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Processing of nanocrystalline FeAlX (X = Ni, Mn) intermetallics using a mechanical alloying and hot pressing techniques

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Fe-40Al-40Ni-20 and Fe-40Al-40Mn-20 (all in at.%) intermetallics were mechanically alloyed for 40 h and followed by hot-pressing at 650°C under 450 MPa for 1 h. As resulted from the X-ray diffraction studies, the ordered B2 structure was formed in the Fe-40Al-40Ni-20 alloy while in the case of Fe-40Al-40Mn-20 alloy, the disordered Fe(Al) solid solution was observed. The chemically homogenous rounded particles of size of about 5 μm were identified using scanning analytical electron microscopy in alloys after 40 h of milling. TEM studies of milled powders revealed a nanostructure in both alloys with grain size of about 20 nm.

The hot pressing process of milled powders allowed to obtain compacts with the density of about 87 and 89% of the theoretical one for Fe-40Al-40Mn-20 and Fe-40Al-40Ni-20 alloys, respectively. The micro-hardness measurements have shown that the alloy with the Ni addition possesses the hardness of about 1200 HV_{20}, whereas in the alloy with the Mn addition it is 1100 HV_{20}. The TEM investigations allowed to identify a nanocrystalline structure of compacts with a mean grain size below 50 nm, with B2 ordered structure in both alloys.

1. Introduction

Ordered FeAl intermetallic alloys are attractive materials for medium and high temperature industrial applications but their use has been restricted due to room temperature brittleness and their poor creep resistance [1]. Significant improvement in these properties can be achieved by two approaches: alloying and process control. The first one is based on the fine control of grain boundary cohesion by using microalloying methods; second one introduces processes which reduce the crystallite size down to nanometer range. A considerable number of experimental observations have confirmed that nanocrystalline materials of the Fe-Al system have improved properties with respect to conventional materials [2–5].

Mechanical alloying (MA) is useful process for production such nanostructured materials on an industrial scale. The MA process of the Fe-Al system results usually in the formation of Fe(Al) nanocrystalline or amorphous phases [6], however high temperatures are required for high density consolidation of milled powders. One of biggest challenges of consolidation procedures is to retain a fine grain size by limiting the time and temperature required for consolidation.

The aim of this work was to produce nanocrystalline powder by MA of Fe-Al-Ni and Fe-Al-Mn powder mixtures and to obtain nanostructure compacts by hot-pressing.

2. Experimental procedure

The pure elemental powdered mixtures with the composition of Fe-40Al-40Ni-20 and Fe-40Al-40Mn-20 (all in at.%) were milled in a high-energy planetary mill Fritsch Pulverisette P/4 rotating at 200 rpm under argon atmosphere. The X-ray analysis (using Mo K_α radiation) was performed after 20 and 40 h of milling using Phillips PW 1840 diffractometer. The scanning electron microscope Phillips XL30 equipped with the energy depressive spectroscopy and the transmission electron microscope Phillips CM20 were used for characterization of microstructure and chemical composition of both milled and pressed powders.

The consolidation of milled powders by uni-axial hot pressing was performed at 650°C under 450 MPa for

