X-Ray Absorption Fine Structure Study for $Fe_{60}Ni_{40}$ Alloy

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Abstract. $Fe_{60}Ni_{40}$ alloys were fabricated by the mechanical alloying process with process periods of 1, 2, 4, 6, 12 and 24 hours, respectively. The formation of alloy and the structural evolution of the alloy were examined by X-ray diffraction and extended X-ray absorption fine structure methods. With increase of alloying time the BCC phase of iron was changed significantly during the mechanical alloying process. The alloying was activated in about 6 hours and completed in about 24 hours.

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INTRODUCTION

Fe-Ni mechanical alloys (MA) have attracted a growing attention to the field of magnetic application because of their soft magnetic [1, 2, 3] and Invar properties [4, 5]. Also, various Fe-Ni based nanocrystalline alloys have been studied to enhance the soft magnetic properties [6]. It was shown that the FCC structure remains and mostly it was a single phase in the mechanical alloyed $Fe_{100-x}Ni_x$ for 30-50 atomic percent of nickel [7]. Recently, the first order martensite-austenite transformation was found in Fe-rich $Fe_{100-x}Ni_x$ alloy [8]. Even though there are many studies of various phenomena of $Fe_{60}Ni_{40}$, the structural analysis is still limited because of the structural disorder due to the mixed phases. In this work, metastable $Fe_{60}Ni_{40}$ alloys were fabricated by the mechanical alloying process with the process periods of 1, 2, 4, 6, 12 and 24 hours, respectively. We performed extended X-ray absorption fine structure (EXAFS) analysis to examine the detailed structural variation of mechanically alloyed $Fe_{60}Ni_{40}$. Also, we performed XRD analysis to examine the structural variation of $Fe_{60}Ni_{40}$.

EXPERIMENTS AND EXAFS DATA ANALYSIS

$Fe_{60}Ni_{40}$ metastable alloys were prepared by the mechanical alloy process using a SPEX 8000 mixer and mill with a stainless steel ball. The starting material was a mixture of pure Fe and Ni powder with a content of 60 atomic percent of iron and 40 atomic percent of nickel. MA was performed under Ar atmosphere to prevent oxidation during the alloying process. The variations in structure were examined by XRD and EXAFS. XRD data were obtained with monochromatic Cu Kα 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. The PLS was operated with an electron energy of 2.5 GeV and a maximum current of 200 mA. The EXAFS spectra were obtained near the Fe K edge (7112 eV) in transmission mode at room temperature. The ion chambers were filled with pure nitrogen gas.

EXAFS data were analyzed by a combination technique of conventional [9, 10] and regularization methods [11]. In data analysis, the normalized EXAFS spectra were extracted and transformed with the software ATHENA of a conventional EXAFS analysis program. The first shell EXAFS spectra were filtered by the inverse Fourier transform in the conventional method. The pair distribution functions (PDF) were obtained from filtered spectra processed by the regularization method. The kernels consists of the scattering parameters of two different atoms including the two different elements, respectively.

RESULTS AND DISCUSSION

In the mechanical alloying process, the mixed powder is undergoing a process of fracture and welding repeatedly. Figure 1 shows the XRD results for the structural evolution due to the MA process. The diffraction peaks decreased slowly in 6 hours while they decreased rapidly after 6 hours. This indicates that the grain size was reduced continuously during 6 hours processing time. The Fe BCC peaks and Ni FCC peaks decreased significantly in 6 hours and the Fe BCC peaks disappeared completely in 24 hours while Ni FCC peaks still remained. The shift of the main peak to low angle indicates that the lattice parameter increased. However, the XRD results were not sufficient to determine the formation of Fe-Ni bonds because the contribution of Ni atoms was hardly separated by XRD analysis.

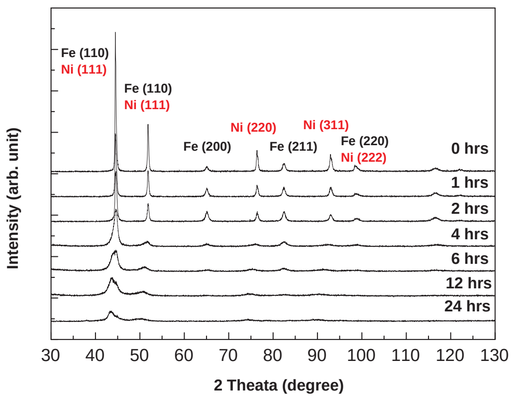

FIGURE 1. X-ray diffraction patterns for $Fe_{60}Ni_{40}$ produced by mechanical alloying.

The XANES and EXAFS can give a direct information about the variation of local structure. We used XANES to examine the variation of core electron configuration and EXAFS to examine the local structure around the Fe ions in the $Fe_{60}Ni_{40}$ alloys. As shown in Fig. 2, the normalized near edge spectra for the processed samples were similar to each other but above the edge the spectra gradually changed. This suggests that the electronic configuration for the Fe central atoms was unchanged but the surrounding around the Fe atoms was changed during the MA processing.

