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Structural and magnetic properties of NiAlC nano-particles

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Abstract : NiAlC nano-powder alloys are prepared by the mechanical alloying process. The magnetic and structural properties of these alloys are studied as a function of milling time and variation of carbon content. Milling times are 1, 2, 4, 6, and 12 hrs for Ni₄₅Al₄₅C₁₀ samples and 12 hrs for (Ni_{0.5}Al_{0.5})_{100-x}C_x (x = 0, 10, 30, 50, 70) samples. The effect of carbon content [(Ni_{0.5}Al_{0.5})_{100-x}C_x] and milling time [Ni₄₅Al₄₅C₁₀] on structural properties have been investigated by using x-ray diffractometer (XRD) and extended x-ray absorption fine structure spectroscopy (EXAFS). Magnetic properties are examined by vibrating sample magnetometer (VSM). Both XRD and EXAFS patterns are shown the alloys occurred after 4 hours of milling time. The nano particles size and shape have examined using scanning electron microscopy (SEM). Magnetic saturation (Ms) is decreased with milling time might be due to two reasons: substitution of atoms Ni by atoms Al and C are increased in resulting magnetic dilution, and also by decreasing of particle size. The coercivity (Hc) is increased with increasing carbon content.

Keywords : NiAlC nano-particles, EXAFS, XRD, and VSM

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Introduction :

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 [1]. 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 or inter-metallic compounds in many

alloy systems [2,3]. This technique has been extensively used in inter-metallic compounds to prepare nano-crystalline structures [1]. MA has proved to be an effective procedure for the synthesis of a large amount of nanostructure powders at low temperatures [4].

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 [5]. Metal-metalloid systems such as Fe-Si and Fe-C have been extensively studied for application to magnetic and electronic devices, recently [3]. The role of the metalloid on the magnetic properties has been studied in Fe-, Co, and Ni- based binary systems [5]. Carbon added magnetic alloys such Fe-C or Co-C, which are commonly used in industry, have been extensively studied due to their complicated nano-composite structure originating on a strongly phase change dependent on the carbon concentration [4]. NiAl inter-metallic compounds have low density, high strength and good corrosion and oxidation resistance [6].

In this work $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ ($x = 0, 10, 30, 50, 60$) alloys were produced by MA. The structural evaluations of these alloys are examined by X-ray diffraction (XRD) and Extended X-ray absorption Fine Structure (EXAFS) as a function of milling time and carbon content. The magnetic properties are measured by using a vibrating sample magnetometer (VSM). Special attention is paid to model the local structure and local ordering around Ni atoms in these alloy system using EXAFS technique. The nano particles size and shape have examined using scanning electron microscopy (SEM).

Experiment

$(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ (x is the atomic percent of carbon) metastable alloys are prepared by the mechanical alloy using a SPEX 8000 mixer and mill with stainless steel ball. The starting material was a mixture of pure Ni, Al, and C powders with different compositions of $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ ($x = 0, 10, 30, 50, 70$). In the next step $\text{Ni}_{45}\text{Al}_{45}\text{C}_{10}$ alloys are prepared with different milling times (1, 2, 4, 6, and 12 hours). The mechanical alloying is performed in Ar atmosphere to prevent oxidation during the alloying process. The ball to powder weight ratio is 5:1. The variations of structure are examined by XRD and EXAFS. XRD data are obtained with a monochromatic Cu K_α radiation. Particle size is calculated using Scherrer formula from XRD data. EXAFS experiments are carried out at the beam line 3C1 EXAFS of the Pohang Light Source (PLS). The PLS was operated with an electron energy of 2.5 GeV and maximum current of 200 mA. The EXAFS spectra were obtained near Ni K edge (8346.59 eV) in the transmission mode at room temperature. The ion chambers are filled with the pure nitrogen gas. EXAFS data are analyzed with FEFF software. The nano particles size and shape have been examined using scanning electron microscopy (SEM). Magnetic moments and coercivity of the samples are measured by using the vibrating sample magnetometer (VSM) with the maximum field 1 kOe.

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Results and Discussion :

Fig. 1 shows the XRD results for the structural evaluation due to the MA process with 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. Beginning of $x = 50$ until 70 there are occurred interstitial alloys which have observed all individual peaks, i.e. Ni, Al and C. It was happen

because for high carbon content ($x \geq 50$), the dissolution of C is not completed, because the huge volume fraction of C in the initial powder mixture are hindered the cold-welding and fracturing of metal powder [5].

