Gas-atomized particles of giant magnetocaloric compound HoB$_2$ for magnetic hydrogen liquefiers

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Received: 19 January 2021 / Accepted: 20 March 2021 / Published online: 1 April 2021
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Abstract
The processing of promising magnetocaloric materials into spheres is one of the important issues on developing high-performance magnetic refrigeration systems. In the present study, we achieved in producing spherical particles of a giant magnetocaloric compound HoB$_2$ by a crucible-free gas atomization process, despite its high melting point of 2350 $^\circ$C. The particle size distribution ranges from 100 to 710 $\mu$m centered at 212–355 $\mu$m with the highest yield of 14–20 wt% of total melted electrode, which is suitable for magnetic refrigeration systems. The majority of the resulting particles are mostly spherical with no contamination during the processing, while unique microstructures are observed on the surface and inside. These spherical particles exhibit sharp magnetic transitions and huge magnetic entropy change of 0.34 J cm$^{-3}$ K$^{-1}$ for a magnetic field change of 5 T at 15.5 K. The high sphericality and the high magnetocaloric performance suggest that the HoB$_2$ gas-atomized particles have good potential as magnetic refrigerants for use in magnetic refrigerators for hydrogen liquefaction.

Keywords Magnetocaloric effect · Gas-atomization · Rare earth compounds · Hydrogen liquefaction

1 Introduction
Magnetic refrigeration is a promising cooling technology for replacing conventional vapor compression refrigeration. It is based on the magnetocaloric effect (MCE) of magnetic materials and has the advantages of high efficiency, energy saving, and environmentally friendliness [1–6]. Among potential applications, there is an increasing interest in hydrogen liquefaction by magnetic refrigeration [7], because liquid hydrogen is one of the efficient form for transportation and storage in the so-called hydrogen society in which hydrogen is used as an energy carrier [8]. In order to establish the magnetic refrigeration technology for hydrogen liquefaction, much effort has been devoted to the search for new magnetic materials with a large MCE [9–12] and the development of highly-efficient refrigeration systems using an active magnetic regenerators (AMR) cycle [13–16].

On the other hand, the candidate materials should be processed into spheres for practical use in the AMR system [17–19]. A large surface area of spheres is desirable for getting a good heat exchange between spheres and a heat-exchanger fluid, which is important for better performance of an AMR system. The smaller particle size, the higher heat exchange efficiency, but the concomitant increase in pressure loss leads to poor performance. Considering the trade-off between the two, it is desirable that the particle diameter is on the order of submillimeter [20].

In 2020, the ferromagnetic material HoB$_2$ has been found to exhibit a giant MCE near the liquefaction temperature of hydrogen (20.3 K) [21]; The magnetic entropy change ($\Delta S_M$) at a Curie temperature ($T_C$) of 15 K has a maximum value of 40.1 J kg$^{-1}$ K$^{-1}$ (0.35 J cm$^{-3}$ K$^{-1}$) for a magnetic field change of 5 T, which is greater than those observed in any bulk materials that have been studied for magnetic hydrogen liquefiers. Due to such the $\Delta S_M$ and $T_C$, HoB$_2$ is expected to work as suitable magnetic refrigerants for hydrogen liquefaction. However, the material properties of this boride make it difficult to process it into the desired spherical shape. For instance, HoB$_2$ has a high melting point of 2350 $^\circ$C comparable to those of ceramics. Accordingly, conventional atomization techniques [22] that melt and/or superheat a target
material in a ceramic crucible cannot be used. Besides, the plasma rotating electrode process [23, 24] is a non-contact type method to produce a high-purity spherical particles, but the brittle nature of HoB₂ hinders making an electrode and using it with high speed rotations during the processing.

In this study, we succeed in fabricating HoB₂ spherical particles for the first time by using an electrode induction melting gas atomization (EIGA) technique, in which the electrode rod of a material is melted without the use of the crucible. The resulting particles are evaluated in terms of morphology, phase confirmation, and various physical properties including magnetocaloric properties.

