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## RESEARCHES ON OBTAINING SINTERED Al<sub>80</sub>Fe<sub>10</sub>Ti<sub>10</sub> FOAMS

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**Abstract:** Amorphous and nanocrystalline alloys based on Al-Fe have superior mechanical and corrosion properties than crystalline Al alloy. These qualities it recommends to be good candidate to obtain ultra-light alloys with special properties. Al<sub>80</sub>Fe<sub>10</sub>Ti<sub>10</sub> amorphous alloy (atomic%) was obtained from elemental powders of Al purity of 99.9% with a particle size <250 nm, Fe (NC100.24) and Ti (purity 99.5% and the particle size between 150-250 µm) by mechanical alloying in high energy planetary ball mill. Mechanical alloying was carried out in hardened steel vials and balls, in argon atmosphere, using as a process control agent (PCA) benzene (C<sub>6</sub>H<sub>6</sub>). The obtained powder was cold pressed in 6 mm diameter hardened steel die, at 500 MPa. The obtained raw compacts were sintered in induction, at 880, 1000 to 1200 °C temperature and 40 to 60 seconds holding time. The obtained powders and sintered compacts were characterized by X-ray diffraction, scanning electron microscopy (SEM) and particle size distribution of the powder was performed by laser analysis.

**Key words:** amorphous foams, powder metallurgy, intermetallic compound, induction sintering, mechanical alloying

### 1. INTRODUCTION

Metal foams are a class of materials widely studied due to their physical and mechanical properties, such as low density in absorbing large amounts of energy to deformation [1, 2]. Porous aluminum and its alloys are the most extensively studied due to low melting temperature and its availability. There are many processes for the production of aluminum metal foam: foaming by direct gas injection into the molten metal [3], foaming molten metal with blowing agents [4], powder compact foaming process [5], obtaining foam by space holder technique using different space holders [6, 7]. Fe-Al based materials are good candidates for applications in high temperature environments because of excellent corrosion resistance, good mechanical properties, low cost and availability of raw materials [8]. In addition to these properties, the amorphous materials with highly porous metal foam presents the characteristics mentioned above.

Mechanical alloying as an alternative to the classical methods of obtaining amorphous alloys by rapid solidification, amorphous alloys can be obtained at room temperature even in systems having a reduced capacity for amorphisation. The method can produce amorphous phases by milling amorphous metal tapes, milling pure elements or diverse compounds [9, 10].

Light amorphous alloys can be obtained by mechanical milling, M. Tavoosi et al., obtained different Al-Fe-X (X=Ti, Ni and V) [11] with high crystallization temperatures (~900 °C). These amorphous alloys might be suitable to obtain bulk amorphous samples. The studies in this paper we pursued the possibility of obtaining porous sintered Al-Fe-Ti amorphous foams, using amorphous Al<sub>80</sub>Fe<sub>10</sub>Ti<sub>10</sub> powders obtained by mechanical alloying.

## 2. EXPERIMENTAL

In order to obtain an  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  (atomic %) amorphous powder was used: aluminum powder (purity 99.9%, particle size  $<250\ \mu\text{m}$ ), iron powder (NC100.24) and titanium powder (purity 99.5% particle size fraction 150-250  $\mu\text{m}$ ). The powders were weighed in a stoichiometric ratios and was mechanically alloyed in a high-energy planetary mill. The balls/ powder weight ratio was 10: 1. Benzene ( $\sim 5\text{ml}$ ) was added as process control agent. The milling process was performed in an argon atmosphere to prevent oxidation of the powder. At certain time powder samples were taken in the glow - box in an argon atmosphere.

The particle size distribution of the powder was measured with a laser particle analyzer Fritsch Analysette 22 - NANOTEC.

Sintered samples were investigated by X-ray diffraction using  $K\alpha$  radiation  $\text{Co}$  ( $\lambda_1 = 1.788965\ \text{\AA}$ ).

Powder compacting was performed by pressing in a hardened steel die with 6 mm diameter at 500 MPa. The powder sintering was performed in induction, in a graphite crucible, at 880, 1000 and 1200  $^{\circ}\text{C}$  temperature, with 40 and 60seconds hold times. The small particle size would require much higher frequencies than those currently available on induction.

