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Submission date: 07-Apr-2019 02:32PM (UTC+0700)
Submission ID: 1107315012
File name: 2012-182-452-1-PB.pdf (632.24K)
Word count: 2451
Character count: 13132

Teknologi Indonesia 35 (2) Edisi Khusus 2012: 25–29

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Local Structure and Magnetic Properties of Ni-Al-C Nanocrystalline Alloys Fabricated by Mechanical Alloying Technique as a Function of Carbon Content

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ABSTRAK

Struktur lokal dan karakterisasi sifat magnetik dari paduan kristal nano $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ *(x = 0, 10, 30, dan 50 dalam %) yang disiapkan dengan Teknik Perpaduan Mekanik (MA) dengan waktu penggilingan selama 12 jam, telah dilakukan dengan detil. Ukuran dan bentuk dari kristal nano diuji dengan menggunakan* scanning electron microscopy *(SEM). Effek dari karbon pada sifat-sifat struktur telah diteliti dengan* x-ray diffractometer *(XRD) dan* Extended x-ray Absorption Fine Structure Spectroscopy *(EXAFS). Tentang sifat-sifat magnetik di uji dengan* Vibrating Sample Magnetometer *(VSM). Semua cuplikan merupakan perpaduan nano kristal. Harga maksimum dari* magnetic saturation *(Ms) adalah sekitar 0.7 emu/g dan harga minimum dari* coercivity *(Hc) adalah sekitar 135 Oe telah diperolehh untuk kandungan karbon sebesar 10%.*

Kata Kunci: *Paduan krsital nano* $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ *, struktur lokal, sifat magnetik, EXAFS, VSM.*

ABSTRACT

Local structural and magnetic characterization of $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ (x = 0, 10, 30, and 50 at. %) nanocrystalline alloys which were prepared by mechanical alloying (MA) technique at 12 hrs milling time, have been carried out in detail. The nanocrystalline size and shape were examined using scanning electron microscopy (SEM). The effect of carbon on structural properties has been investigated using x-ray diffractometer (XRD) and extended x-ray absorption fine structure spectroscopy (EXAFS), whereas magnetic properties was examined by vibrating sample magnetometer (VSM). All the samples revealed nanocrystalline alloys. The maximum magnetic saturation (*Ms*) at about 0.7 emu/g and the minimum coercivity (*Hc*) at about 135 Oe would be found for 10% carbon content.

Keywords: $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ *nanocrystalline alloys, Local structural, Magnetic Properties, EXAFS, VSM.*

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INTRODUCTION

Nanocrystalline materials obtained by high-energy ball milling are of great interest since it is known that in the nano regime, due to finite size effects, those materials may exhibit different electrical, magnetic, optical, and other physical properties.[1]

Mechanical Alloying (MA) is a non-equilibrium processing method that induces a high density of defects, with a comparable number of atoms in defect core and in regular lattice site. The MA process involves several steps: loss of long range order, reduced crystallite sizes, induced micro strains, loss of short range order and lattice expansion.[2] MA has been used widely to prepare meta-stable phases such as supersaturated solid solution, amorphous phases and nanostructure powders, starting from a mixture of elemental components up to inter-metallic compounds in many alloy systems.[3-4] This technique has been extensively used in inter-metallic compounds to prepare nanocrystalline structures.[2] MA has been proved to be an effective procedure for the synthesis of a large amount of nanostructure powders at low temperatures.[5]

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In the last century, a large number of amorphous transition-metal-metalloid (TM-M) alloys have been extensively investigated for their structural, electronic and magnetic properties. The magnetism in TM-M alloys is far from being understood.[6] Metal-metalloid systems such as Fe-Si and Fe-C have been extensively studied for application to magnetic and electronic devices, recently.[4] The role of the metalloid on the magnetic properties has been studied in Fe-, Co, and Ni- based binary systems.[6] Carbon added magnetic alloys such as Fe-C or Co-C, which are commonly used in industry, have been extensively studied due to their complicated nanocomposite structure originating on a strongly phase change dependent on the carbon concentration.[5]

NiAl inter-metallic compounds has characteristics such as low density, high strength and good corrosion and oxidation resistance.[7] In this work $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ (x = 0, 10, 30, and 50 at. %) alloys were produced by MA. The nano particles size and shape have examined using scanning electron microscopy (SEM). The structural evaluations of these alloys were examined by X-ray diffraction (XRD) and Extended X-ray Absorption Fine Structure (EXAFS) as a function of carbon content. The physical properties of the meta stable alloy can be explained by analizing the microstructure with local structural ordering,[8] while the magnetic properties were measured using a vibrating sample magnetometer (VSM). Special attention is paid to a model of the local structure and local ordering around Ni atoms in these alloy system using EXAFS technique.