2 Experimental details

Figure 1 depicts the diagram of preparation process of HoB₂ spherical particles. The HoB₂ electrode rods were made by arc-melting in a water-cooled copper hearth arc furnace. Stoichiometric amounts of Ho (3N) and B (3N) elements were arc-melted under an Ar-atmosphere. Each rod was flipped and melted several times for homogenizing the sample. The resulting electrode rods were 100–300 g in weight. In the present EIGA process, one end of HoB₂ electrode rod was fixed with a chuck in an Ar-atmosphere. The rod was immersed into an induction coil and inductively melted at the other end. The molten metal then freely fell through the orifice, atomized by jetting Ar-gas, and solidified into spheres. After the atomization, the collected powder was sieved into six fractions, <100 μm, 100–212 μm, 212–355 μm, 355–500 μm, 500–710 μm, and >710 μm, through a series of JIS Z 8801 standard sieves with a FRITSCH vibratory sieve shaker ANALYSETTE 3. The sieved powder consisted of spherical particles and off-round ones, so the former were separated from the latter by rolling them on a sloped belt conveyor.

Microstructural observation was carried out using a Hitachi SU-70 scanning electron microscope (SEM) operated at 20 kV. Powder X-ray diffraction (XRD) measurements were performed at room temperature by a Rigaku MiniFlex600 diffractometer with Cu Ka radiation. The temperature dependence of magnetization (M–T curves) at various magnetic fields (μ₀H) ranging from 0.01 to 5 T were measured between 2 and 50 K in field cooling processes by a Quantum Design SQUID magnetometer. The isothermal magnetization curves (M–H curves) were collected at 2 K between 0 and 5 T. The magnetic entropy change was evaluated from a series of the M–T curves according to a Maxwell’s relation

\[
\Delta S_M(T, \mu_0 \Delta H) = \mu_0 \int_0^H \left( \frac{\partial M}{\partial T} \right)_H dH,
\]

where \(\mu_0 \Delta H\) is the magnetic field change from zero to \(\mu_0 H\).

In the magnetization measurements, the bulk sample was formed into a rectangular shape with dimensions of 2.4 × 0.5 × 0.5 mm³, and dozens of the spherical particles in contact with each other were arranged vertically long with the aspect ratio of about 2–4. The magnetic field was applied along the longitudinal directions of both the rectangular and the arranged spheres. The specific heat data at 0 T were measured by a thermal relaxation method with a Quantum Design PPMS.

3 Results and discussion

Figure 2 shows the particle size distribution for HoB₂ spherical particles. The gas atomization process was performed repeatedly, and the average values of the resulting yield are shown here. We find that the yield of the spherical particles has a significant value in the range from 100 to 710 μm, where the distribution patterns are centered around 212–355 μm with the yield of 14–20 wt% of the total melted electrode. The obtained particle size range is suitable for AMR systems. This may be due to the fact that the atomizing gas pressure in this study was set to 1.5–3.5 MPa, which is lower

![Diagram](Fig. 1 (Color Online) The diagram of preparation process of HoB₂ spherical particles)
than the values used in conventional EIGA experiments for producing fine powder [25–27], in which the particle size is typically 100 μm or less [25–29].

Figure 3a shows a SEM image of surface for HoB₂ particles with 355–500 μm diameter. The majority of the particles are mostly spherical and has no satellites as is seen in other gas-atomized materials [29–31]. By using an image analysis software Image-J (National Institute of Health, US), the roundness of these particles is evaluated to be about 0.88. This quantity, defined as 4 × (Area)/(π × (Major axis)²), equals 1 for a circular object and less than 1 for an object that departs from circularity. The obtained value of the roundness guarantees the good sphericality of HoB₂ gas-atomized particles.

As shown in Fig. 3b, however, the surface of each particle is not smooth but has a turtle shell-like structure. Moreover, a more complex inner structure with voids can be seen in Fig. 3c. It should be noted that such internal voids do not result from the gas atomization process because similar internal texture is observed in the HoB₂ electrode (Fig. 3d). These microstructures on the surface and inside can be attributed to the crystal structure of HoB₂. This diboride has a layered structure with a hexagonal B-network as shown in Fig. 3e, so the crystals are expected to grow while being oriented along the ab-plane. When the sample is solidified relatively quickly in the gas-atomization process and the arc-melting process, it is possible that the crystals are oriented inhomogeneously, and consequently, the microstructures are formed.

