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# Nanocrystalline Al-Fe intermetallics - light weight alloys with high hardness

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## 1. Introduction

Iron aluminides can be of considerable technological interest due to their advantageous properties, in particular a high specific strength, high specific stiffness, good strength at intermediate temperatures and excellent corrosion resistance at elevated temperatures [1]. Al-rich iron aluminides are characterised by low density, but also by lower strength and hardness than Fe<sub>3</sub>Al or FeAl ones. However, strength and hardness can be improved by grain size refinement, especially to nanometric scale. Nanocrystalline materials exhibit enhanced properties, such as high strength and hardness, compared to the materials with conventional grain size [2–4]. Having their strength increased, Al-based alloys will possess high specific strength.

Nanocrystalline materials are extensively produced by the mechanical alloying (MA) process, but in the form of a powder [5], hence consolidation is a necessary step for milled powders to have possible practical applications. Compaction of nanocrystalline powders into bulk, full-density material providing nanocrystalline structure's maintenance is difficult, since application of high temperature, which is required for good consolidation of powders, i.e. to remove all porosity and to obtain good interparticle bonding, can cause grain growth. To overcome the problem of nanoscale microstructure coarsening, the employment of a high pressure during consolidation and limiting of the high temperature

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#### ABSTRACT

Nanocrystalline Al–Fe alloys containing 60–85 at.% Al were produced by consolidation of mechanically alloyed nanocrystalline or amorphous (Al85Fe15 composition) powders at 1000 °C under a pressure of 7.7 GPa. The hardness of the alloys varied between 5.8 and 9.5 GPa, depending on the Al content. The specific strength, calculated using an approximation of the yield strength according to the Tabor relation, was between 544 and 714 kNm/kg. Based on the results obtained, we infer that application of high pressure affected crystallisation of amorphous Al<sub>85</sub>Fe<sub>15</sub> alloy, influencing the phase composition of the crystallisation product, and phase changes in nanocrystalline Al<sub>80</sub>Fe<sub>20</sub> alloy, inhibiting them.

exposure time can be utilised. High pressure hot-pressing method has been successfully used for producing bulk nanocrystalline samples [4,6–9]. Recently, we have demonstrated that application of a high pressure influences grain growth at elevated temperature by hindering it [4,6,8]. Considering the grain growth as a diffusional process, this hindering can be explained by the fact that the diffusion coefficient decreases with pressure [10]. Hence, high pressure can reduce grain boundary mobility. We have also shown that the nanocrystalline FeAl intermetallic as well as FeAl–TiC nanocomposites produced by MA followed by high pressure hotpressing consolidation possess relatively high hardness (namely, 1235HV0.2 (12.12 GPa) and 1608HV0.2 (15.77 GPa) for FeAl and FeAl–TiC respectively) in comparison with their microcrystalline counterparts [6,4].

Aluminium-rich Al–Fe powder alloys have been prepared using the MA process [11–13]. However, works devoted to consolidation of these powders are very scarce [14,15].

In the current work, we obtained bulk nanocrystalline Al–Fe alloys containing 60–85 at.% Al by consolidation of mechanically alloyed nanocrystalline or amorphous powders. The structural and phase transformations taking place during consolidation were studied and the produced compacts were characterised.

## 2. Experimental

 $Al_x$ -Fe<sub>100-x</sub> (x = 60, 65, 70, 75, 80, and 85) powder alloys (all compositions are given in at.% throughout this paper) were synthesized by mechanical alloying in a SPEX 8000 D ball mill. The details of this experimental step can be found in Ref. [13].





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A press equipped with a toroid-type high pressure cell was used for consolidation of the milled powders. The shape of the cell and the material of the gasket ensured that the compacting conditions were close to isostatic ones. The compaction process was performed under a pressure of 7.7 GPa at a temperature of 1000 °C for 3 min. The loading, at a rate 0.5 GPa/min, was done prior to the heating. The heating and cooling rate was 1000 °C/min.

The structural investigations of consolidated samples were carried out by X-ray diffraction (XRD) method using a Philips 1830 diffractometer using CuK<sub>α</sub> radiation. The lattice parameter and the mean crystallite size, the latter determined by the Williamson-Hall method, were calculated from the XRD data taking into account CuK<sub>α1</sub> radiation, after K<sub>α2</sub> stripping using the Rachinger method. The instrumental broadening was determined using an Si standard and subtracted from the experimental breadth to obtain a "physical" broadening of each diffraction line, which was then used for the Williamson-Hall calculations.