The variation of lattice parameters and particles size with carbon concentrations are shown in Fig. 2. The lattice parameters and particle sizes (Debye Scherer formula) are calculated from XRD data. The lattice parameter is increased with increasing carbon content up to 50 % and then decreased with increasing 'x'. This conforms the formation of substitutional alloys below $x \leq 50$ % and above $x > 50$ % interstitial alloy. The average particle size of all samples varied in the range 5 to 9 nm. In the present work the particle size is decreased with increasing carbon content up to $x = 50$ % beyond these compositions particle size is increased. No data on the dependence of particle size on composition $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ is available in the literature for comparison.

Fig. 3 shows the XRD spectra of $\text{Ni}_{45}\text{Al}_{45}\text{C}_{10}$ for the structural evaluation due to the MA process with variation of milling time. Alloying formation is occurred from 4 h milling time onward which has occurred broadening of peaks but at the same time peak intensity is decreased due to perfect alloy formation. From these XRD results the lattice parameter is increased from 4.079 - 4.082 Å and the particle sizes is decreased from 30 – 7 nm with increasing the milling time.

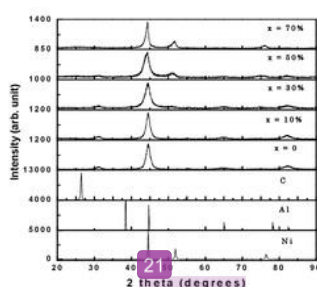


Fig. 1. X-ray diffraction patterns for $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ ($x = 0, 10, 30, 50, 70$) produced by the mechanical alloy.

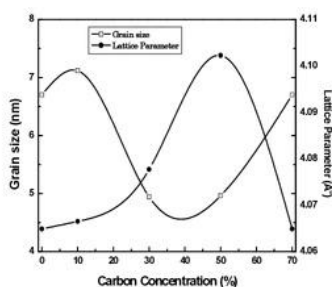


Fig. 2 Variation of lattice parameters and particle sizes with carbon concentration.

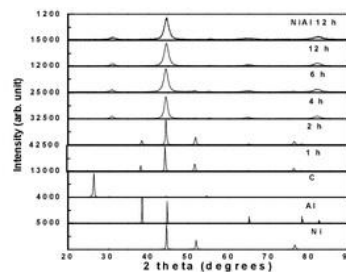


Fig. 3. XRD patterns of mechanically alloyed $\text{Ni}_{45}\text{Al}_{45}\text{C}_{10}$ and $\text{Ni}_{50}\text{Al}_{50}$ for variation of milling time.

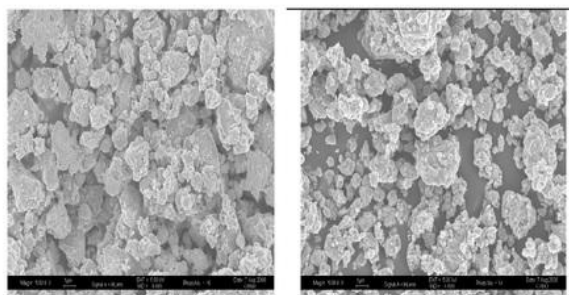


Fig. 4 Typical SEM images of $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ nano-powders for (a) $x = 10\%$ (b) $x = 50\%$ and milling time at 12 hrs.

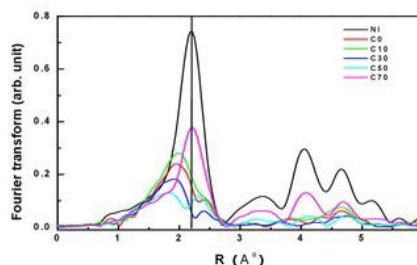


Fig. 5. Fourier transform of EXAFS spectra of mechanical alloyed $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ ($x=0, 10, 30, 50, 70$) with milling time 12 hrs.

Fig. 4 (a) and (b) present typical SEM images showing the variations in particle shape and size in studied $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ powders with increasing carbon content $x = 10$ and $x = 50$ %, respectively. The SEM images indicated that samples of all compositions are polycrystalline. The average grain size estimated from the SEM images is found to decrease with increase of carbon content up to $x = 50$ % and then again grain sizes are increased with carbon content. Similar particle shapes of SEM pictures are obtained for all other carbon content samples. All the samples are very small particles to the surfaces of big particles occurred and the powder particles tended to form a matrix of randomly welded thin layers of highly dense particles. Similar particle shape of SEM images of $\text{Ni}_{45}\text{Al}_{45}\text{C}_{10}$ also observed with different milling times but not shown here.

