Local Ordering Study of Nanostructure FeMnAl Alloys

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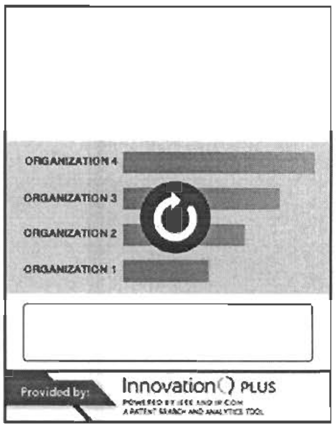

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$Fe_{55}Mn_{10}Al_{35}$ alloys were prepared by mechanical alloying. Local ordering around the central atom was examined with milling time by using extended X-ray absorption fine structure. The structural and magnetic evolution was analyzed by EXAFS and Mossbauer study as a function of milling time. The first shell of the Fourier transformed spectra changed with milling time, indicating a change in the local ordering around the central Fe atom. X-ray diffraction patterns indicate a bcc phase after 24 h milling time. For 1 h milled sample, Mossbauer spectra analysed three sextets as bcc-Fe phase, broad sextet and Fe surrounded Al and Mn phase. The magnetization showed decrease with milling time corresponding to magnetic dilution.

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I. Introduction

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Local Ordering Study of Nanostructure FeMnAl Alloys

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Abstract: $Fe_{65}Mn_{16}Al_{19}$ alloys were prepared by mechanical alloying as a function of milling time. Local ordering around the central atom was examined by using extended X-ray absorption fine structure (EXAFS). The structural and magnetic evolution was analyzed means by EXAFS and Mössbauer spectroscopy. The first shell of the Fourier transformed spectra changed with milling time, indicating a change in the local ordering around the central Fe atom. X-ray diffraction patterns indicate a bcc phase after 24 hrs milling time. For 1 hr milled sample, Mössbauer spectra analyzed three sextets as bcc-Fe phase, broad sextet and Fe surrounded Al and Mn phase. The magnetization showed decrease with milling time corresponding to magnetic dilution.

Introduction:

- Fe based metallic alloys have been extensively studied for the applications of the magnetic devices [1].
- The ferromagnetic behavior observed in Fe based alloys is due to the presence of Fe clusters on grain boundaries or due to the formation of Fe-rich magnetic phases even if the starting composition is non-magnetic element rich composition. However, the mechanism formation of these phases, the influence of structural properties that gives rise to ferromagnetism and the effect of nano sized structure on the magnetic properties of these systems are far from being understood, especially [2].
- Mechanical Alloying (MA) has been used widely to prepare metastable phases such as supersaturated solid solution, amorphous phases and nanostructure powders, starting from a mixture of elemental components or inter-metallic compounds in many alloy systems [3,4]. The research with milling time is convenient to investigate mechanism of magnetic properties due to the influence of structural change.
- The micro-structural properties are related to their atomic ordering in alloy composition. Atoms in solid have some regularity in the arrangement including the short range order and the long range order. The long range order can be examined by the X-ray diffraction (XRD) study, and the short range order is examined by the extended X-ray absorption fine structure (EXAFS) or scattering analysis with neutron or X-ray sources.
- EXAFS technique has been used to examine the local structure and the ordering around concerned atoms in amorphous, nanocrystalline and crystalline materials. Our previous studies were devoted to reveal the local atomic structure on binary or ternary alloys such as Fe-Si, Fe-C, Fe-Co-Cu, and Ni-Ga-Mn alloy [5].

Experiment:

- Fe-Al-Mn alloys were prepared by MA method using a SPEX 8000 mixer and mill with stainless steel balls and vial. Starting elements consist of appropriate amounts of Fe, Al and Mn (-80 mesh, 99.9%) powders.
- MA was performed under an Ar atmosphere to prevent oxidation during the alloying process. The milling time was varied from 1 hr to 24 hrs.
- Chosen composition was $Fe_{65}Al_{19}Mn_{16}$ in order to study the influence of Mn and Al in Fe based system on the structural evolution properties.
- Structural change for prepared samples was examined by XRD and EXAFS spectroscopy. XRD traces were obtained with a monochromatic Cu Kα radiation. EXAFS experiments were carried out at the beam line 3C1 EXAFS of the Pohang light source (PLS) in the Pohang Accelerator Laboratory (PAL) in Korea. The PLS was operated with an electron energy of 2.5 GeV and the maximum current of 220 mA.
- Mössbauer spectra were collected at 300 K, with a conventional constant acceleration spectrometer in transmission geometry with a ^{57}Co source diffused into a Rh matrix.
- The hyperfine structure was refined using the MOSFIT program [6].

Results and discussion:

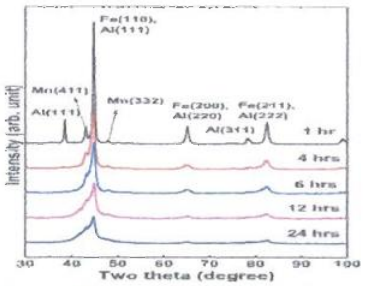

Fig. 1. XRD profiles of mechanically alloyed $Fe_{65}Al_{19}Mn_{16}$ powder with milling time.

- The 1 hr milled sample consists of mixed phase as bcc-Fe, bcc-Mn and fcc-Al.
- The Al and Mn diffraction peaks rapidly weak after 1 h alloying while the remaining Bragg peaks from the bcc-Fe phase became weaker and broader as the milling time increased. It means that the Al and Mn atoms diffuse into the Fe structure and then bcc phase is formed after 24 hrs milling time.

