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# Structural characterization and magnetic properties of $\text{Al}_{82}\text{Fe}_{16}\text{TM}_2$ (TM: Ti, Ni, Cu) alloys prepared by mechanical alloying

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## ABSTRACT

In this work, the structural evolution of Al–16 at.%Fe–2 at.%TM (Transition Metals (TM): Ti, Ni, Cu) alloys during mechanical alloying and their magnetic properties were investigated. The evolution of the phase composition and microstructure of the alloys with the milling time was studied using X-ray diffraction (XRD) and Transmission Electron Microscopy (TEM). The phase composition of the alloys was determined using the Rietveld refinement of the XRD profiles. It was shown that the interaction between the components of the powder mixtures during milling started with dissolution of Fe in the Al crystalline lattice and Al in the Fe crystalline lattice. Upon further milling, ferromagnetic  $\text{AlFe}_3$  (DO3) formed and further transformed into paramagnetic bcc-AlFe and later into an amorphous phase. It was found that the TM alloying elements significantly influence the kinetics of the transformations during milling: the  $\text{Al}_{82}\text{Fe}_{16}\text{Ti}_2$  alloy was fully amorphous after 40 h of milling, the  $\text{Al}_{82}\text{Fe}_{16}\text{Ni}_2$  alloy required 50 h of milling to achieve complete amorphization, and the  $\text{Al}_{82}\text{Fe}_{16}\text{Cu}_2$  alloy was only partially amorphous after 60 h of milling. The interpretation of the observed alloying effect has been proposed. The magnetic properties of the alloys were correlated with the results of the structural characterization.

## 1. Introduction

Mechanical alloying (MA) is a solid-state powder processing technique that offers possibilities to produce both stable and metastable phases [1,2]. During milling, the powder particles experience repeated deformation, while alternating fracture and welding processes lead to homogenization of the chemical composition, refinement of the crystallite size and defect generation in the crystalline structure of the materials. Depending on the energy of the milling process and thermodynamic properties of the constituents, a mixture of metallic powders can be processed into intermetallic phases, solid solutions, nanocrystalline composite structures and amorphous alloys. Intermediate states obtained by mechanical alloying are usually complex states in terms of the crystalline structure presenting multiple possibilities for the materi-

als design and property tailoring.

The preparation of Al-based amorphous alloys by mechanical alloying was reported in refs [3–5]. The structure of alloys containing > 80 at.% of Al is quite complex. Different phases, such as bcc and fcc phases, Fe(Al) supersaturated solid solutions and amorphous phases have been reported in these alloys [6,7]. As intermediate phases formed in these alloys during mechanical alloying can be both magnetic and nonmagnetic, the overall magnetic properties of the alloys depend on their phase composition and microstructure. It is known that the coercive force of nanocrystalline soft magnetic materials strongly depends on the crystallite size. By using different milling durations, alloys with different values of the coercive force can be produced. Furthermore, it is of interest to study the saturation magnetization of the alloys since the reduction of the grain size to the single magnetic

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domain size offers a possibility to eliminate the influence of magnetic walls [8].

In the present work, we report the preparation of Al–16 at.%Fe–2 at.%TM (Transition Metals (TM): Ti, Ni, Cu) alloys by mechanical alloying. The structural changes in the alloys up to complete (or partial) amorphization with the progress of milling were investigated and the relationships between the microstructure and magnetic properties of the alloys were determined.

## 2. Experimental

Elemental powders of Al (–100, +325 mesh), Fe, Ti and Ni (10  $\mu\text{m}$ ) were used as the starting materials (> 99.5% purity). The powder mixtures with the Al–16 at.%Fe–2 at.%TM (TM: Ti, Ni, Cu) nominal compositions were prepared.

The mixtures were ball-milled in a Fritsch Pulverisette-6 planetary ball mill at 300 rpm using stainless steel vials and hardened steel balls with the ball to powder weight ratio of 20:1. In order to prevent the sticking phenomenon, *n*-hexane was used as a process control agent. The milling process was periodically interrupted (every 30 min) to avoid excessive heating of the material inside the vial.

The evolution of the crystalline structure of the powders was studied by X-ray diffraction (XRD) using Cu-K $\alpha$  radiation. Rietveld refinement was used to analyze the XRD patterns employing the Maud software [9,10].

A high-resolution transmission electron microscope (TEM-JEOL JEM-2100e) was used to observe the fine structure of the alloys. Selected area electron diffraction (SAED) patterns were taken to analyze the details of the crystalline structure of the alloys. The SAED patterns were indexed using the JEMS software [11].

Magnetization curves of Al–16 at.%Fe–2 at.%TM (TM: Ti, Ni, Cu) milled samples were measured by vibrating system magnetometers (VSM, MicroSense, EV9) in which each sample was measured five to ten times and an average value was obtained.

## 3. Results

### 3.1. Evolution of the crystalline structure

The XRD patterns of the Al–16 at.%Fe–2 at.%TM (TM: Ti, Ni, Cu) powders milled for different milling times are shown in Fig. 1. In the patterns, the intensities are plotted against the scattering vector ( $Q = 4\pi\sin\theta/\lambda$ , where  $\theta$  is the Bragg's scattering angle and  $\lambda$  is the radiation wavelength). A general observation can be made that under continuous milling, Al and Fe reflections become wider, which can be caused by a decrease in the crystallite size and accumulation of defects in the crystalline structure of the metals. The Al (111) reflection ( $\sim 27 \text{ nm}^{-1}$ ) shifts slightly towards higher scattering vectors ( $Q$ ) as the milling time increases. At the same time, the Fe (011) reflection ( $\sim 31 \text{ nm}^{-1}$ ) shifts towards smaller  $Q$ . These effects indicate that the corresponding lattice spacing of  $\alpha$ -Al decreases and that of  $\alpha$ -Fe increases, which can be due to the formation of  $\alpha$ -Al(Fe) and  $\alpha$ -Fe(Al) solid solutions.

