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Consolidation of Pure $Sm_2Fe_{17}N_x$ Powder by Shock Compression

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Shock-compression recovery experiments were performed on the magnetically aligned pure $Sm_2Fe_{17}N_x$ powder with the Th_2Zn_{17} structure in order to prepare the full dense $Sm_2Fe_{17}N_x$ magnet with no binder and without decomposition, taking advantage of the very short duration, high pressure and shear strain of shock compression. The pure $Sm_2Fe_{17}N_x$ (x=2.6-2.7) bulk bodies, whose porosities were less than 5 %, were prepared by the shock compressions in a certain low pressure region. It was confirmed by the X-ray diffraction method and chemical analysis that the structure and composition did not change. In addition, it was found that the some pure bulk bodies consolidated by shock compression showed the Curie temperature of approximately 475° C and the higher coercivity than that of the starting material.

1. INTRODUCTION

Since the excellent intrinsic magnetic properties of $Sm_2Fe_{17}N_x$ were first reported in an academic paper by Coey and Sun in 1990 [1], so many fundamental and application investigations have been performed on this type compounds [2-6]. It has been predicted that the $Sm_2Fe_{17}N_xC_y$ type compounds have anomalous magnetic capabilities as well as or better than Nd-Fe-B system magnets. The preparations of the sintered bulk magnets using binder with low melting temperature or the bond magnets with polymer have been currently investigated. But, such materials could not exhibit the intrinsic magnetic properties. If full dense $Sm_2Fe_{17}N_x$ bulk body is prepared with no binder, a new strong magnet with high Curie temperature is expected to be realized. However, the $Sm_2Fe_{17}N_xC_y$ bulk body has not been prepared with no binder by the normal sintering, because this type of compounds decompose to α-Fe and samarium nitride above about 500-600° C.

In this study, the shock-compression recovery experiments of the magnetically aligned pure $Sm_2Fe_{17}N_x$ powder with the Th_2Zn_{17} structure were performed to prepare a full-dense $Sm_2Fe_{17}N_x$ magnet with no binder and without decomposition, taking advantage of the very short duration, high pressure, shear strain, etc. of shock compression. Shock loading was undertaken by the high-speed impact conducted using a powder gun. The recovered specimens were investigated by means of the X-ray diffraction, hardness measurement, magnetic hysteresis measurement experiments, etc.

2. EXPERIMENTAL PROCEDURE

The $Sm_2Fe_{17}N_x$ samples were prepared from the Sm_2Fe_{17} powder. The Sm_2Fe_{17} powder was obtained by induction melting in a

vacuum, homogenized at 1000° C for 12 hours, and then crushed. The $Sm_2Fe_{17}N_x$ powder was prepared by nitrogenating the Sm_2Fe_{17} powder in a N_2 gas (1 atom) at about 500° C for 1 hour, and by crushing with jet-milling pulverization to about 3.5 μm-diam irregular particles. The powder sample was confirmed to consist of the hexagonal Th_2Zn_{17} structure phase, whose lattice parameters were a_0=0.873 nm, c_0=1.264 nm. The chemical composition of the starting material was estimated to be $Sm_2Fe_{17}N_x$ (x=2.6-2.7) by the instrumental chemical analysis of nitrogen, carbon and oxygen. The magnetically aligned powder pellets were obtained by pressing at 0.3 GPa under magnetic field of 12 kOe. The porosities of the powder pellet were about 48 %.

Shock-compression recovery experiments were conducted using a powder gun [7,8]. The powder pellet was enclosed in the capsule made of brass or iron capsule (SS-41) of about 12 mm in inside diameter and about 5 mm inside height. Shock loading was undertaken by impacting the sample capsule with an aluminum alloy (2024-Al) or copper (Cu) flat flyer plate of about 3 mm in thickness, which was accelerated to the velocities up to 1.6 km/s. The impact velocities were measured by the electromagnetic method [9]. Shock pressures in the capsule and sample were estimated by the impedance-matching method from the measured impact velocity, the Hugoniots of aluminum alloy, copper, brass, steel [10] and the specimen.

The recovered specimens were investigated by means of the X-ray diffraction (XRD), instrumental chemical analysis, hardness measurement and magnetic hysteresis measurement. Powder XRD analyses were carried out by using Cu-Kα radiation with a Rigaku Goniometer. The instrumental chemical analysis of nitrogen, carbon and oxygen was carried out using the TC-436 and WR-112 of LECO Corp. The magnetic hysteresis measurements were carried out using the VSM-3 of the TOEI, after magnetization under magnetic field of 70 kOe. The bulk densities were measured by the Archimedean method. The hardness were measured by the Vickers micro-hardnesses tester, type-M of Shimadzu Co., Ltd. to evaluate the consolidation state.