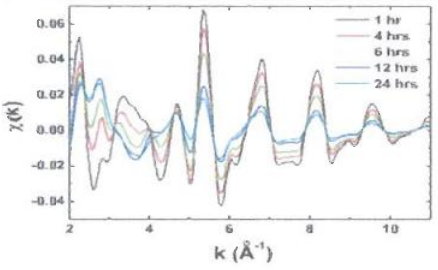

Fig. 2. EXAFS spectra of $Fe_{65}Al_{19}Mn_{16}$ alloys with milling time.

- Variations of EXAFS spectra are related to information on the structural changes of alloys at atomic scale.
- Mostly the reduction of the amplitude of EXAFS spectrum is caused by the disorder in local structure.
- The phase shift of EXAFS spectrum is related to the change of chemical order. Before 6 hours, the reduction of amplitude was dominant. This indicates that the deformation of structure with fracture and welding.
- However variations of the phase were dominant after 6 hours. This indicates that the start of alloying in this period. It seems that the amount of the diffusion of Al and Mn atoms into Fe structure increased gradually as the milling time increased.
- Especially, the spectra near k=6.3 $Å^{-1}$ shows that there was no significant change before 6 hrs. This means that no significant variation occurred in the local structure but the structure was changed gradually after 6 hrs.

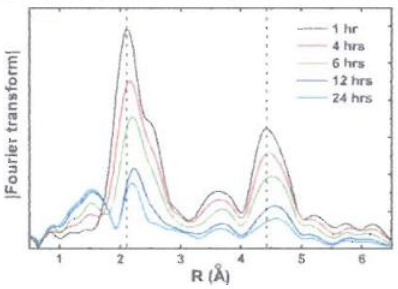

Fig. 3. EXAFS spectra of $Fe_{65}Al_{19}Mn_{16}$ alloys with milling time.

- The vertical dot lines indicate the first shell and the third shell of pure Fe which is guide line to compare with alloyed samples.
- The magnitude of the Fourier transformed spectra decreased gradually as the milling time increased. This suggests that the number of Fe-Fe bond decreased due to the diffusion of Al and Mn atoms into the bcc Fe shells.
- With increasing milling time, the first shell moves to longer atomic distance corresponding to the increase of Fe-Mn and Fe-Al bonding.
- The shift of first shell peak represents the change of local structure around the Fe atoms due to formation of alloys with milling time.
- The higher shell peaks showing the long range ordering in Fe-Fe in the Fourier transform of EXAFS spectra also decreased and shifted to longer atomic distance. This indicates that the long range order also reduced with the increase of milling time.

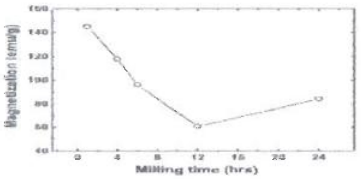

Fig. 4. Variation of magnetization for $Fe_{65}Al_{19}Mn_{16}$ alloys with milling times.

- The magnetization decreased with the increase of milling time. This indicates that the mixed powder undergoes magnetic transform when the alloying of the mixed powder is progressed.
- With increasing milling time, welding and fracture of the powder particles occurs repeatedly. Thus, inter-diffusion of the Fe and Al and Mn increases, resulting in magnetic dilution, i.e., decrease of the magnetization.

Conclusion:

- The local structural and magnetic properties of mechanically alloyed $Fe_{65}Al_{19}Mn_{16}$ powders were studied by using XRD, EXAFS, VSM measurement and Mössbauer spectrometry as a function of milling time.
- The structural evolution with XRD and EXAFS indicated the formation of bcc phase after 24 hrs milling time.
- Fourier transformed EXAFS spectra exhibited the atomic distribution surround Fe central atom with milling time.
- The Al and Mn atoms diffused into the bcc phase with the increase of milling time.
- For Mössbauer study represented the three kinds of component and the increase of bcc phase while the bcc-Al still was slightly remained after 24 hrs milling time.
- The role of Mn was being less important due to its low content.

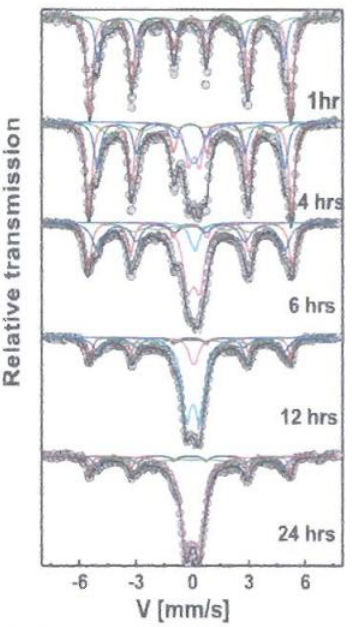

Fig. 5. Mössbauer spectra of $Fe_{65}Al_{19}Mn_{16}$ powders mechanically alloyed for several times. Their decomposition is also reported.

- For the 1 hr alloyed sample, the Mössbauer spectrum shows the presence of a magnetic sextet corresponding to bcc-Fe phase.
- As the alloying process progresses, the Mössbauer spectra are characterized by broadened lines due to the variation of Fe environment with Al and Mn diffusion.
- They can be decomposed into 3 magnetic components: one with an Hyperfine field of 33.1T, a second one at 30.6T and a third one at 27.5 T.
- The first sextet is clearly attributed to Fe in bcc environment which is consist of 8 nearest neighbors of Fe and 6 next nearest neighbors of Fe.
- The second one can be assigned to Fe with 7 nearest neighbors of Fe, 1 nearest neighbors of Al and 6 next nearest neighbors of Fe resulting from the milling.
- The third sextet can be assigned to 6 nearest neighbors of Fe, 2 nearest neighbors of Al and 6 next nearest neighbors of Fe.