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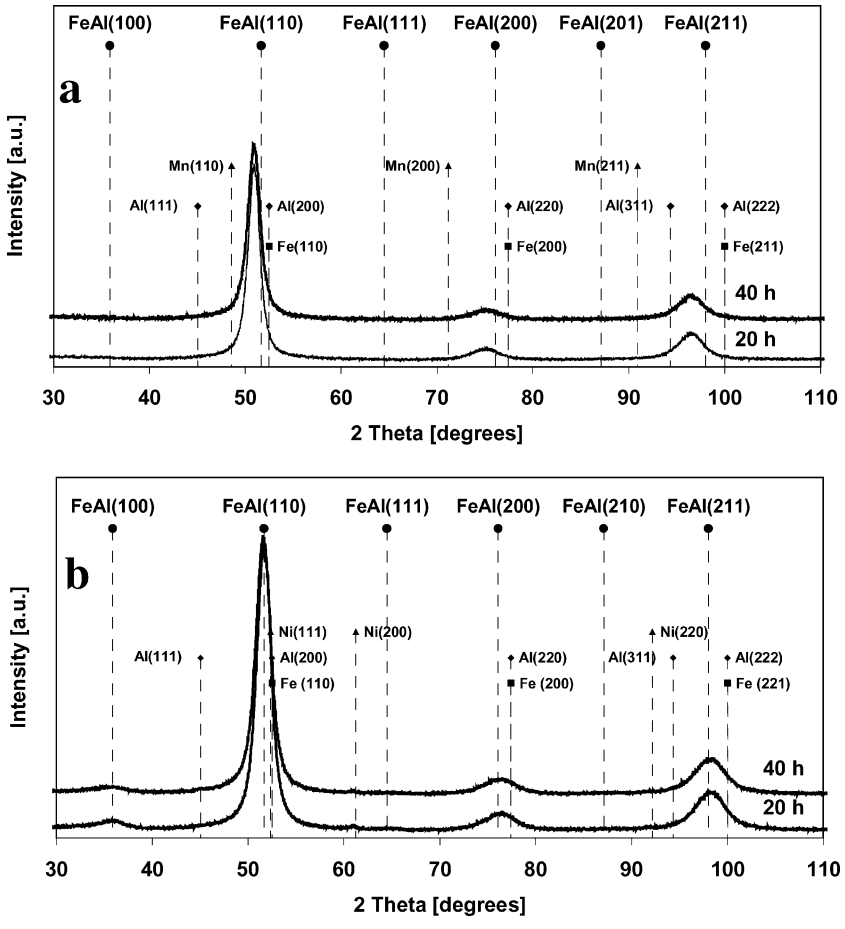

Figure 1 The X-ray diffraction patterns of (a) Fe-40Al-20Mn and (b) Fe-40Al-20Ni alloys prepared by MA (milling times: 20 and 40 h).

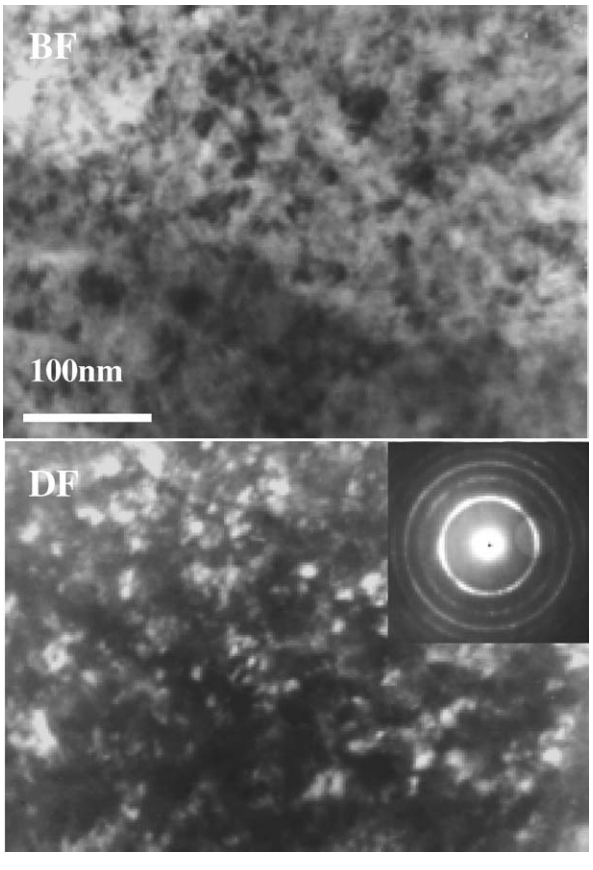

Figure 2 The TEM bright and dark field micrographs and corresponding selected area diffraction pattern of Fe-40Al-20Mn alloy after 40 h of milling.