In order to check the quality of consolidation, a Nikon Epiphot 200 light microscope was used for observations of the produced materials' surface. Samples for light metallography were prepared using standard polishing technique.

The Vickers microhardness and hardness of the compacts were measured under a load of 200 g and 1 kg respectively, imposed for 15 s. Vickers microhardness value was the average of at least 25 indentations. The density of the bulk samples was determined using a Gibertini E154 balance equipped with a device for measuring the density of solids (Archimedes method). Basing on mass measurements performed during density determination, open porosity of the consolidated samples was calculated.

#### 3. Results and discussion

The phase and structural evolution occurring in the Al–Fe powders during mechanical alloying and the characterisation,

including study of thermal behaviour, of the milling products prepared for consolidation have already been described and analysed in detail [13]. It has been shown that three kinds of structure were produced: (i) nanocrystalline supersaturated Fe(Al) solid solution for the  $Al_{60}Fe_{40}$   $Al_{65}Fe_{35}$  alloys, (ii) nanocrystalline  $Al_5Fe_{20}$  intermetallic, at least partially ordered, for the  $Al_{75}Fe_{25}$  and  $Al_{80}Fe_{20}$  alloys, (iii) amorphous for the  $Al_{85}Fe_{15}$  alloy. In the case of  $Al_{70}Fe_{30}$  composition, a two-phase  $Al_5Fe_2 + Fe(Al)$  alloy was obtained [13].

Fig. 1 shows the XRD patterns of the milled powders before and after consolidation. Comparing the spectra of bulk samples with those of the powders before consolidation, one can see that in all the alloys, except the Al<sub>75</sub>Fe<sub>25</sub> one, hot-pressing caused phase changes. For the alloys containing 60, 65 and 70% of Al, the (100) and (111) superlattice reflections of the ordered B2 structure appear, which evidences the ordering of the Fe(Al) solid solution and its transformation into an FeAl intermetallic compound. In the case of the Al<sub>65</sub>Fe<sub>35</sub> alloy, besides the ordering, precipitation of an Al<sub>5</sub>Fe<sub>2</sub> intermetallic from the supersaturated Fe(Al) solid solution is evident from the XRD pattern. The lattice parameter of the FeAl phase decreased in comparison with the one of the solid solution before consolidation and is equal to 2.911 Å and 2.908 Å for Al<sub>60</sub>Fe<sub>40</sub> and Al<sub>65</sub>Fe<sub>35</sub> respectively. The same phase changes were noticed during heating of the as-milled alloys containing 60, 65 and 70% of Al in the calorimeter [13]. For the Al<sub>80</sub>Fe<sub>20</sub> alloy, appearance of diffraction lines, which are attributed to an Al<sub>13</sub>Fe<sub>4</sub> phase, besides those of Al<sub>5</sub>Fe<sub>2</sub> intermetallic is evident. In the case of the Al<sub>85</sub>Fe<sub>15</sub> alloy instead of the diffraction halo, peaks are present, which indicates that the amorphous phase crystallised during consolidation. However, these diffraction peaks cannot be assigned to any phase in Al-Fe system [16] nor to any Al-Fe phase in the ICDD PDF4 database, hence products of the crystallisation under high pressure of the amorphous alloy are metastable phases. Some of the peaks can be indexed in cubic system and attributed to a phase with bcc structure and unit cell parameter of 2.964 Å. It is worthwhile to

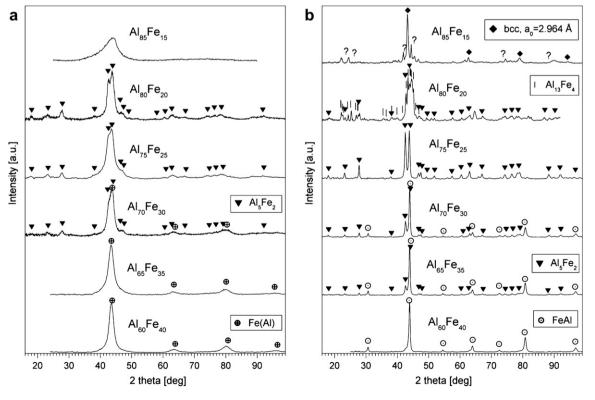


Fig. 1. XRD patterns of the Al-Fe alloys: (a) before consolidation, (b) after consolidation.

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