Using the Al-Fe binary phase diagram [12], the XRD patterns were processed by the Rietveld refinement technique [9,10] assuming the possibility of the presence of four Al-Fe phases. The changes in the phase contents in the alloys with the milling time are shown in Fig. 2. Based on the analysis of the phase evolution, the alloying process can be described as follows. During the first 30 h of milling, there is a gradual decrease in the content of the fcc-Al phase. This phase can contain iron and is paramagnetic; therefore, it is referred to as fcc-Al(Fe)-para. It remains in the Al-Fe-Ti alloys milled for 20 h and in the Al-Fe-Ni and Al-Fe-Cu alloys milled for 30 h. There is also a gradual reduction in the quantity of the ferromagnetic bcc-Fe phase. This is predominantly bcc-Fe, but it may contain a small amount of Al in all the milled samples and is, hence, referred to as bcc-Fe(Al)-ferro.

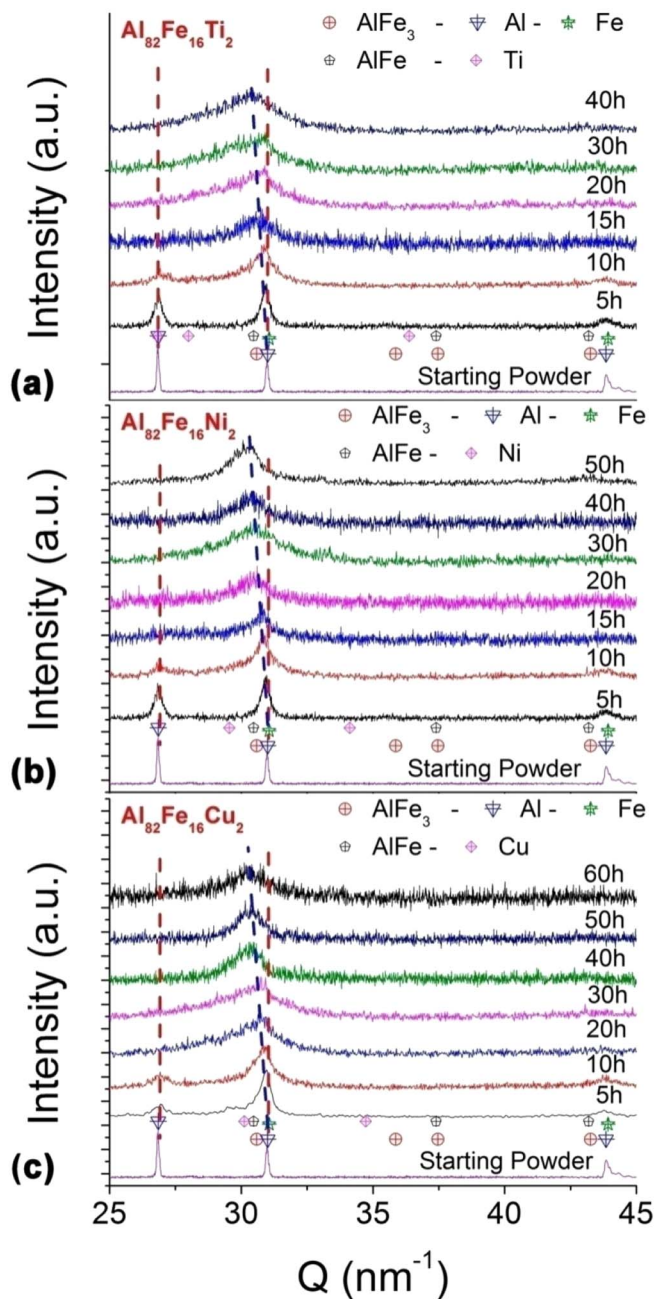


Fig. 1. XRD patterns of the powders milled for different milling times (intensity vs. scattering vector  $Q$ ): (a) Al–16 at.%Fe–2 at.%Ti. (b) Al–16 at.%Fe–2 at.%Ni. (c) Al–16 at.%Fe–2 at.%Cu.

The third phase, ferromagnetic fcc- $\text{AlFe}_3$  (DO3), starts forming at the early stages of milling. Starting from 10 h of milling, there is an increasing contribution from a paramagnetic bcc phase. This phase is identified as the AlFe phase and is, therefore, referred to as bcc-AlFe-para. Apparently, upon further milling, bcc-AlFe-para transforms into an amorphous phase. The evidence of the formation of an amorphous phase is an increase in the background level and a halo between 27 and 32  $\text{nm}^{-1}$  observed on the profiles. The  $\text{AlFe}_3$  (DO3)-ferro and bcc-AlFe-para phases were the phases remaining until complete amorphization of the  $\text{Al}_{82}\text{Fe}_{16}\text{Ti}_2$  and  $\text{Al}_{82}\text{Fe}_{16}\text{Ni}_2$  alloys. The bcc-AlFe-para was the only crystalline phase that was observed in the  $\text{Al}_{82}\text{Fe}_{16}\text{Cu}_2$  alloy after 50 h of milling.

Noteworthy is the influence of the added TM alloying elements. As the concentrations of these elements in the powder mixtures were lower than 5 wt%, the presence of these elements could not be detected by the

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