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Research Article

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Microstructure Evolution and Mangnetique Proprieties of Nanocrystalline $Fe_{60} Cu_{30}Al_{10}$ Prepared by Combustion Processes

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Abstract

Nanostructured disordered iron-aluminium-copper alloy of Fe-30% AI-10% Cu composition was prepared by Self-propagating high-temperature synthesis (SHS) and thermel explosin (TE) techniques of mechanically activated mixture of Fe, AI, Cu powders. The transformations occurring in the material during combustion were studied with the use of X-ray diffraction. Auger spectroscopy and atomic emission spectroscopy (AES) was used to determine the phase composition of the phase formation. Finally, the Magnetic properties were also investigated, for combution processers the magnetic behavior slightly softened becoming a semi hard ferromagnetic.

Keywords: Combustion processes; Noncrystalline; $Fe_{60} Cu_{30}Al_{10}$; AES; Magnetic

Introduction

Recently a great deal of interest has been generated in the synthesis of advanced engineering materials in their stable, metastable, nanocrystalline and amorphous phases and making their scope much wider. For that, nanocrystalline alloys are relatively new functionally important materials that demonstrate superior properties in a wide range of conditions and have a number of important applications. Mechanical alloying (MA) is one of the most promising and rapidly developing methods for nanocrystalline materials on an industrial scale [1,2]. Some properties, such as high strength and hardness [3,4] ductilization of brittle materials [5,6] and enhanced diffusivity [7], that are superior to those of the conventional materials, may result from the nanocrystalline structure. In comparison with the solidification route, MA is an expensive and simpler technique to process the material into nanocrystalline or amorphous state. Furthermore, it is easier to produce the nanocrystalline phase in a wider composition range by MA than by solidification methods. Additionally, since MA processing is carried out in the solid state, phase diagram restrictions do not seem to apply to the phases produced by the technique [1,8,9]. Further, it appears that the mechanism by which a nanocrystalline or an amorphous phase is formed is different between solid-state processed and liquidstate processed alloys. The Fe-Al alloy systems are attractive for their potential magnetic and mechanical applications [10,11]. Moreover, they have n excellent corrosion resistance at elevated temperatures, and a low cost and relatively low density compared to Ni-, Co-, and Fe-based superalloys [12-15]. FeAl has a B2 (CsCl) structure and exists over a wide range of Al concentration at room temperature (34-52% Al) [16].

In recent years, a number of studies have been reported on mechanical alloying of Fe-Al and Fe-Cu binary systems. It is established that the formation of the supersaturated solid solution (SSS) α -Fe(Al) as a final product of MA takes place with $x \leq 60$ at.% Al though in a number of papers the formation of SSS was found with x = 75 at.% Al [17-22]. The Fe-Cu system has negligible mutual solid solution solubility in equilibrium at temperatures below 700°C (a miscibility of approximately 3%) [23,24] because of the large positive enthalpy of mixing ($\Delta H_{mix} = 13$ kJ mol⁻¹ for Fe-Cu system of 50: 50 in mol % according to the Miedema model) [25]. Eckert et al. [26] have reported that single phase FCC alloys and single phase BCC alloys are formed by MA in the FexCu100-x system with x b 60 and with x>80, respectively.

Moreover, Majumdar et al. [27] and Gaffet et al. [28] have deduced that the formation of the BCC Fe-Cu solid solution is restricted to 0-20 wt.% Cu. According to their investigation, this process occurs in two steps: a nanocrystallization step and an Fe (Cu) and/or Cu(Fe) formation step. Knowledge of ternary additions to B2 alloy systems is one of the key points in understanding and controlling the mechanical proprieties of these materials and is critical to alloy design. Moreover, successful application of Fe-Al based intermetallics at elevated temperatures is critically dependent on improvement of their creep resistance [29].

Strengthening by second phase particles represents a potential method in this direction [30,31]. Disordering of FeAl intermetallics has been a subject of numerous theoretical [32,33] and experimental investigations [34-37] over the past two decades. Their results indicated that crushing creates, through plastic deformation, a large number of antiphase boundaries leading to antiphase domains with average linear dimensions of about six atomic distances. The influence of structural disorder on the magnetic properties has been evidenced, in FeAl, in different types of microstructures such as cold worked single crystals [33,38-40] quenched or cold worked polycrystalline materials [33,40,41] or ball milled and mechanically alloyed nanoparticles [33,42-44]. It is well established that magnetic properties of FeAl alloy, show a strong dependence on the degree of atomic order [38]. Disordering the alloy induces paramagnetic \rightarrow ferromagnetic transitions. It was actually argued [33] that the origin of the magnetic interactions in disordered FeAl may not arise solely from nearest-neighbor magnetism but also from changes in the band structure of the material induced by Δa (increase in the lattice parameter). Noguès et al. [33], have shown, besides the effects of the local environment of the magnetic ions, disorder-induced lattice changes and they demonstrate experimentally and theoretically that about 35-45% of the magnetic moment of Fe₆₀Al₄₀ alloy arises from lattice expansion effects induced during