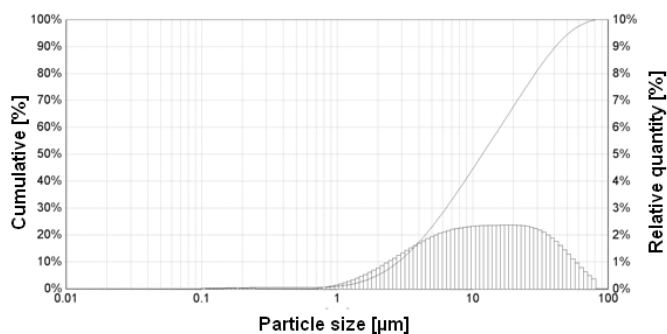
The powder and sintered samples structure was done by scanning electron microscope JEOL JSM 5600 LV.

Sample density was calculated by dividing weight to the volume calculated from the samples dimensions.

Compression test of samples was performed on Instron 250 kN type a universal testing machine with a crosshead velocity of 1 mm/min.

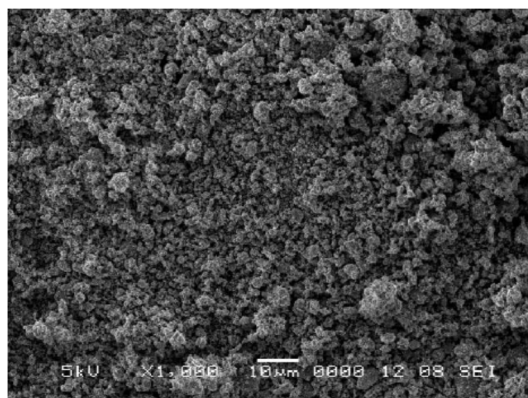
## 3. RESULTS AND DISCUSSIONS

The particle size distribution of  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  powder after 20 hours of mechanical alloying is shown in Figure 1. The size of the particles is between 0.9 and approx. 100  $\mu\text{m}$ . The X-ray diffraction revealed that amorphous powder was obtained after 20 hours of mechanical alloying.

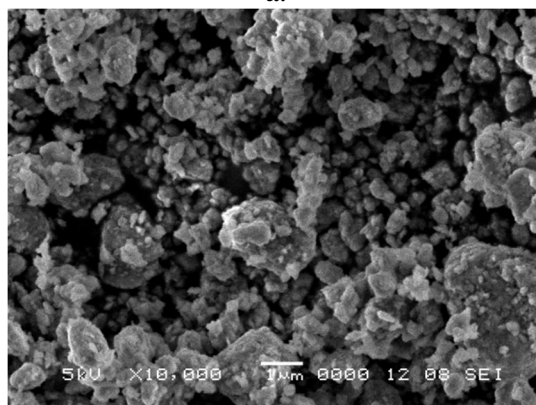


**Fig. 1.** Particle size distribution of  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  powder mixture at 20 h

As can be seen from the images presented in Figure 2 the milled powder consist of agglomerations of small ( $<1\ \mu\text{m}$ ), polyhedral shaped particles. These agglomerations could not be separated during the ultrasonic treatment process prior to the particle size analysis.



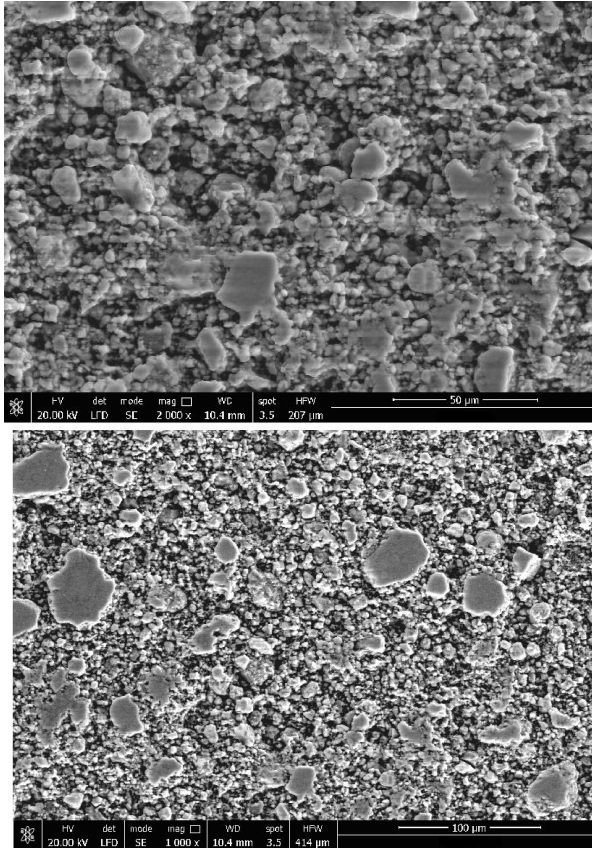
a.