The powder XRD patterns shown in Fig. 4 confirm that both HoB₂ spherical particles and HoB₂ electrode have the main phase of the hexagonal HoB₂ with the space group of P6/mmm. The position and width of HoB₂ peaks are the same before and after atomizing, which suggests no change in crystallographic properties of HoB₂ phase by the atomization process. Furthermore, no differences in the peaks are found in all the atomized samples. Even though the solidification rate should depend on the particle diameter, it does not seem to affect the crystal structure of HoB₂. As an impurity phase, a tiny amount of Ho₂O₃ is found for the electrode while it disappears after atomizing. Instead, a bit trace of of HoB₄ is observed in all the atomized particles. HoB₂ is a peritectic system in which it decomposes into solid HoB₄ and a Ho-rich liquid phase at 2200 °C before completely melted at 2350 °C [32]. Thus, HoB₂ probably appeared when the HoB₂ electrode was melted in the atomization process. Nevertheless, there are no other impurities, implying that it is free from contamination during the processing.

Now let us investigate the physical properties of the HoB₂ atomized particles. Figure 5a shows the M–T curves at 0.01 T in field cooling processes. Here the data for the electrode are scaled by 0.65 for visibility. In both the electrode and all the particles with different diameters, the magnetization rapidly increases as the temperature decreases toward 15 K, which is indicative of a ferromagnetic transition. The Curie temperature T_C, defined as a peak temperature of dM/dT, is evaluated to be 15.5 K for all the samples (see the inset of Fig. 5a). Furthermore, all the M–T curves exhibit a kink anomaly around 11 K, which is associated with the spin-reorientation phenomenon [33]. The kink temperature, named by T*, does not change before and after the atomization process. As shown in Fig. 5b, the well-defined peaks in specific heat at 0 K are observed around T_C and T*, meaning that clear phase transitions take place at each temperature. These peaks in the HoB₂ particles are as sharp as those in the electrode. In addition, the M–H curves at 2 K for all the samples have a similar magnetic field dependence and quantitatively agree with each other in high magnetic fields (Fig. 5c). All these results suggest that the HoB₂ spherical particles are homogeneous and of good quality comparable to the bulk counterpart.

The disagreement in M–H curves below 1 T between the electrode and the particles should reflect the intrinsic demagnetization effect: The lower aspect ratio of the arranged particles can result in a larger demagnetization field. The difference between the M–T curves is also probably due to that of the aspect ratio. Accordingly, we conclude that there is no intrinsic dependence of magnetic properties on the particle diameter. In the gas-atomization process, the quenching effect on atomized particles is expected, and the
The cooling time of atomized particles can vary by diameter. No diameter dependence implies that the quenching effect has no impacts on the magnetic properties of HoB$_2$. This is consistent with no differences in physical properties between the electrode and the particles. Note that it is difficult at this stage to discuss the relationship of the quenching effect with the physical properties in depth, because the nature of HoB$_2$ itself has not been clarified.

Fig. 3 (Color Online) a SEM image of surface and b its enlarged image for HoB$_2$ particles with 355–500 μm diameter. The cross-sectional SEM image of c the HoB$_2$ particles and d the HoB$_2$ electrode. e The crystal structure of HoB$_2$ with the space group of $P6/3mmc$. 

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Fig. 4 (Color Online) Powder X-ray diffraction patterns of HoB₂ electrode and HoB₂ particles with various diameters from 100 to 710 μm. The marks indicate the Bragg peaks of Ho₂O₃ (•) and HoB₄ (•).

Fig. 5 (Color Online) Temperature dependence of a magnetization at 0.01 T and b specific heat at 0 T in HoB₂ electrode and HoB₂ particles. c Isothermal magnetization curves at 2 K. The inset in a depicts the temperature derivative of magnetization dM/dT at 0.01 T.
It should be also discussed whether the impurity \( \text{HoB}_4 \) affects the physical properties of the \( \text{HoB}_2 \) particles. This tetraboride is known to exhibit two antiferromagnetic-like transitions at 7.1 K and 5.7 K, at which the magnetization shows a kink or drop and the specific heat shows sharp peaks \([34, 35]\). As is evident in Fig. 5a and b, no such anomalies are found in the data for the \( \text{HoB}_2 \) particles. Thus, it is likely that \( \text{HoB}_4 \) with a small amount has no influence on the final physical properties of \( \text{HoB}_2 \). This is true for a small amount of \( \text{Ho}_2\text{O}_3 \), which has an antiferromagnetic transition temperature of 2 K \([36]\).