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the disordering process. MA of magnetic Fe-based alloys leads to the formation of supersaturated solid solution, multiphase or possibly amorphous structure. Increasing milling time is accompanied by both a decrease of grain size and an increase of internal microstrain, which is a common behavior to all metallic systems prepared by mechanical alloying. In general, some magnetic properties can be improved when the grain size is reduced to the nanoscale, while the presence of stresses and defects introduced by MA impairs the magnetic behavior; the overall magnetic property is a competition between decreasing grain size and increasing strain [45].

In the present work, $Fe_{60}Al_{30}Cu_{10}$ powders were mechanically alloyed by high energy ball milling. The fabrication of $Fe_{60}Al_{30}Cu_{10}$ by different combustion techniques such as SHS [46-48] and electrothermal explosion (ETE). The advantage of this two methods is shown by comparing structural properties of $Fe_{60}Al_{30}Cu_{10}$ synthesized both by SHS and the ETE.

The crystalline phases of this material are examined magnetic properties of the bulk such as Hc, Js, Br are studied by a vibrating sample magnetometer (VSM) under the applied field of 1500 kA/m.

Experimental

Elemental powders of Fe, Al and Cu with purity of 99.99% of the nominal composition $Fe_{60} Cu_{30}Al_{10}$ powder (in wt.%) were mixed and milled for 10 h. The mechanical alloying experiments were performed in a high energy planetary ball mill (Fritsch Pulverisette 6) in argon atmosphere using stainless steel vials and balls with diameter of about 12 mm. The rotation speed could be varied within the range 400–600 rpm. In order to avoid oxidation during alloying, the ball mill was filled with high purity argon gas. The vial was opened in mill for 60 and 120 min to assure high homogenization. The powders were compacted into small discs (2-4) × 13 mm in size at the compacting pressure P = 7000 psi and then subjected to SHS (Self-propagating high-temperature synthesis), thermal explosion, and annealing upon heating with a heavy current (400 A) for 1 to 4 min under an Ar pressure of 10 atm (Figure 1).

The structural properties were determined by XRD using a Philips diffractometer (Co-K α radiation). Further structural characterizations

were carried out by energy dispersive X-ray microanalysis (EDX), scanning electron microscopy (SEM). AES and depth profiling measurements were carried out in the UHV cell of a scanning Auger spectrometer (Microlab VG MKII) equipped with a hemispherical analyzer. In addition we have used the using a vibrating magnetometer (VSM) to explore the magnetic properties and domains of the $Fe_{cn}Al_{a0}Cu_{10}$ phase after synthesis.

Results and Discussion

Figures 2a-2c shows the SEM pictures of raw Fe, Cu and Al powders. The powder particles of Cu were found to be nearly spherical shaped with an average size in the range of 5-50 µm. the Al and Fe elemental particle powders with irregular shapes. For the powders subject to 10 h of milling, it is very clear from Figure 3 that all the initial shape of the powders was changed and powders having composite structure started to form. An increase in the particle size is noticed, indicating a "primary" welding of very small particles to the surface.

Figure 4 shows the X-ray diffraction patterns of the $Fe_{60} Cu_{30}Al_{10}$ mixture at milling time of 10 h of the combustion products formed in SHS (Self-propagating high-temperature synthesis) and ETE reaction. Clearly, in Figures 4a and 4b after a combustion of SHS products and ETE reaction, the peaks on phase formation of Nanocrystalline Fe(Al, Cu) appeared in all the samples (this is can be explained par the formation of the BCC Fe(Al, Cu)) and though a little FeAl phase. Its mean crystallite size are refined as the milling time increases and reached a final value of about 20 nm. The mean crystallite size at 10 h of milling is on the same level in the two coexists, Fe(Al) and Fe (Al, Cu) phases.

According to this model, when the grain size is smaller than magnetic exchange length L_{ex} , the origin of the soft magnetic properties in the nanocrystalline materials is ascribed to average out the magnetocrystalline anisotropy. Theoretically, as the L_{ex} for Fe-based alloys is of the order of 20-30 nm that corresponds approximately to D value of the Fe (Al, Cu) alloy in the present study at earlier stage of processing. Further milling does not reduce the grain size appreciably. For this reason the coercivity does not show great changes after 4 h of milling.



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Figure 2: SEM images of the powders (a) Fe (b) Cu, (c) Al.