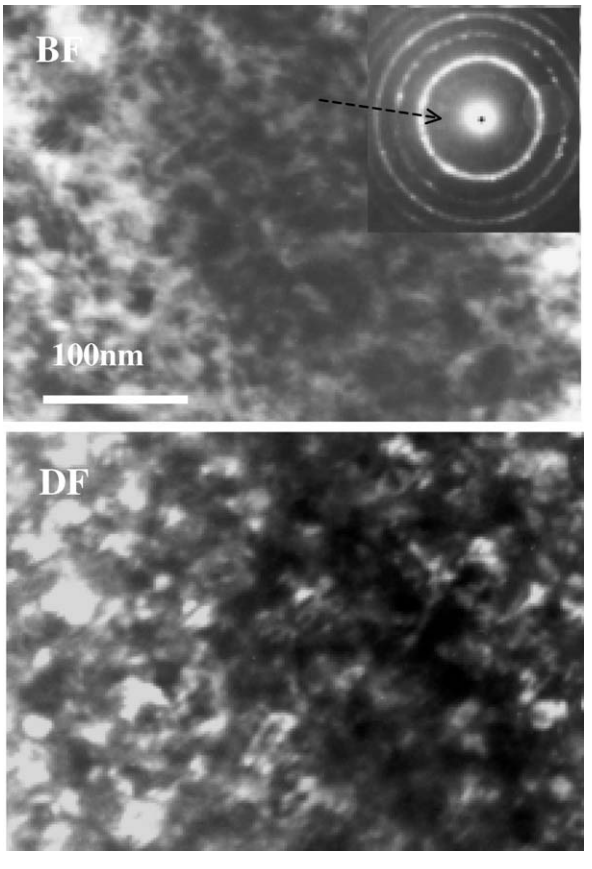

Figure 3 The TEM bright and dark field micrographs and corresponding selected area diffraction pattern of Fe-40Al-20Ni alloy after 40 h of milling.

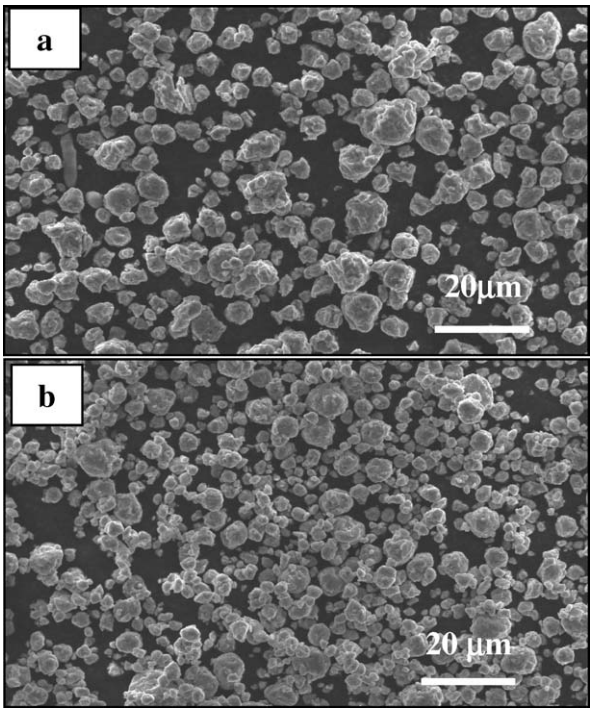

Figure 4 The SEM microstructure of (a) Fe-40Al-20Mn and (b) Fe-40Al-20Ni alloys after 40 h of milling.

1 h in flow argon. The preliminary characterization of the quality of pressed samples was performed by the micro-hardness test with the load of 20 g and determination of density. The thin foils of pressed sample for TEM observations were prepared by dimpling and ion milling using Gatan equipment whereas milled powders were embedded in resin and thin slices were cut by microtome.

3. Results and discussion

Fig. 1a and b present sets of diffraction patterns of milled powders (after 20 and 40 h) for two investigated alloys. The formation of nanocrystalline ordered B2-FeAl phase was observed for the alloy with the manganese addition and of Fe(Al,Ni) solid solution for the alloy with nickel addition (after 20 h of milling). The prolongation twice a milling time up to 40 h, did not change the structure, but only the refinement of crystallite size was observed. This suggests that milling process at above-mentioned conditions does not lead to the amorphization of the structure. The X-ray diffraction pattern has shown that in the case of alloy with Ni addition, the diffraction peak occurs at 2Θ position between 30 and 40 degrees, which corresponds to {100} plane of ordered B2-FeAl phase. This peak disappears in the case of alloy with Mn addition, but the shift the peak maximum toward the lower 2Θ angles is visible. This indicates that β-FeAl phase is disordered and its lattice parameter increases.