Fig. 5 shows Fourier transform of the EXAFS spectra of mechanical alloyed $(\text{Ni}_{0.5}\text{Al}_{0.5})_{100-x}\text{C}_x$ with different carbon concentrations ($x = 0, 10, 30, 50, 70$) and milling time at 12 hrs. There are two groups trend local structures, i.e. with $x = 10, 30$, and 50 in one group and with $x = 70$ % in another group. Also the Fourier transforms of the EXAFS spectra obtained from pure Ni foils at the Ni K edge absorption is included for comparison. The Fourier transform EXAFS spectrum of $x = 70$ % look similar with pure nickel spectrum, indicating that local structure surrounding Ni atoms has a behavior similar to the pure Ni. The alloys have not happened because cold-welding has not occurred. Local structures with $x = 10, 30$, and 50 have similar trend. Intensity is decreased with increasing the carbon content. The difference of intensity comes from the different amplitude reduction factor of Ni atoms. It is also noted that the first shell slightly shifted to shorter distance which is corresponding to a changing of the dominant atoms around the central atoms.

Fig. 6 shows the Fourier transform of EXAFS spectra of mechanically alloyed $\text{Ni}_{45}\text{Al}_{45}\text{C}_{10}$ and $\text{Ni}_{50}\text{Al}_{50}$ with different milling times (the vertical solid line indicates the location of Ni first shell). With increase alloying time, the intensity is reduced corresponding to the change of nearest neighbor structure, that is, Ni-Ni bonding changes to Ni-Al or Ni-C. For 1 and 2 hrs milling time the alloys have not occurred yet. Start from the alloying time of 4 hrs, the atomic distance of 1st shell is significantly shifted shorter distance. Inter-diffusion between Ni, Al and C atoms are happen and the new structures have occurred. The local structure of NiAl alloy without carbon is look similar with NiAlC alloy, because of carbon atoms become the substitutional atoms in NiAlC alloys. Both of them have alloying time 12 hrs.

Fig. 7 shows the magnetization and coercivity as a variation of carbon content. For carbon content 10 %, the magnetic saturation is maximum, but the coercivity is minimum then increased follow the added of carbon content. The coercivity is decreased for carbon content smaller than 10 % [7]. The dissolution of carbon would be low for the samples of high carbon content. The dissolution of carbon is not completed, because the huge volume fraction of C in the initial powder mixture is hindered the cold-welding and fracturing of metal powder [5].

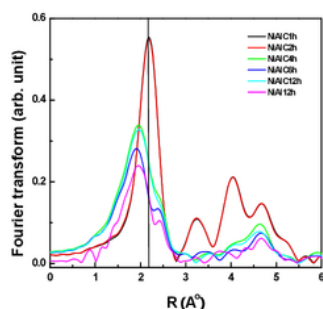


Fig. 6. Fourier transforms of EXAFS spectra of mechanically alloyed $\text{Ni}_{45}\text{Al}_{45}\text{C}_{10}$ and $\text{Ni}_{50}\text{Al}_{50}$ with milling times.

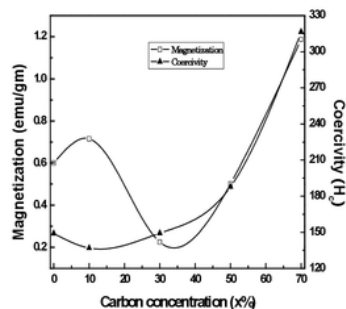


Fig. 7. Variation of magnetization and coercivity as function of carbon content

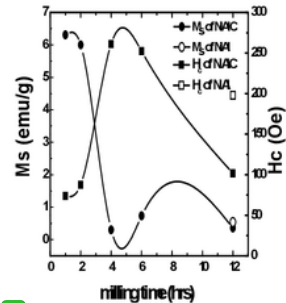


Fig. 8. Variation of magnetization and coercivity as function of milling time

Figure 8 shows the variation of magnetization and coercivity with milling time. Magnetization rapidly decreased in the initial stage of milling time due to the repeated welding and fracture process. After 4 hrs milling time, the magnetization is almost stable and reached 0.36 emu/g. This variation in the magnetization could come from the dilution of magnetization Ni by Al and C with increasing milling time. The variation of coercivity is related to the particle size variation. With increased milling time, the particle size is reduced to nano-sized structure as shown in XRD result. In the large particle size, the coercivity comes from the multi-domain status and it will be changed to single-domain status with an increase in milling time. In the figure, the decrease of coercivity indicates the increase of the single-domain status due to the particle size reduction.

Conclusion :

In this work we analyzed the structure and magnetic properties of NiAlC metastable alloys produced by the mechanical alloying. Mechanically alloyed NiAlC for different of both carbon content and milling times are studied by XRD, EXAFS, SEM and VSM. In the initial stage of alloying time, the Ni and Al phase rapidly decreased. The alloys is formed after 4 hrs milling time, structural phase showed fcc NiAlC structure and there is no long range ordering around Ni central atom. The magnetization shows small value compared to pure Ni because of magnetic dilution. Magnetic saturation and coercivity strongly depend on Ni content and particle sizes. Carbon in small content (around $x = 10$ at. %) can be decreasing coercivity and increasing magnetization.

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