Figure 3: SEM micrographs of the powder mixture of Fe, Al and Cu milled for 10 h.

It suggests that SHS and ETE is an effective methods to accelerate the formation of BCC Fe(Al, Cu) phase. X-ray diffraction is a convenient method for determining the mean size of nanocrystallites in nanocrystalline bulk materials.

This can be attributed to the fact that "crystallite size" is not synonymous with "particle size", while X-Ray diffraction is sensitive to the crystallite size inside the particles. From the well-known Scherrer formula the average crystallite size, *L*, is:

$$L = \frac{K\lambda}{\beta \cos \theta} \tag{1}$$

Where λ is the X-ray wavelength in nanometer (nm), β is the peak

width of the diffraction peak profile at half maximum height resulting from small crystallite size in radians and K is a constant related to crystallite shape, normally taken as 0.9. The value of β in 20 axis of diffraction profile must be in radians. The θ can be in degrees or radians, since the cos θ corresponds to the same number.

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Table 1 shows the crystallite size values for different peaks at combustion of SHS products and ETE reaction. The XRD patterns are observed in Figures 4a and 4b with gradual sharpness of the peaks as the soaking combustion processes (SHS,ETE), indicating the growth and increase of crystallite size. Relatively gradual decrease in β .cos θ and almost increase in *L* values L= (const/ β .cos θ) is observed with the increase of 2 θ . The AES data in Figures 5a and 5b (corelevel excitation) show the peaks around the kinetic energies $E_k = 505$, 918, 1392 and 1833 eV corresponding to O, Cu, Al and Fe respectively, according to the energy Atlas of AES.

In Figures 6a and 6b it is shown the hystere sis curves at 5 K and 300 K of Fe₆₀ Cu₃₀Al₁₀ alloy produced by SHS (Self-propagating hightemperature synthesis) and ETE(electrothermal explosion). It can be seen that increasing the temperature, all the magnetic parameters studied (Ms, Hc, Mr and (Mr/Ms)) as expected, decrease due to the thermal excitation (The rapid decrease in Ms is mainly due to the early interaction between the Fe atoms which are ferromagnetic with Al and Cu atoms that are non-ferromagnetic in nature. Al decreases the magnetic moment of individual Fe sites due to a decrease in the direct ferromagnetic interaction between Fe-Fe sites, and also an antiferromagnetic super-exchange interaction between Fe sites mediated by Al atoms as suggested by Plascak [49]. Increasing temperature in a ferromagnetic material causes an increase of the thermal vibrations of atoms and therefore the magnetic moments are free to rotate and are then arranged randomly. For this reason the magnetic parameters are affected by temperature, reducing them quantitatively and significantly. According to studies carried out by Hu et al. [50] and Amils et al. [51] among others, it has been confirmed the transition from paramagnetic to ferromagnetic state in 48Fe₂₂Al₂₀Cu at % alloy by MA even at room temperature.

Conclusions

Nanocrystalline Fe(Al, Cu) powder with crystallite of 10 nm has been successfully synthesized from SHS (Self-propagating hightemperature synthesis) and ETE (electrothermal explosion). The single



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$2\theta_{hkl}$ Degrees	Values of β .cos θ for different peaks		Calculated L (nm)
	SHS products	ETE products	
35,341	-	3,84×10 ⁻³	35,93
38,155	4,6×10 ⁻³	4,6×10 ⁻³	30
40,218	5,58×10 ⁻³	5,58×10 ⁻³	24,73
42,761	7,4×10 ⁻³	7,4×10 ⁻³	18,65
45,011	5,50×10 ⁻³	5,50×10 ⁻³	25,09
46,75	-	12,31×10 ⁻³	11,21
48,66	-	3,36×10 ⁻³	38,33
60,59	7,1×10 ⁻³	7,1×10 ⁻³	19,43
73,01	-	8×10 ⁻³	17.25
81,20	6,5×10 ⁻³	-	21.23
96,20	6,7×10 ⁻³	-	20,59

Table 1: Calculation of the crystallite sizes of Fe-30% AI-10% Cu composition was prepared by Self-propagating high-temperature synthesis (SHS) and thermel explosin (TE) techniques of mechanically activated mixture of Fe, AI, Cu powders.





phase of BCC Fe (Al,Cu) was formed. It suggests that SHS and ETE is an effective method to accelerate the formation of Fe (Al,Cu) phase. From the magnetic point of view there is a correspondence between microstructural evolution and magnetic behavior in the form of that decreasing the crystallite size increases the magnetism by increasing coercivity and squareness ratio. Consequently as low crystallites size is, stronger hard ferromagnetic material results. Due to thermal vibrations higher temperature produces a decrease in magnetic properties.

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