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Local structure and magnetic properties of $\mathbf{Fe}_{60}\mathbf{Co}_{40}$ mechanical alloy

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Abstract. Fe₆₀Co₄₀ alloys were fabricated by the mechanical alloying process with the periods of 1, 2, 4, 6, 12 and 24 hours, respectively. The structural variations during the formation of the alloy were examined by the X-ray diffraction and the extended X-ray absorption fine structure. The activation for structural transform begins in 6 hour milling. Substantial structural changes were observed in 12 hour milling. The alloying process was almost completed in 24 hours. EXAFS analysis showed that BCC shells appeared in the alloy milled for 6hours. The magnetization increased as milling time increased.

1. Introduction

Fe-Co alloys have been extensively studied for the application of magnetic thin film devices and nanowire recording systems [1, 2, 3]. Nano structured Fe-Co alloys were made by Mechanical alloying (MA) and Mechanochemical alloying (MCA) method to obtain the enhanced soft magnetic materials [4].

The mechanical alloying method is useful to produce a metastable phase and nonequilibrium solid solution which are hardly obtained by the conventional method. The physical properties of the metastable alloy produced by mechanical alloy has versatile properties compared to the alloys made by the conventional method. Atoms in solid have some orders in the atomic arrangement. The magnetic properties can be related to the short or long range ordering of magnetic atoms. The atomic arrangement has been analyzed by the X-ray diffraction or the extended X-ray absorption fine structure (EXAFS) study [5, 6, 7].

In this work, $Fe_{60}Co_{40}$ alloys were produced by the mechanical alloying techniques. Magnetic properties were measured by the vibrating sample magnetometer(VSM). The structural evolution was examined by the X-ray diffraction(XRD) and the extended X-ray absorption fine structure (EXAFS) analysis. The magnetic properties were discussed in connection with the local structure of the alloys.

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Figure 1. Variation of saturation magnetization in $Fe_{60}Co_{40}$ milled for 1, 2, 4, 6, 12 and 24 hours.

2. Experiments

Fe₆₀Co₄₀ metastable alloys were made by using the SPEX 8000 Mixer and Mill with 5:1 ball to mixed powder weigh ratio. Mechanical alloying was performed under an Ar atmosphere to prevent oxidation. Each sample was made in 1, 2, 4, 6, 12 and 24 hour milling time, respectively. Variations of saturation magnetization of the each alloy powders was measured by the vibrating sample magnetometer (VSM) at room temperature in a maximum field of 10 kOe. Structural evolution due to the mechanical alloying was studied by XRD and EXAFS. XRD data were obtained by X-ray diffractometer with a monochromatic Cu K α radiation. EXAFS experiments were carried out at the beam line, 3C1 EXAFS of the Pohang light source (PLS) in the Pohang Acceleratory Laboratory (PAL) in Korea. PLS was operated with an electron energy of 2.5 GeV and the maximum current of 200 mA. The EXAFS spectra were obtained near Co K edge(7709 eV) in the transmission mode at room temperature. The ion chambers placed before and after sample for detection of X-ray intensity were filled with pure nitrogen gas. EXAFS data were analyzed by the conventional and the regularization method.

3. Results and Discussion

Figure 1 shows the magnetization of $Fe_{60}Co_{40}$ alloy produced by mechanical alloying technique with 1, 2, 4, 6, 12 and 24 hours, respectively. The magnetization decreased between 1 and 2 hour milling times. However, the magnetization increased significantly in 12 and 24 hour milling times. This indicates that there are substantial changes in 12 and 24 hour milling. During mechanical alloying, the Co atoms were interacted with Fe atoms to increase the magnetization.

Figure 2 shows the X-ray diffraction patterns for the structural variation due to the internal stress caused by the mechanical alloying process. The peaks intensity of both Fe and Co reduced gradually up to processing time of 4 hours due to decrease of particle size of Fe and Co cluster. Co (101) peak disappeared after 4 hours milling. This indicates that Co cluster disappear and Co atoms begin to diffuse into iron cluster. Fe peaks remained until 24 hour milling times. This indicates that Fe cluster remained until 24 hour milling. The structural change agrees with the variation of magnetic properties. However, the XRD results were not sufficient to show the local structural variation between 6 and 12 hour milling samples.

The extended X-ray absorption fine structure was used to examine the atomic

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Figure 2. X-ray diffraction patterns of $Fe_{60}Co_{40}$ mixed powder processed for 1, 2, 4, 6, 12 and 24 hours, respectively.



