samples was influenced by cooling rate during free-fall in helium gas cooling atmosphere and oil quenching. We concluded the possibility of the continuously preparation process under microgravity of $TbFe_2$ giant magnetostrictive alloy by drop-tube system.

Keywords: Giant magnetostrictive alloy; Microgravity; Free-fall; Drop-tube; Tb–Fe alloys

1. Introduction

The giant magnetostrictive alloys (GMM) have been used for acoustic transducer and torque sensor. An improvement of the magnetostrictive properties may induce wider application fields of electric-mechanical energy conversion, such as precision-controlled mechanisms. The GMM, such as $TbFe_2$, $SmFe_2$ and Terfenol-D, have been widely studied because of high magnetostriction at room temperature [1,2]. For example, $TbFe_2$ single crystal exhibits about 2000 of magnetostriction at 25 kOe in crystallographic direction $\langle 1\,1\,1 \rangle$. However, it is very difficult to obtain homogenous morphology in Tb–Fe alloy due to the existence of double peritectic transformation and easy oxidation. These materials are currently prepared either by zone-melting technique, floating zone technique, Bridgman technique and so on [3–12].

In the past 30 years, there has been an increased interest in utilizing an orbital space environment to carry out microgravity (μG) solidification experiments. There are many approaches to achieve μG conditions in utilizing an orbital space environment, for example, international space station (ISS), free flyer (FF) and space shuttle [13–18]. Those systems were more expensive than utilizing earth-based experiments, for instance, drop-tower and drop-tube system. We reported that the unidirectional solidification of Tb–Fe alloys under μG condition by drop-tower in Japan Microgravity Center (JAMIC) might give the alloy with large magnetostrictive properties [19–21]. Then, a fontanels μG environment can be simulated on earth for short periods of time (1–6 s) by using the drop-tube system [22,23]. The drop-tube system is frequently used to study the physical mechanism of solidification and microstructure evolution, since high vacuum, containerless state and even μG condition is obtained during free-fall [24–26]. The drop-tube system has repetitive and continuous operation with cheaper cost than the other systems. Thus, a 6 m drop-tube allowing about 1 s of free-fall was used.

The purpose of this study was to investigate the possibility of producing the continuously preparation system of $TbFe_2$ alloy by drop-tube system.

2. Experimental

Alloys with $TbFe_2$ of the desired composition were prepared by arc melting in argon gas atmosphere. The arc-melted specimens, weighing approximately 0.5 g were treated with

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5 vol.% HNO_3–ethanol solution to remove a thick oxide layer of the sample surface. A 6 m drop-tube system allowing about 1 s of free-fall was used. The treated specimens were placed in a quartz crucible located in a chamber on top of the 6 m drop-tube system. A vacuum pumping system achieved a background pressure of less than 10^{-3} Pa in the drop-tube system. The drop-tube system was filled with helium gas or argon gas at the appropriate pressure of 50 Pa [27]. The specimen was heated by gold image furnace. Just after melting, the specimen in liquid, state was pushed out into the drop-tube from a quartz crucible with an orifice by filled gas pressure. During the free-fall, the specimen in liquid state was cooled by helium gas. After free-fall, samples were suspended in diffusion pump oil as shock absorber and coolant installed at bottom of the drop-tube system.

A microstructure of the samples was observed by an optical microscope and a scanning electron microscope (SEM). The composition of the samples was analyzed by energy dispersive X-ray spectroscopy (EDX). A crystal structure of the samples was determined by X-ray diffraction (XRD). The magnetostriction of the samples was measured by a strain gauge and cantilever methods [14].

3. Results

Fig. 1 shows that a relationship between the diameter for orifice of the crucible and the diameter of samples prepared by 6 m drop-tube system filled with helium gas atmosphere at the appropriate pressure of 50 Pa. The diameter of samples and deviation of diameter of samples increased with increasing the diameter of the crucible orifice. It should be pointed out that spherical droplets with diameter under 2.1 mm were obtained. However, once the diameter exceeded 2.4 mm, no spherical droplets were obtained. Numerous spherical droplets of about the diameter of 1.5 mm were obtained. In argon gas atmosphere, no spherical shape droplet was obtained in all samples.

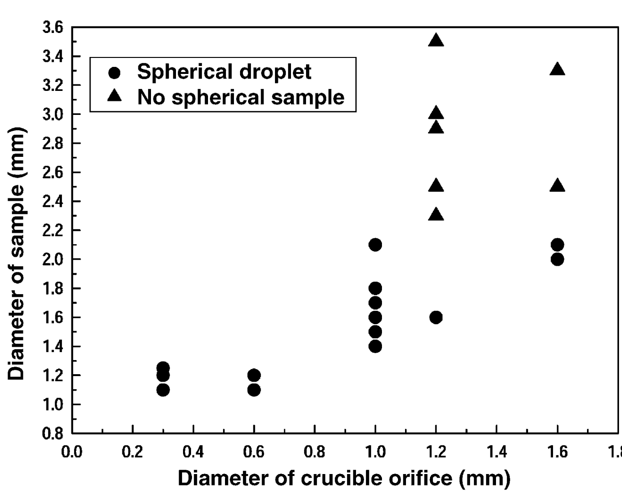

Fig. 1. Experimental distribution of diameter of samples vs. diameter of crucible orifice.

