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# Preparation of fine-grained bulk materials in the Fe–Co system by shock compression

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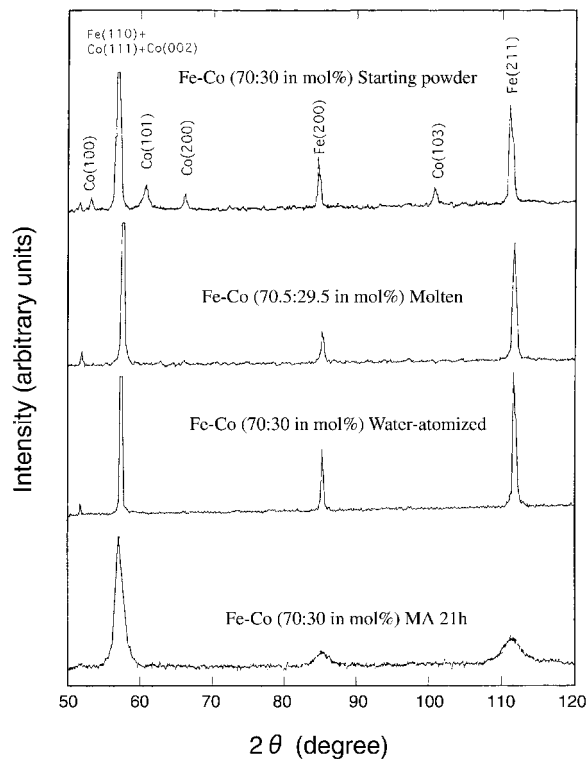
## Abstract

Fine-grained bulk alloys with no crack in the 70:30 mol% Fe–Co system were prepared by means of shock compression of water-atomized powder and mechanical alloying (MA) treated ones. The grain size of the water-atomized bulk body was smaller ( $<50 \mu\text{m}$ ) than that of the molten bulk body (about  $100 \mu\text{m}$ ). The grain size decreased greatly with the MA treatment time, and ones for 21 h were estimated to be about 15 nm from the x-ray diffraction patterns. The coercivity value of the water-atomized bulk body was much larger than that of the molten bulk body. The coercivity value of the MA-treated bulk body increased with the MA treatment time, and then decreased, despite the very small grain size, probably due to the effect of ferromagnetic exchange interaction.

## 1. Introduction

Iron (Fe)–cobalt (Co) system alloys show high magnetization, and are widely used as soft magnetic materials. The fine-grained Fe–Co alloy was expected to show large coercive force and small magnetic inductive capacity compared to those of the conventional alloy obtained by an ordinary melting method. Such materials can be used as the target materials for PVD, sputtering, etc. Mechanical alloying (MA) has recently been used for preparation of nonequilibrium powders of metastable solid solution phase, amorphous phases, nanocrystals, etc. MA experiments had been reported on Fe–Co systems, and the fine-grained alloys had been prepared [1–3]. However, it is difficult to consolidate the metastable powders to a bulk body maintaining the original state. On the other hand, the shock compression is quite different from static compression in terms of the short duration, shear stress, etc, and it can be used as an effective consolidation method for metastable powders without recrystallization or decomposition [4–7].

The purpose of this study is to prepare fine-grained bulk alloys in the Fe–Co system by means of shock compression of the water-atomized powder and MA-treated ones. The molar fraction ratio of the Fe–Co system samples used in this study was 70:30 (molar), where the magnetization value is largest in the Fe–Co system.



**Figure 1.** XRD patterns of the starting powder, the molten bulk body, the water-atomized one and the MA-treated one (21 h).