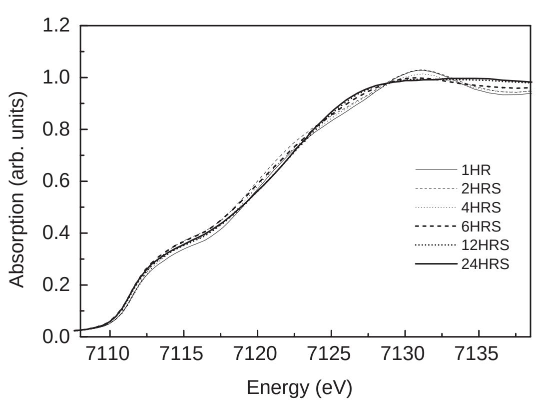

FIGURE 2. The XANES spectra of $Fe_{60}Ni_{40}$. Each line represents the processing time.

The systematic variations of the amplitude and the phase in the EXAFS spectra confirmed that alloying at atomic scale occurred during the MA process. The reduction of the amplitude is related to the disorder of local structure and the variation of the phase is related to the change of chemical order. Figure 3 shows the EXAFS spectra of $Fe_{60}Ni_{40}$ processed for 1, 2, 4, 6, 12 and 24 hours, respectively. The EXAFS spectra were obtained from the absorption spectra by removing the background with AUTOBK. The decrease of the amplitude before 6 hours indicates that the fracture was dominant. Between 6 and 24 hours, the phase was shifted significantly. This indicates that the Fe atoms were diffused into the Ni FCC complex and new materials were produced during this process. The amount of the new alloy increased as the processing time increased.

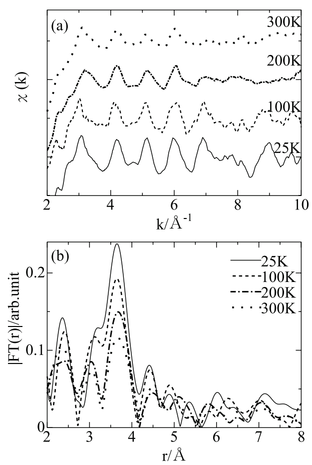

FIGURE 3. The EXAFS spectra of $Fe_{60}Ni_{40}$ with indicated processing times.

As shown in Fig. 4, the magnitude of the first peak in the Fourier transformed spectrum decreased gradually as the processing time increased. This suggests that the number of Fe-Fe direct bond decreased due to both the fracture of crystalline and the alloying with other kind atoms. The long range order was reduced gradually up to 6 hours processing and the order disappeared completely in 24 hours processing. We can see that the final material is almost in the amorphous state because XRD intensity of the alloy was reduced significantly and the medium range order in the Fourier transformed spectra disappeared completely.

The first peaks in Fig. 4 were filtered to be analyzed by the regularization method. Pair distribution functions for Fe-Fe and Fe-Ni pairs were obtained from the filtered spectrum. Figure 5 shows the pair distribution function obtained from the EXAFS spectra for samples milled for 12 (dot line) and 24 hours (solid line). As shown in Fig. 5, the Fe-Ni ordering increased while the Fe-Fe ordering decreased as the processing time increased.

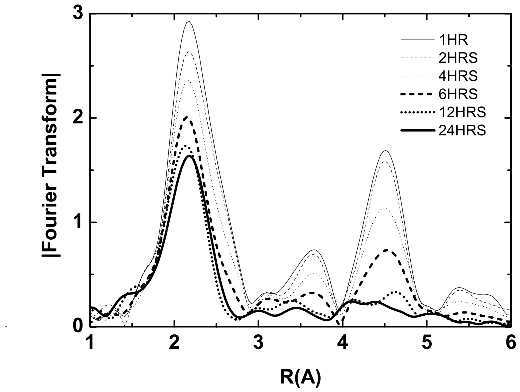

FIGURE 4. Fourier transform of the EXAFS spectra weighted with k^2 for $Fe_{60}Ni_{40}$ with indicated processing times.

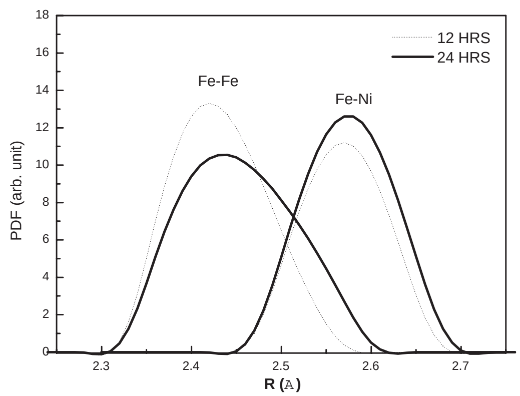

FIGURE 5. Pair distribution functions of $Fe_{60}Ni_{40}$ processed for 12 hours (dot line) and 24 hours (solid line).

This indicates that the Fe-Ni alloying progressed as the milling time increased.

XANES and EXAFS analysis shown in Fig. 2 and Fig. 3 give the self consistent results for the alloying process. The formation of alloy can be explicitly shown in the EXAFS spectra by the variation of amplitude and phase between 6, 12 and 24 hours milling times. The significant change of the phase confirms that new material was formed in this process.

CONCLUSION

In this work we analyzed the local structure of $Fe_{60}Ni_{40}$ metastable alloys produced by the mechanical alloying. The formation of the alloy was explicitly shown by the XRD and EXAFS analysis. Based on the EXAFS results we conclude that the alloying process was activated in about 6 hours and saturated in about 24 hours.

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