The TEM observations of milled powders for 40 h (Figs 2 and 3) allowed to identify the nanostructure with the mean grain size of about 20 nm for both alloys and confirmed results of X-ray diffraction measurements. Comparing the selected area diffraction patterns for two investigated alloys, the additional weak diffraction ring (indicated by arrow in Fig. 3) corresponding to (100) FeAl superlattice plane was identified for alloy with nickel addition. The point chemical analyses

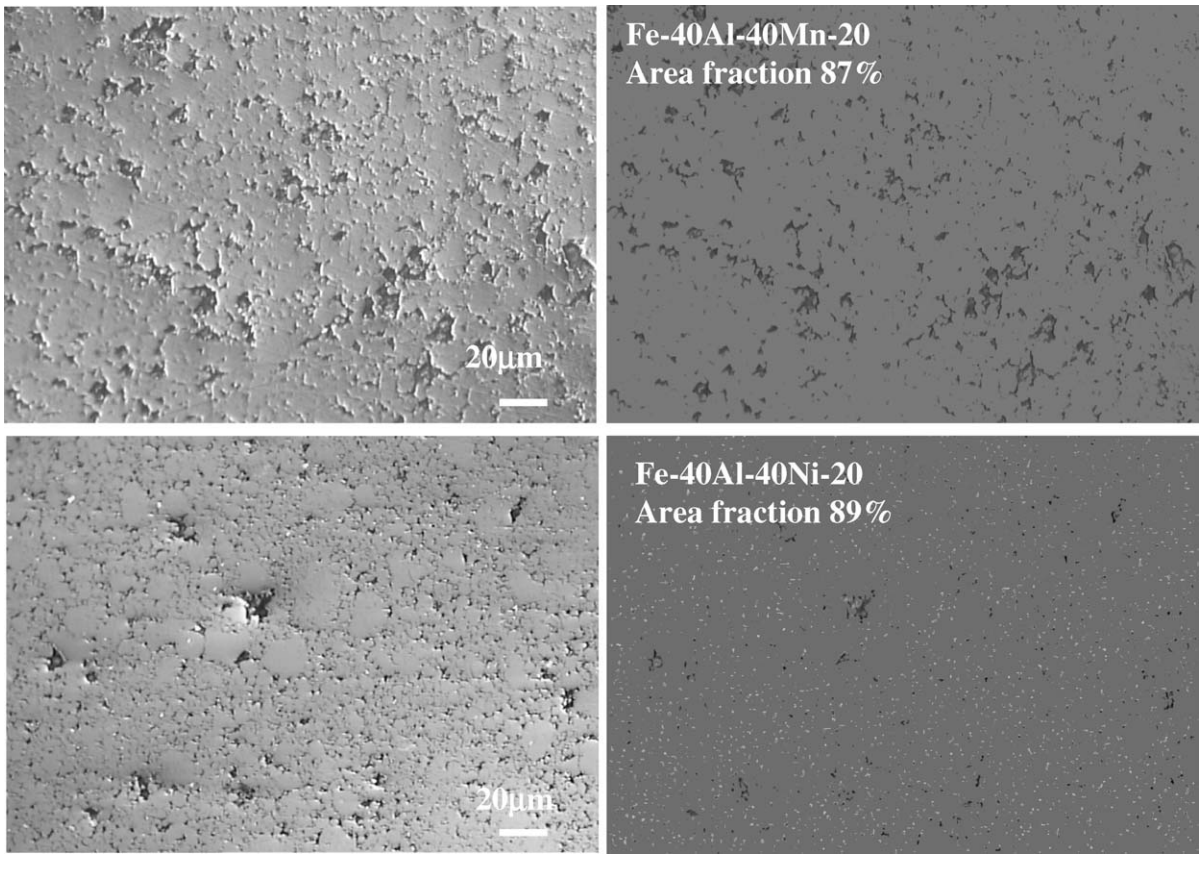

Figure 5 Optical microstructure of Fe-40Al-20Mn (top) and of Fe-40Al-20Ni (bottom) alloys after hot pressing and corresponding binary images.