Finally, we will evaluate the magnetocaloric properties of the \( \text{HoB}_2 \) atomized particles. Figure 6a shows the magnetic entropy change \( \Delta S_M \) for \( \mu_0 \Delta H = 5 \, \text{T} \) in the spherical particles and the electrode. Here we take \( \text{J} \, \text{cm}^{-3} \, \text{K}^{-1} \) as the units of \( \Delta S_M \) as this unit is meaningful from an engineering point of view \([4]\). Besides, it has shown that the demagnetization effect does not seriously affect the evaluation of \( \Delta S_M \) for the large \( \mu_0 \Delta H \) \([37]\). The \( \Delta S_M \) is qualitatively and quantitatively the same between all the spherical particles and the bulk counterpart: It peaks at \( T_c \) and \( T^* \) and takes the maximum value of about 0.34 \( \text{J} \, \text{cm}^{-3} \, \text{K}^{-1} \) at \( T_c \). These results are almost consistent with those in our previous work \([21]\). Therefore, it is found that \( \text{HoB}_2 \) spherical particles prepared in this study have the same magnetocaloric properties as bulk material. Furthermore, we would like to compare here \( \text{HoB}_2 \) with other promising magnetocaloric materials that have similar magnetic transition temperatures. It can be seen from Fig. 6b that the \( \Delta S_M \) of \( \text{HoB}_2 \) is not only large in peak value, but also has the advantage of keeping a large value up to high temperatures. It is worth noting that the values of \( \Delta S_M \) in \( \text{HoB}_2 \) are comparable to those in \( \text{HoAl}_2 \) even at above 30 K. This feature of \( \text{HoB}_2 \) is attractive as magnetic refrigerants in that it is able to work over a wide temperature range.

4 Conclusion

We have succeeded in producing the spherical particles of a giant magnetocaloric compound \( \text{HoB}_2 \) by the electrode induction melting gas atomization technique. The obtained particle size range is suitable for AMR systems: it ranges from 100 to 710 \( \mu \text{m} \) centered at 212–355 \( \mu \text{m} \) with the highest yield of 14–20 wt% of total melted electrode. The resulting particles have almost perfect spherical shape and no contamination during the processing. Furthermore, they have the characteristic microstructures on the surface and inside, which may originate from the crystallographic nature of \( \text{HoB}_2 \). The physical properties of the spheres are quite similar to those of the bulk counterpart, in which the sharp magnetic transitions and the giant magnetic entropy change are observed. All the results suggest that the gas-atomized \( \text{HoB}_2 \) particles have good potential as magnetic refrigerants for use in magnetic hydrogen liquefiers.

Not limited to \( \text{HoB}_2 \), magnetocaloric materials are often high melting point materials, which may hinder processing them into particles by conventional atomization methods. Our experimental results have demonstrated that the present atomization process is a viable route to fabricate spherical particles, even for the materials with melting points above 2000 °C. This fact would encourage the production of particles for various magnetocaloric compounds, which makes a significant contribution for developing magnetic refrigeration systems.

![Fig. 6](Color Online) a Magnetic entropy change for \( \mu_0 \Delta H = 5 \, \text{T} \) in \( \text{HoB}_2 \) particles and \( \text{HoB}_2 \) electrode. b The comparison of the \( \text{HoB}_2 \) particles with 355–500 \( \mu \text{m} \) diameter (\( \triangledown \)) and other promising magnetocaloric materials: \( \text{ErAl}_2 \) (\( \bigcirc \)) \([38]\), \( \text{HoN} \) (\( \square \)) \([39]\), \( \text{EuS} \) (\( \triangle \)) \([40]\), and \( \text{HoAl}_2 \) (\( \diamond \)) \([41]\).
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Acknowledgements This work was supported by JST-Mirai Program Grant Number JPMJMI18A3, Japan.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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