3. RESULTS AND DISCUSSION

The recovered capsules were carefully peeled out on the lathe. The disk-shaped bulk samples were obtained from the shock compressions of over 1.1 km/s in impact

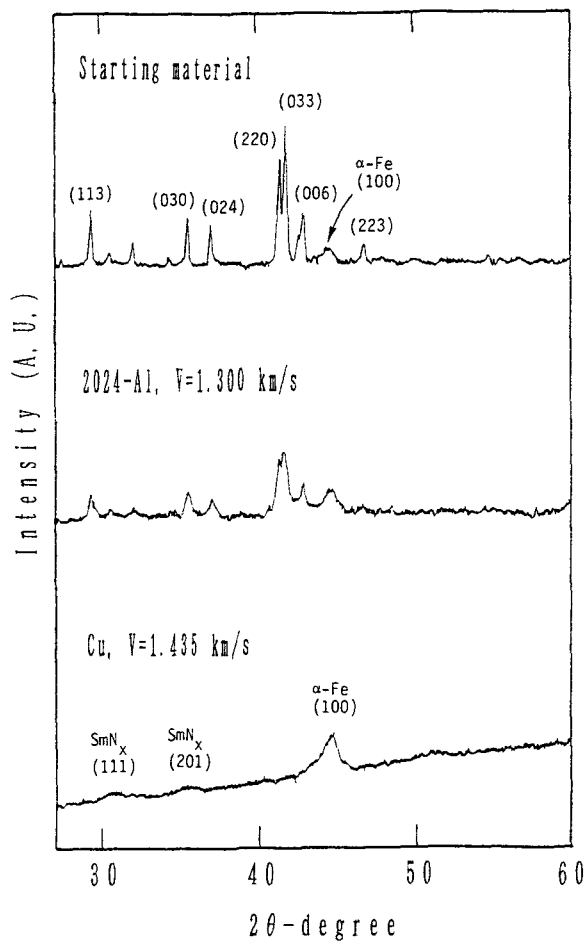

Fig. 1. Powder XRD patterns of the starting material and the shock-loaded specimens with the impact velocities of 1.300 km/s (2024-Al impactor) and 1.435 km/s (Cu impactor) by using Cu-Kα radiation.

velocity (2024-Al impactor). The bulk densities and the Vickers micro-hardness values of the recovered specimens from the shock compression of over 1.1 km/s in impact velocity (2024-Al impactor) were 7.44-7.56 g/cm^3 and within 700-1200 kg/mm^3, respectively, while the recovered specimen from the shock compression of 0.878 km/s in impact velocity did not consolidate enough. The porosities were calculated to be less than 5 %. Considering the visible shape, bulk density and hardness, the minimum necessary impact velocity (2024-Al impactor) for the consolidation of the powder pellet (48 % in porosity) was estimated to be about 1.0 km/s.

Figure 1 shows the powder XRD patterns of the starting material and the recovered specimens which were shock-loaded with the impact velocities of 1.300km/s (2024-Al impactor and brass capsule) and 1.435 km/s (Cu impactor and SS-41 capsule). The driving pressures in the capsule were calculated to be 15.8 and 30.4 GPa, respectively. The starting material contained a small amount of α-Fe, as shown. For the specimen of 1.300 km/s in impact velocity (2024-Al impactor), the XRD pattern was not much different from that of the starting material. The lattice parameters of the recovered specimen were determined to be a_0=0.873 and c_0=1.264 nm, which were not much differnt from those of the starting material. In addition, it was confirmed by chemical analysis that the contents of nitrogen, carbon and oxygen did not change much. But, for the specimen of 1.435 in impact velocity (Cu impactor), the diffraction peaks of $Sm_2Fe_{17}N_x$ disappeared, but, the large peak of α-Fe (100) and small peaks of SmN_x could be seen. The decomposition to α-Fe and SmN_x may be caused by the high temperature after shock compression. The average temperature after shock compression of 1.435 km/s (Cu impactor) in impact velocity was estimated to be higher than 550° C, which was as high as or higher than the decomposition temperature of this material. The maximum impact velocity of 2024-Al impactor for the consolidation of the powder pellet (48 % in porosity) without decomposition was estimated to be about 1.5 km/s.

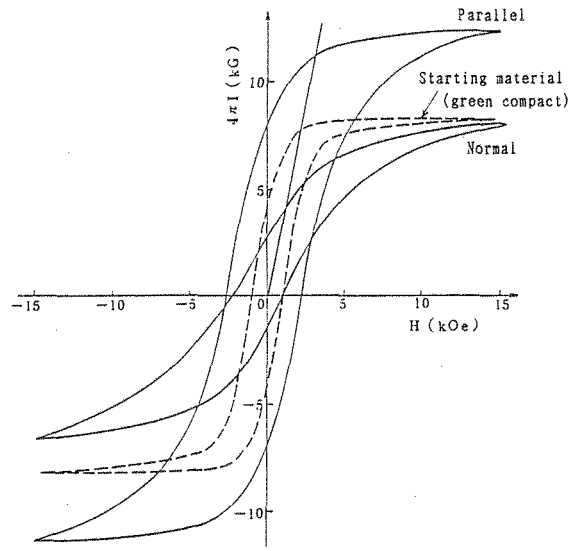

Fig. 2. Magnetic hysteresis of the starting material and the shock-loaded specimen with the impact velocity of 1.300 km/s (2024-Al impactor).

Figure 2 shows the magnetic hysteresis loops of the starting material and the shock-loaded specimen with the impact velocity of 1.300 km/s (2024-Al impactor). The coercivity of the shock-loaded one was 2.6 kOe (parallel), which was greater than the starting material of 1.1 kOe. The Curie temperature was measured to be 475° C by the thermomagnetic analysis. The reason of the improvement in magnetic property was now unknown. Under shock compression, it is assumed that so many cracks, dislocations, stacking faults, etc., appear or grow in powder particle or consolidated bulk. These structure changes may be related to the present result. If the $Sm_2Fe_{17}N_xC_y$ powder with the higher magnetic property is used as a starting material, a new strong magnet is expected to be realized. The detailed and further results will be reported elsewhere.

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