EXPERIMENTS

$(Ni_{0.5}Al_{0.5})_{100-x}C_x$ (x = 0, 10, 30, and 50 at. %) or Ni-Al-C metastable alloys were prepared by mechanical alloying using a SPEX 8000 mixer with stainless steel balls and vial. The starting material was a mixture of pure Ni, Al and C powders (used commercial Ni, Al and C powders as the precursors). The weight ratio of balls-to-powder mixture was 5:1. The Ni-Al-C alloys were mixed and grounded for 12 hrs milling time. This process was performed in Ar ambient to prevent oxidation during the alloying process. After the preparation, the particle size and their shapes were checked by scanning electron microscope (SEM). Structural data were obtained by X-ray diffractometer (XRD) using the Cu-K_α radiation. The data were analyzed using Materials Data Inc. (MDI) software. The local structural data were collected from extended x-ray absorption fine structure spectroscopy (EXAFS) which was operated at energy of 2.5 GeV, and a maximum current of 200 mA. EXAFS spectra were obtained at Ni K-edge (8333.233 eV) in the transmission mode at room temperature. During the experiment, the sample's chamber was filled with pure nitrogen gas. The EXAFS data were analyzed by Ifeffit software, an interactive program for

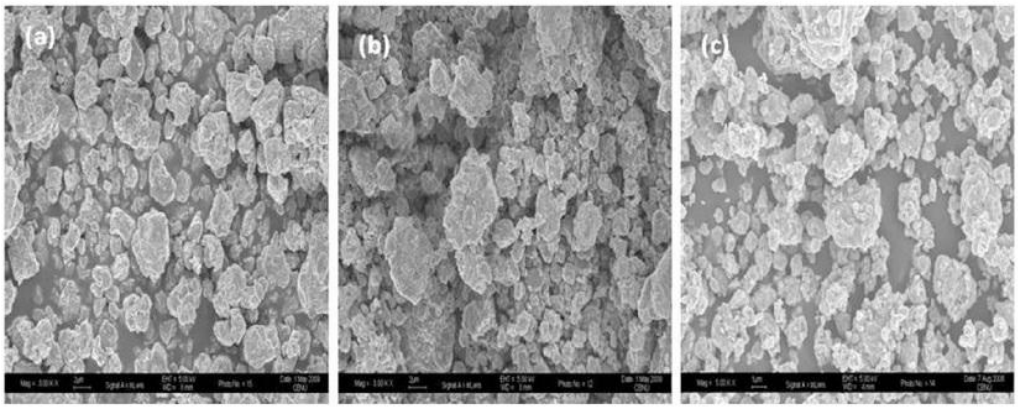

Figure 1. Typical SEM images of $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ nanocrystalline alloys for (a) x = 0, (b) x = 10 and (c) x = 50 with 12 hrs milling time.

XAFS analysis. The magnetic saturation (*Ms*) and coercivity (*Hc*) of the samples were measured using the vibrating sample magnetometer (VSM) at the maximum field of 1 kOe.

RESULTS AND DISCUSSIONS

Figure 1 (a), (b) and (c) present a typical SEM images which are showing the variations in particle shapes and sizes for the studied $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ powders by increasing carbon content x = 0, 10, and 50, respectively. The SEM images indicated that samples of all compositions are polycrystalline. The average grain size estimated from the SEM images is found to be decreased with the increase of carbon content. Although it is not shown here, particle shapes of all samples that were obtained quite similar for all carbon content.

Figure 2 shows the XRD results for the structural evaluation due to the MA process with the variation of carbon content. For samples with carbon content, x = 0, 10 and 30 are occurred substitution alloys which have seen AlNi peaks as the host with space group Pm-3m. While on sample with x = 50, it occurs an interstitial alloys which have observed at all individual peaks, i.e.