b.

**Fig. 2.** Scanning electron microscopy images of the 20 h milled powder a). x1000, b). x 10000

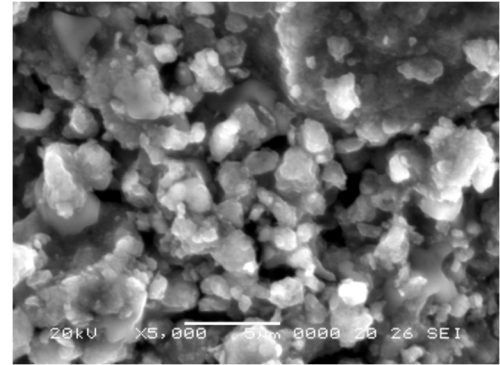
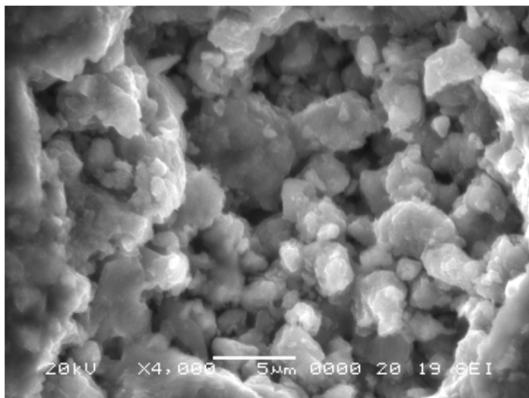
Sintering of amorphous  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  green compacts was performed in induction at different temperatures and hold times, to obtaining metal foams



**Fig. 3** SEM images of  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  samples sintered at  $880\text{ }^{\circ}\text{C}$

In Figure 3 shows SEM images of the sintered samples at  $880\text{ }^{\circ}\text{C}$ . At this sintering temperature for up to 5 minutes, the powders are in an early stage of sintering. Because of this, compacts were sintered at higher temperatures of  $1000$  and  $1200\text{ }^{\circ}\text{C}$  with 40 to 60 seconds holding times.

At higher sintering temperature formation of sintering necks between powder particles and presence of small pore (less than  $5\mu\text{m}$ ) in the material structure (Fig. 4) can be observed.



**Fig. 4.** SEM images of the  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  samples sintered in the induction at temperature: a)  $1200\text{ }^{\circ}\text{C}$ , 40 seconds hold time; b)  $1200\text{ }^{\circ}\text{C}$ , 60 seconds hold time.

The XRD patterns of  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  samples sintered in the induction at  $1100\text{ }^{\circ}\text{C}$  for 60 seconds and  $1200\text{ }^{\circ}\text{C}$  at 40 hold times and the starting  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  amorphous powder obtained by wet mechanically alloyed are presented in Figure 5. Short sintering times have not ensured the formation of an amorphous metallic foam, due to the fact that the sintering temperature is high (between  $1000$  and  $1200\text{ }^{\circ}\text{C}$ ) and exceeds the first crystallization temperature of the amorphous powder, which according to DSC analysis is about  $360\text{ }^{\circ}\text{C}$  [12].

After crystallization of the  $\text{Al}_{80}\text{Fe}_{10}\text{Ti}_{10}$  amorphous powder during induction sintering, intermetallic compounds resulted:  $\text{Al}_{13}\text{Fe}_4$ ,  $\text{AlFe}$ ,  $\text{AlFe}_3$ ,  $\text{Al}_3\text{Ti}$ , aluminum and titanium carbide ( $\text{Al}_4\text{C}_3$ ,  $\text{TiC}$ ). We can say that the induction sintering of amorphous powder samples yielded intermetallic compound based foams.