Figure 3. Fourier transform of the EXAFS spectra for $Fe_{60}Co_{40}$ measured at Co K-edge with processing time.

arrangement in local structure of the samples. In EXAFS analysis, the spectral variations give information on the local structure at atomic scale.

Figure 3 shows the Fourier transform of EXAFS spectra measured at Co K-edge with processing times. As shown in Fig. 3, the intensity of first shell peak near 2.0Å in the Fourier transformed spectrum decreased gradually as the processing time increased. This suggests that the number of Co-Co direct bond decreased due to the diffusion of Co atoms into the Fe cluster. The higher shell peaks exhibit the long range ordering in Co-Fe alloy in the Fourier transform of EXAFS spectra. The higher shell peaks at 4.1Å also reduced significantly between 2 and 6 hours. Also, the peak position moved from Co HCP shell peak near 4.1Å to Fe BCC shell peak near 4.5Å. This shows that Co atoms diffused into Fe shells in this period. In 12 and 24 hour milling, the peak at 4.5Å increased. This indicates that the ordering Co atoms in Fe shells increased in this milling time.

The first peak of the Fourier transformed spectra has the local structural information such as the coordination number, the bonding distance and vibrational information of neighboring atoms. The first peaks were filtered and transformed again by the inverse transformation.

The first peaks in the Fourier transform shown in Fig. 3 were filtered, and



Figure 4. Pair distribution functions of Co-M (M=Co, Fe) pairs in $Fe_{60}Co_{40}$ as a function of the processing times.

analyzed by the regurization method [7], and the pair distribution functions for Co-M (M:Co or Fe) pairs were obtained from the filtered spectrum by solving the inverse problem. The filtering window in the inverse transform was about r=(1.51-2.85)Å. It was assumed that Co or Fe neighboring atoms has same atomic scattering factor.

Figure 4 shows the pair distribution functions obtained from the filtered EXAFS spectra for samples milled for 1, 2, 4, 6, 12, and 24 hours, respectively. The pair distribution functions were obtained by the regularization techniques.

The pair distribution functions of the samples milled for 1, 2 and 4 hours have single shell near R=2.50Å showing the HCP crystal structure. This means that the HCP shell remained until 4 hours in MA process. The magnitude of the first shell decreased slowly between one and two milling time. However, the magnitude of the first shell was reduced significantly. This indicates that there is a structural transform between 4 and 6 hour milling periods. The second shell near R=2.86Å which is the second nearest neighbor in BCC structure appeared from 6 hour milling time and their magnitude increased as milling time increased. This means that Co atoms substituted Fe atoms keeping the BCC structure and the ordering increased as milling time increased. The pair distribution functions confirm that the BCC like shells were formed in the Fe60Co40 mechanical alloy.

The fraction of Co-BCC can be estimated from the XRD and EXAFS results shown in Figs. 2-4. Figure 5 shows the fraction of Co-BCC shells in the Co-HCP shells. The Co-BCC shells increased significantly after 4 hours milling. We can see that the shell transformation was occurred significantly between four and twelve hours milling.

EXAFS analysis in Fig. 3 and Fig. 4 shows that the structural transform occurred between 4 and 6 hours. Comparing these structural variation to the magnetization shown in Fig. 1, we can confirm the Co atoms substituted Fe atoms forming BCC Fe-Co alloy and Fe-Co magnetic interaction increased the magnetic moment. EXAFS analysis showed the self-consistent results in the local structural analysis. As shown in Fig. 3. The HCP higher shell near 4.1 Åtransformed to BCC higher shell near 4.1Å. This is consistent with the structural transform in Fig. 4.

The formation of alloy can be explicitly shown in the EXAFS spectra between 6 and 12 hours processing times. The significant change of the pair distribution function confirms that new chemical bonding between the central Co atom and Fe atoms increased during the MA process. The regularization technique also estimated successfully the pair distribution functions as a function of the processing time.

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Figure 5. The fraction of Co-BCC shells as a function of the processing times.

4. Conclusion

In this work, the magnetic properties and the structure of $Fe_{60}Co_{40}$ metastable alloys produced by the mechanical alloying were examined by VSM, XRD and EXAFS analysis. From this work, we found that Co atoms substitute Fe atoms forming the BCC shells as milling time increases. Also, we noticed that the alloying was activated in 6 hour milling time and completed in 24 hours. The magnetization increased with the increase of Fe-Co bond in the local structure. The pair distribution functions by the EXAFS regularization method provided clear information on the structural variation.

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