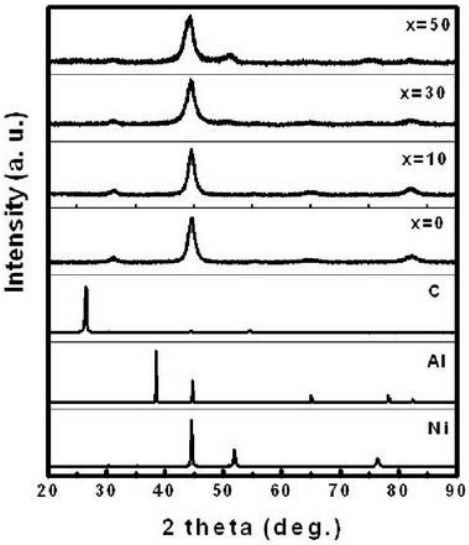

Figure 2. X-ray diffraction patterns for $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ (x = 0, 10, 30, and 50 at. %) produced by the mechanical alloying.

Ni, Al and C. This happen due to at high carbon content (x ≥ 50), the dissolution of C had not completed, because the huge volume fraction of C in the initial powder mixture are hindered the cold-welding and fracturing of metal powder.[9]

The variation of lattice parameters and crystallite size with carbon concentrations were shown in Figure 3. The lattice parameters and crystallite size were calculated from XRD data

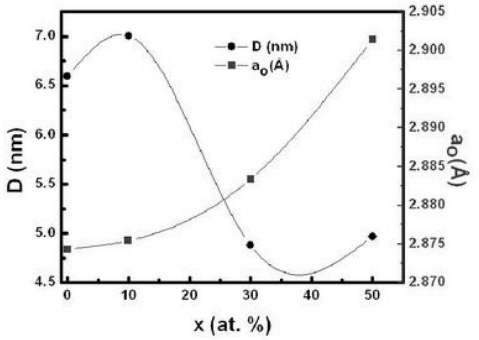

Figure 3. Variation of the crystallite size and lattice parameter with carbon concentration.

based on (110) peak used Bragg's and Scherrer's equations. One can see, the lattice parameters increased with the increaseing of the carbon content in both of the substitution alloys (x ≤ 30 %) and interstitial alloy (x ≥ 50%). This was most probably caused by increasing of the carbon concentration would decrease density that impacted to increase the lattice parameter. We can see also, the average crystallite size of all samples varied in the range of 5 to 7 nm. We have not found any data as comparison on any literatures about the dependency of crystallite size for the different composition of $(Ni_{0.5}Al_{0.5})_{100-x}C_x$.

EXAFS result is also used to examine the local structure of the samples. Variations in amplitude and the phase shift of an EXAFS peak give information about the structural changes at atomic scale occurring in the MA process. The reduction of amplitude in EXAFS spectrum might be caused by the spatial and chemical disorders. Most spatial disorder causes the variation of phases in EXAFS spectra.[3-5]

Figure 4 shows Fourier transformation of the EXAFS spectra of mechanical alloyed samples of

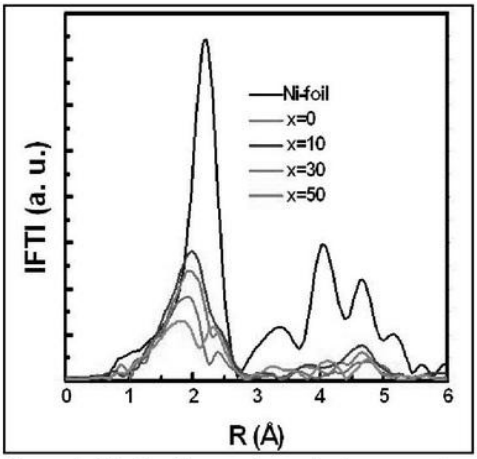

Figure 4. The Fourier transformation is measured at the Ni K-edge.

$(Ni_{0.5}Al_{0.5})_{100-x}C_x$ (x = 0, 10, 30, and 50 at. %) with 12 hrs milling time. The Fourier transformation of EXAFS spectra measured at the Ni K-edge for variation of carbon contents.