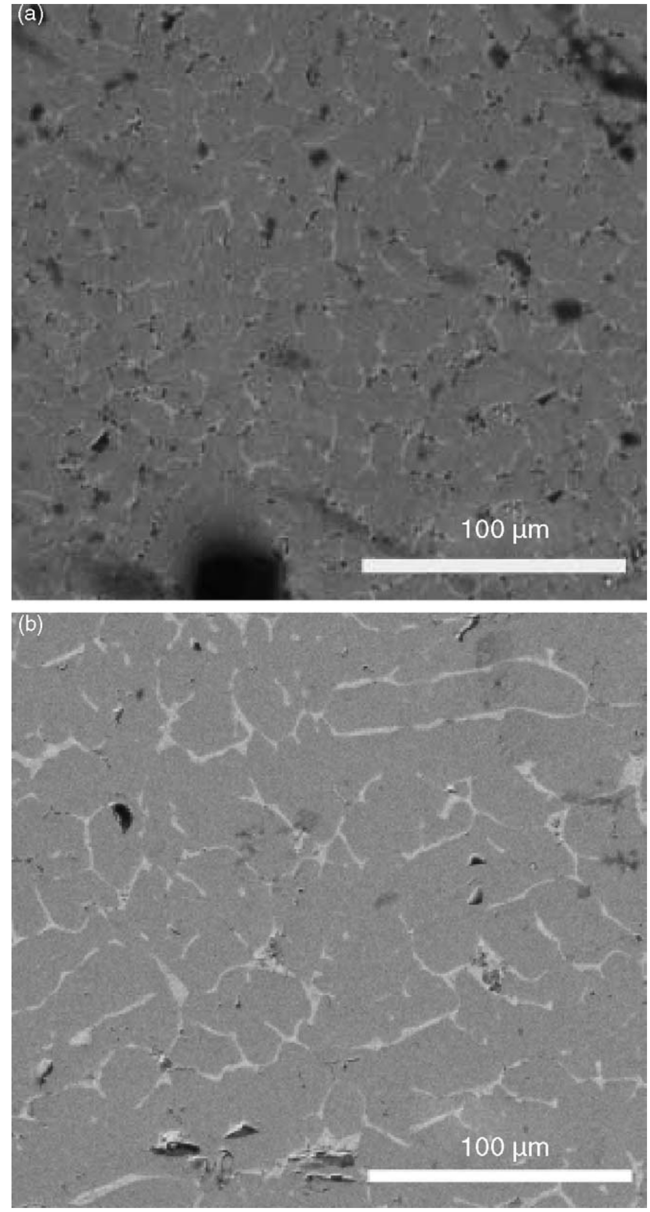

Fig. 2. Cross-sectional backscattered electron image of the (a) 1.1 mm diameter droplet and (b) 2.1 mm diameter droplet.

Fig. 2(a and b) shows the backscattered electron images of the droplet with diameter 1.1 and 2.1 mm, respectively. In the figures, gray area showed the $TbFe_2$ phase and ashy gray area was Tb phase by the result of EDX. It can be seen in both droplets that the precipitation of Tb phase occurred in grain boundaries. However, it is clear that the droplet with diameter of 2.1 mm had larger area of Tb phase than that with diameter of 1.1 mm. This result suggested segregation of Tb paramagnetism phase in grain boundaries.

Fig. 3(a) shows cross-sectional optical microphotographs of the 1.1 mm droplet and (b) is 2.1 mm droplet. In microstructure of Fig. 3(a and b), a grain size of 1.1 mm droplet was smaller than a grain size of 2.1 mm droplet.