The presence of carbides in the material structure is due to benzene used as a process control agent. It was reported that the use of process control agents of mechanical alloying, including benzene ( $\text{C}_6\text{H}_6$ ) produce the powders contamination with carbon. During sintering at elevated temperatures carbide formation is favored [13].

The value of the relative density determined by calculation, for the sintered samples at  $1000\text{ }^{\circ}\text{C}$  with holding time of 60 seconds, is about 0,7.

According to the classification behavior of metallic foams compressive made by R. Florek et al. in [14], our metallic foams behaves in compression as a very brittle foam (Fig. 6).

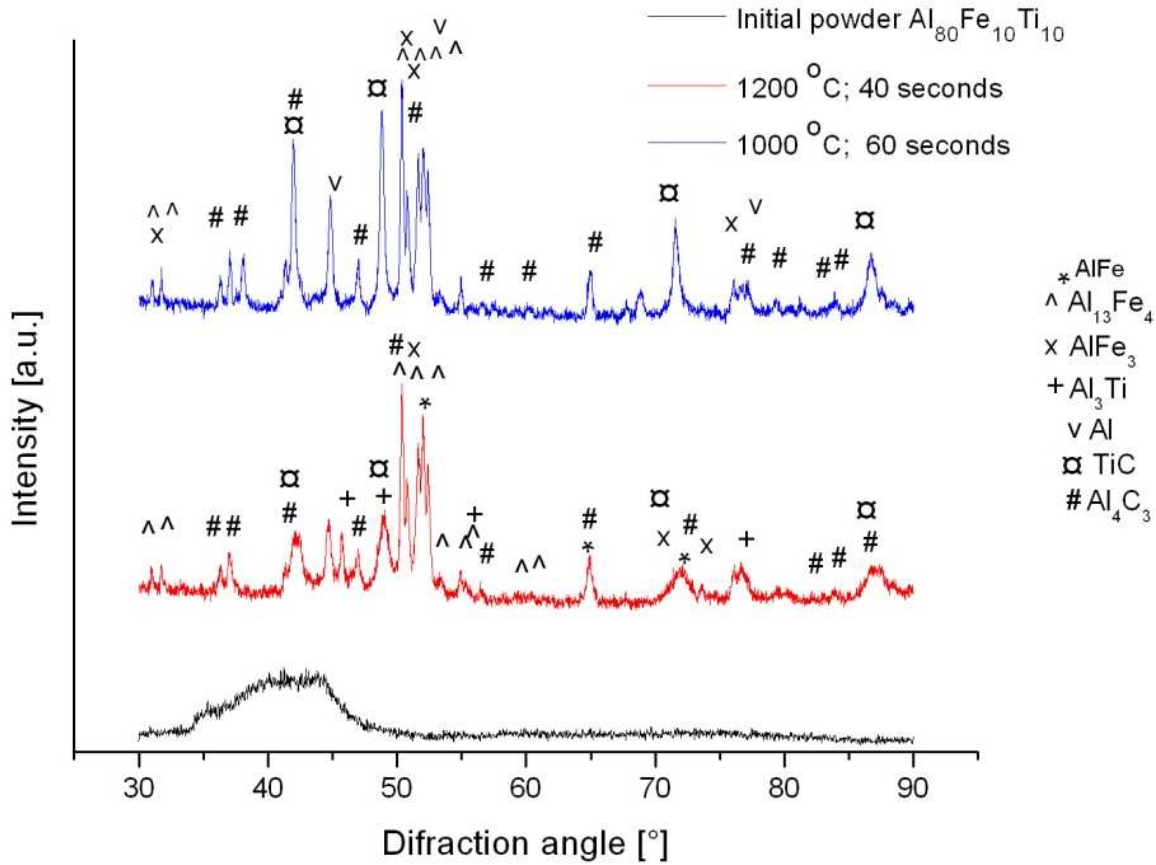


Fig. 5 XRD pattern of amorphous powder and sintered samples in induction

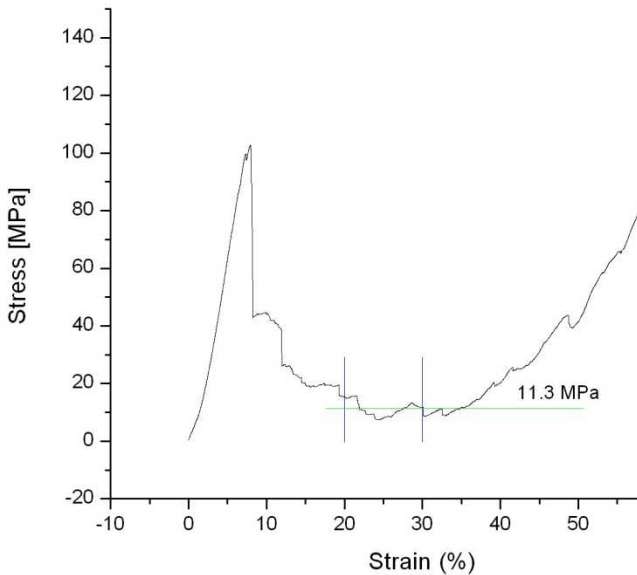


Fig. 6 Compressive stress-strain diagram for induction sintered samples at 1000 °C with 60 seconds hold time

The first maximum compressive strength has a value of 103 MPa. The strain range from 8,2 to 11,9 % compressive stress decrease from 43 to 39 MPa, and 11.9% strain, the compressive stress drops to around 26MPa. The plateau stress is about 11,3MPa. Foam densification occurs at

about 40% strain. Compression testing revealed that the foams have a low deformability, which is characteristic of intermetallic compounds.

#### 4. CONCLUSIONS

Al<sub>80</sub>Fe<sub>10</sub>Ti<sub>10</sub> amorphous powders compacted at 500 MPa, induction sintered at 880 °C provides a low sintering because the powder particles are not welded together.

Induction sintering of the amorphous powders pressed compacts (500 MPa), at sintering temperature 1100 to 1200 °C, with hold times of 40 and 60 seconds lead to obtaining the basis intermetallic compounds porous structure with about 0.7 relative density.