The Fourier transformation of the EXAFS spectra obtained from pure Ni foil at the Ni K-edge absorption is included as the comparison. The radial atomic density in the real space can be seen in the Fourier transformation spectra. [10] The magnitude of the Fourier transformed spectra decreased when carbon content increased. This suggests that the number of Ni-Ni bonds decreased due to the inter diffusion of Ni, Al and C atoms. Also, the first shell is shifted, corresponding to the increase of Ni-Al-C bindings. This indicates that the short and long-range orders along increased the with increasing of the carbon content. Clearly, the x = 0, 10, 30, and 50 samples exhibit different positions of phase in the first shell compared to the first shell of Ni-foil. It can be explained that the Ni-Ni order is changed due to the alloy formation of Ni-Al-C. Such results are in good accordance with the XRD data. These changes in the local structural order caused the variation of the magnetic properties of the samples.

Figure 5 shows the *k* weighted EXAFS spectra of $(Ni_{0.5}Al_{0.5})_{100-x}C_x$ (x = 0, 10, 30, and 50 at. %) powders. The significant change in the phase indicates that the alloying was occurred

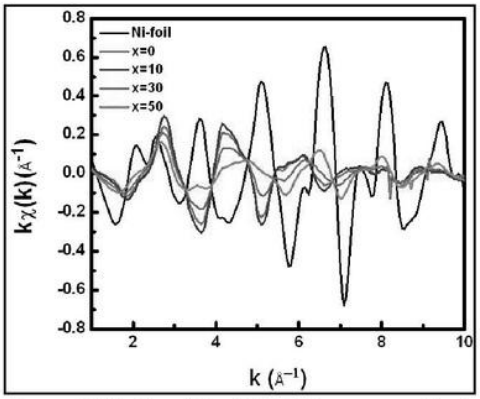

Figure 5. The *k* weighted EXAFS spectra mechanically alloyed for different of carbon content.

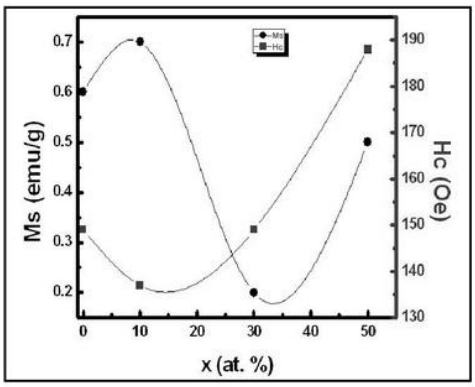

Figure 6. Variation of magnetization and coercivity as function of carbon content.

and a new phase was formed for all compositions which were different from that of Ni-foil signal.

Such situations strongly influence on the magnetic properties of the samples as presented on Figure 5. EXAFS data must undergo conversion from energy space *E* to momentum space *k* according to where the experimentally determined absorption edge is. It can be shown that the EXAFS oscillations are periodic in terms of *k* (the electron wave number) and the equation is frequently written as, where is the slowly varying background absorption (or attenuation) from an isolated absorbing atom and other processes. Above the absorption edge where is the absorption coefficient due to the particular edge of the element of interest in the sample and is the absorption coefficient of an isolated atom.[11-13].

Figure 6 shows the magnetization and coercivity as a variation of carbon content. It can be seen that for 10% carbon content, the magnetic saturation is maximum at about 0.7 emu/g and the coercivity is minimum at about 135 Oe. The coercivity decreased when carbon content is smaller than 10%. This is due to the dissolution of carbon which would be low for the samples of high carbon content. The dissolution of carbon had not completed, because the huge volume fraction of C in the initial powder mixture hindered the cold-welding and fracturing of metal powder. [10]

CONCLUSION

Based on EXAFS analysis, we found the alloyed metastable for all samples (x = 0, 10, 30, and 50 at %). It can be seen clearly on the intensity and phase of peak, especially when first shell is decreased and shifted compared to Ni foil peak. The minimum value of coercivity and the maximum value of the magnetization are occurred in a small amount carbon (around x = 10 at %).

ACKNOWLEDGMENTS

We thank to Dr. S. C. Yu and Dr. D. S. Yang who helped us in took the data at Applied Physics Laboratory, Chungbuk National University and Pohang Accelerator Laboratory in South Korea.

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Received: 25 May 2012 Revised: 30 May 2012 Accepted: 2 June 2012

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