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Study of coercivity origin in nano-structured Co-Zr alloys

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Introduction

Since the discovery of the Nd-Fe-B-type material over two decades ago, a great demand for a new and superior hard magnetic phase has been increased. Through the intensive research works many promising candidate materials for an application of permanent magnet have been developed, and these include $Sm_2Fe_{17}N_x$, $SmFe_7N_X$, $Sm_3Fe_{29}N_X$, $Nd_2Fe_{14}B/Fe(Fe_3B)$ nanocomposite materials. More recently, the Co-Zr phase has also been investigated as a candidate for an application of permanent magnetic material. In the Co-Zr alloy system, there are several equilibrium phases, those phases, however, are known to be a non-hard magnetic phase. It has been known, however, that in the Co-Zr alloys processed in non-equilibrium way such as melt-spinning, some non-equilibrium metastable phases can be formed in the alloy system, and some of the metastable phases have been known to have a hard magnetic properties suitable for the application of permanent magnet [1,2]. Although some intensive research works have been made, the hard magnetic properties of the Co-Zr phase is not yet quite promising for the application of permanent magnet. Further systematic studies are yet needed to understand the magnetic properties of the hard phase and to exploit this phase as a promising permanent magnetic material. Even the coercivity origin of the non-equilibrium processed Co-Zr alloy is not fully understood, and there is still a controversy over the coercivity origin of the material. Meanwhile, a mechanical alloying technique has been considered to be an useful way of preparation of non-equilibrium material together with the melt-spinning technique. One of the advantages in the mechanical alloying technique is that a compositional and structural homogeneity through out the whole material can be obtained. In the present study, the origin of coercivity of nano-strucutured Co-Zr alloy prepared by using a mechanical alloying technique was investigated.

Experimentals

$Co_{100-X}Zr_X$ alloy (x = 10 – 40) alloys were prepared by a mechanical alloying technique using the high purity component elements. The elemental powders were put into a milling pot together with hardened steel balls. The mass ratio of steel balls and the elemental materials was 20:1. The milling pot was evacuated and then filled with a high purity argon gas. The charged material was milled for various time periods in a shaker mill. The milled powder was retrieved in a glove box filled with high purity argon gas. The retrieved milled powder was annealed in a vacuum at the temperature range of 500 ℃ – 800 ℃. The crystallization of the milled powder was studied using a differential thermal analyzer (DTA). Magnetic characterization of the materials at various conditions was carried out using a vibrating sample magnetometer (VSM) with maximum magnetic field of 15 kOe. Prior to the VSM measurement, the specimen was pre-magnetized using a pulsing field of 4.5 T. Self demagnetizing field was not corrected in this measurement. The thermomagnetic analyzing technique (TMA) was also used to examine the thermomagnetic behavior of the milled and annealed powder. The phase study of the materials at various conditions was performed using an X-ray diffractometer (XRD) (Cu *Kα* radiation) together with the TMA. Microstructure of the samples was observed using a high resolution SEM and TEM.

Results and discussion

Fig. 1 shows the XRD patterns of the mechanically alloyed $Co_{82}Zr_{18}$ alloy at various conditions. As can be seen, the material is in an amorphous state after the milling, and the thermal annealing at elevated temperatures led to a crystallization of the amorphous material. The phase constitution of the annealed material appears to be independent on the annealing temperature, and it consists of three phases of $Co_{23}Zr_6$, Co_5Zr, and fcc Co. However, the relative amount of each phase in the annealed material seems to be influenced significantly by the annealing temperature. As can be seen in Fig. 1, in the material annealed at higher temperature (1000 ℃) most of the material composed of $Co_{23}Zr_6$ phase and small amount of Co_5Zr and fcc Co coexisted. In the material annealed at lower temperature (550 ℃), however, the relative amount of Co_5Zr and fcc Co was increased significantly. The magnetic properties of the material annealed at different temperatures were measured and the obtained demagnetization curves were shown in Fig. 2. The material annealed at higher temperature (800 ℃), which may consist mainly of $Co_{23}Zr_6$ phase, shows a soft magnetic feature with little coercivity. Meanwhile, the material annealed at lower temperature (550 ℃), which contains the three phases of fcc Co, $Co_{23}Zr_6$, and Co_5Zr, shows a hard magnetic feature with an appreciable coercivity. These results indicate that among the phases formed in the annealed material the Co_5Zr phase is a hard magnetic phase, and there appears to be no doubt that the origin of coercivity in the mechanically alloyed Co-Zr alloy is the Co_5Zr phase. In this article, the effects of annealing temperature and alloy composition on the phase constitution of the mechanically alloyed Co-Zr material is to be discussed. TMA study was carried out for the mechanically alloyed Co-Zr material at various conditions, and the results were correlated to the magnetic properties, in particular to the coercivity origin of the material.

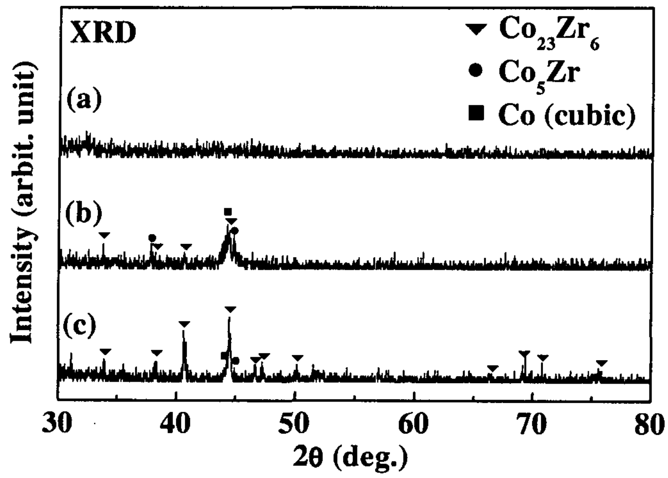

Fig. 1. XRD patterns of the mechanically alloyed $Co_{82}Zr_{18}$ alloys at various conditions. (a) as-milled (b) annealed at 550 ℃ for 20 min. (c) annealed at 1000 ℃ for 20 min.

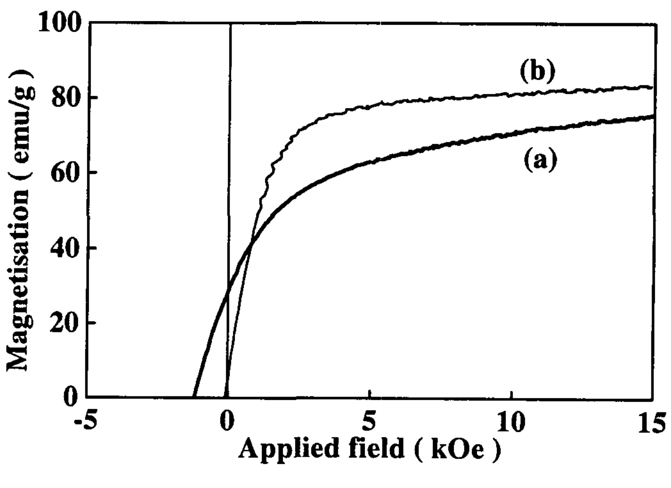

Fig. 2. Demagnetisation curves of the mechanically alloyed $Co_{82}Zr_{18}$ material annealed at (a) 550 ℃ and (b) 800 ℃ for 20 min.

Reference

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[2] Tetsuji Saito, IEEE Trans. Magn., vol. 39, no. 5 Sep. 2003