Compressive behavior of metal foams made from Al<sub>80</sub>Fe<sub>10</sub>Ti<sub>10</sub> amorphous powder by induction sintering process is fragile metallic foams feature. The first maximum compressive strength has a value of 103MPa.

The obtained foams have a low deformability characteristic to the intermetallic compounds.

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**CERCETĂRI ASUPRA OBTINERII SPUMELOR SINTERIZATE DIN ALIAJUL  $Al_{80}Fe_{10}Ti_{10}$** 

**Rezumat:** Aliajele amorfe și nanocristaline poroase pe bază de Al-Fe prezintă proprietăți mecanice și de coroziune superioare aliajelor cristaline poroase de Aluminiu. Aceste calități ale lor le recomandă a fi bune candidate pentru obținerea de aliaje ultra ușoare cu proprietăți speciale. Aliajul amorf  $Al_{80}Fe_{10}Ti_{10}$  (% atomice) a fost obținut din pulberi elementale de Al puritate de 99,9 %, cu dimensiunea particulelor < 250  $\mu m$ , pulbere de Fe (de tipul NC100.24) și Ti (puritate 99,5 % și dimensiunea particulelor cuprinsă între 150 – 250  $\mu m$ ) prin aliere mecanică în moară planetară de înaltă energie. Alierea mecanică s-a realizat în containere cu bile din oțel călit, în atmosferă de argon, utilizând ca agent de control al procesului benzene ( $C_6H_6$ ). Pulberea obținută a fost compactizată la rece, uniaxial – bilateral, în matriță de oțel călit cu diametrul de 6 mm, la o presiune de compactizare de 500 MPa. Compactele crude obținute au fost sinterizate prin inducție la temperaturi de 880, 1000 și 1200 °C, cu durata de menținere de 40 și 60 secunde. Pulberile și compactele sinterizate obținute s-au caracterizat prin difracție de raze X, microscopie electronică de baleiaj (SEM), iar distribuția dimensiunii particulelor de pulbere s-a realizat prin analiză cu laser.

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