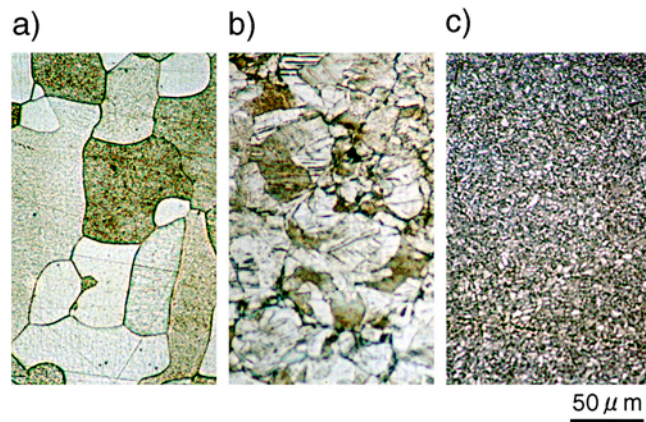
## 2. Experimental procedure

Fe and Cu starting powders, which were provided by Rare Metallic Co., Ltd, consisted of irregular particles of 4–7  $\mu\text{m}$  and 325 mesh ( $<44 \mu\text{m}$ ) in diameter and the purities of Fe and Co, as listed in the catalogue, were greater than 99.5 and 99.9 wt%, respectively. The MA treatment was performed on powder mixture of Fe and Co by using a planetary microball mill (p-7 of Fritsch Co., Ltd) in an argon atmosphere. Fine-grained alloy powder prepared by the water-atomizing method was also used. A shock-compression recovery experiment was carried out, conducted using a propellant gun. The magnetic properties were measured by using an automatic DC fluxmeter on a ring specimen.

## 3. Results and discussion

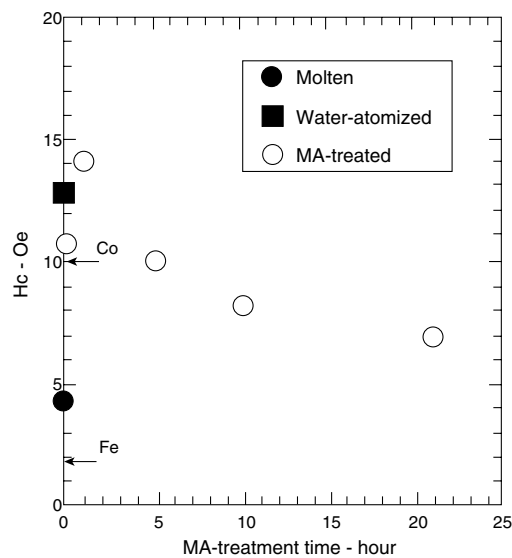
Figure 1 shows the x-ray diffraction (XRD) patterns of the starting powder, the bulk body obtained by an ordinary melting method, and the shock-consolidated bulk bodies from the water-atomized powder and one MA treated for 21 h. The XRD patterns showed that the molten bulk body, water-atomized one and MA-treated one (21 h) all consisted of BCC structure. For the MA-treated ones, the longer the MA treatment time was, the weaker and broader the peaks of the BCC structure became.

Figure 2 shows optical microphotographs of the etched surfaces of the molten bulk body (a), water-atomized one (b) and MA-treated one (21 h) (c). No visible crack can be



**Figure 2.** Optical microphotographs of the etched surfaces of the molten bulk body (a), water-atomized one (b) and MA-treated one (21 h) (c).

(This figure is in colour only in the electronic version)



**Figure 3.** Coercivity values versus MA treatment time of the MA-treated bulk bodies together with those of the molten and water-atomized ones.

seen in these shock-consolidated bulk bodies. The density of the water-atomized bulk body was comparable to that of the molten bulk alloy, and the ones of the MA-treated bulk bodies were slightly smaller. The hardnesses of the water-atomized bulk body and MA-treated ones were 2.8–3.4 and 6.2–9.7 GPa, respectively, which were larger than that of the molten bulk body (1.9–2.1 GPa). The grain size of the water-atomized bulk body was smaller ( $<50 \mu\text{m}$ ) than that of the molten bulk body (about  $100 \mu\text{m}$ ). However, the grain size of the bulk body MA-treated for 21 h was estimated to be about 15 nm from the XRD patterns.

Figure 3 shows the coercivity values versus the MA treatment time of the MA-treated bulk bodies together with those of the molten bulk body and the water-atomized one. The coercivity

value of the water-atomized bulk body was measured as 12.8 Oe, which was much larger than that of the molten bulk body (4.3 Oe). The coercivity value of the MA-treated bulk body increased with the MA treatment time, and exceeded that of the water-atomized bulk body, then decreased again. The coercivity value for the 21 h MA-treated specimen was 6.9 Oe, despite very small grain size. This may be caused by the effect of ferromagnetic exchange interaction, while the magnetic property for large grained magnets might be influenced by magnetocrystalline anisotropy [1].

It is expected that the Fe–Co bulk alloys with the larger coercivity could be prepared by controlling the grain size, i.e. the MA treatment time, etc.

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