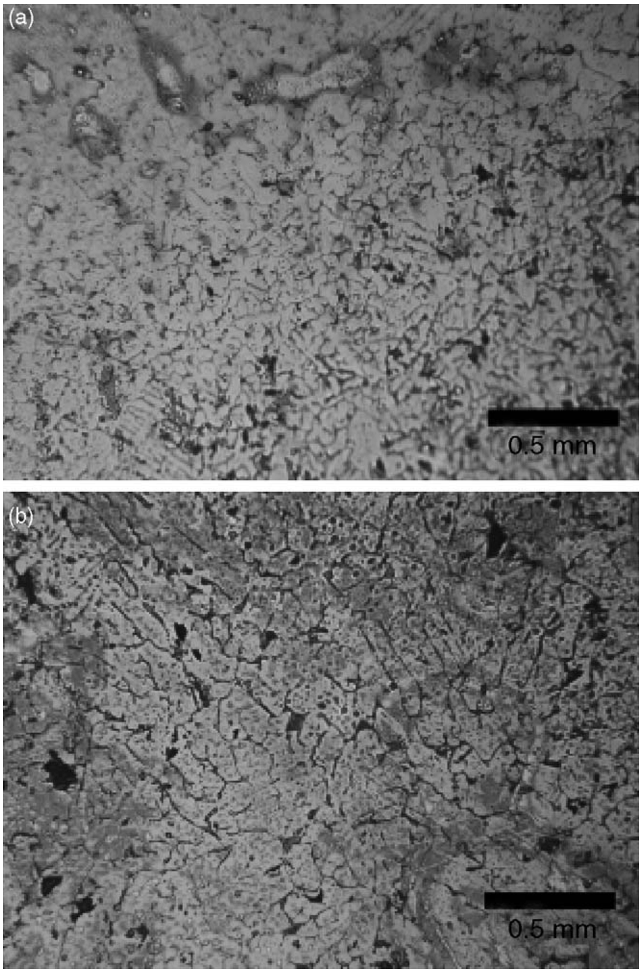

Fig. 3. Cross-sectional optical microphotographs of the (a) 1.1 mm diameter droplet and (b) 2.1 mm diameter droplet.

The field dependencies of magnetostriction along the droplet with diameter of 1.1, 1.4, 1.8 and 2.1 mm are shown in Fig. 4. It is clear that the droplet with diameter of 1.1 mm showed larger magnetostriction value than the other droplet.

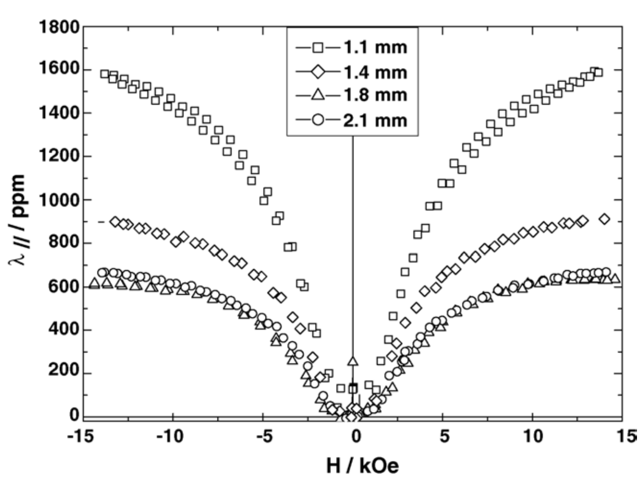

Fig. 4. Magnetostriction characteristics of droplet samples along the droplet diameter.

The magnetostriction of 1.1 mm droplet showed maximum value, about 1600 ppm at the applied 15 kOe.

4. Discussion

4.1. The backscattering electron images

The segregated Tb phase was observed in grain boundaries in Fig. 2(a and b). It suggest two possibilities of cooling process: (a) the sample have completed solidification by only helium gas before entering the diffusion pump oil and (b) the sample have solidified by helium gas cooling and oil quench. The result of backscattered electron images suggested that the both droplet are taken as possibility (b). However, in 1.1 mm droplet, the helium gas cooling in free-fall was predominant factor during solidification than oil quench. And in 2.1 mm droplet, the oil quench was predominant factor during solidification than helium gas cooling. Because, the precipitation of Tb phase occurred in grain boundaries by oil quench. Furthermore, the droplet size increases with decreasing undercooling level in drop-tube system [28,29].

4.2. Microstructure

As mentioned above, solidification of all of droplets have not finished completely by only helium gas cooling before entering the oil (possibility (b)). However, the microstructure future suggests the following two next possibilities: (c) on completion of recalescence in the sample, some part of the alloy remelted, immediately this remelting occurred, the sample quench the oil at the bottom of the drop-tube system or (d) the recalescence was not finished in the sample when it quenched the oil. Herlach et al. reported that combined with the subsequent containerless processing and high cooling rate [25], larger undercooling could be attained in droplets [30]. Thus, the microstructural features of the 1.1 mm droplet are taken as possibility (c) and the 2.1 mm diameter droplet is (d).

4.3. Magnetostriction

The magnetostriction of 1.1 mm droplet shows the highest value of about 1600 ppm at the applied 15 kOe in Fig. 4. The result was in good agreement with our present work using JAMIC drop-tower [19–21]. Clark et al. reported the magnetostriction of poly crystalline $TbFe_2$ compound showing about 1700 ppm at the applied 25 kOe of the crystallographic direction ⟨1 1 1⟩ [31]. In the larger diameter droplet, the segregation of Tb paramagnetism phases occurred with decreasing magnetostriction in Fig. 2.

5. Conclusion

In this paper, we investigated the preparation of $TbFe_2$ GMM by 6 m drop-tube system. Spherical droplets of $TbFe_2$

GMM with the diameter ranged from 1.1 to 2.1 mm have been obtained in 6 m drop-tube system filled with helium gas atmosphere. The magnetostriction of 1.1 mm droplet shows the highest value of about 1600 ppm at the applied 15 kOe. Then, the magnetostriction of $TbFe_2$ samples increase with decreasing the droplets diameters. The droplets of lower magnetostriction were observed the segregation of Tb paramagnetism phase in grain boundaries. We concluded that the 1.1 mm droplet occurred the recalecence in the period of helium gas cooling and solidification was not finished before entering oil.

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