performed using TEM within milled powders revealed that for alloy with manganese addition, the mean composition is as follows: Fe-44.4, Al-35.3, Mn-20.3 (in at.%), whereas for alloy with nickel addition it was: Fe-51, Al-24.5, Ni-24.5, Ni-24.5. The differences in amount of dissolved elements within Fe(Al) solid solution during MA may influence the ordering of the solution.

The SEM microstructures of 40 h milled powders are presented in Fig. 4. The homogenous rounded particles of size of about 5 μm were observed. In the case of the alloy with the nickel addition, the particles have tendency to agglomeration. The measurements of the chemical composition performed on at least ten particles have shown a high degree homogeneity in both alloys. The composition of single particles was almost identical to that of the initial mixture.

The hot pressing technique performed at 650°C under 450 MPa for 1 h under argon atmosphere allowed to obtain compacts with the density of about 87 and 89% of the theoretical one for Fe-40Al-40Mn-20 and Fe-40Al-40Ni-20 alloys, respectively. Fig. 5 presents the optical microstructures and corresponding binary images of hot pressed samples. The binary transforms were used for separation of pores in compacts and for the calculation of the area fraction of bulk phases. One can see that in the case of alloy with manganese addition, the pores are larger and more inhomogeneously distributed while in the sample with nickel addition, there is a large amount of very small pores (bellow 1 μm) and only a small amount of bigger ones. The character and distribution of pores influences the densification of samples. It was shown that area fraction of bulk phases is higher in compacts with the manganese addition than that in compacts with nickel addition.

Fig. 6 presents the X-ray diffraction patterns of Fe-40Al-40Mn-20 and Fe-40Al-40Ni-20 alloys after hot pressing at 650°C under 450 MPa for 1 h. One can see that for both alloys, a nanocrystalline fully ordered B2-FeAl structure was formed. The measured crystallite size was 20 and 30 nm for Fe-40Al-40Mn-20 and Fe-40Al-40Ni-20 alloys, respectively. The transmission electron microscopy investigations (Fig. 7) allowed to identify a nanocrystalline structure of compacts with a mean grain size below 50 nm. The ordered structure was identified in both pressed samples. Additionally, the hot pressed samples with the manganese addition possess finer grain size (below 30 nm).

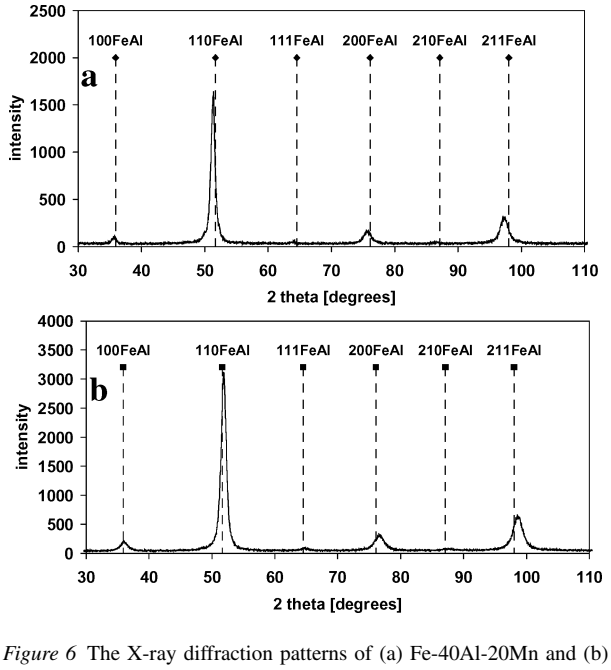

Figure 6 The X-ray diffraction patterns of (a) Fe-40Al-20Mn and (b) Fe-40Al-20Ni alloys after hot pressing at 650°C under 450 MPa.

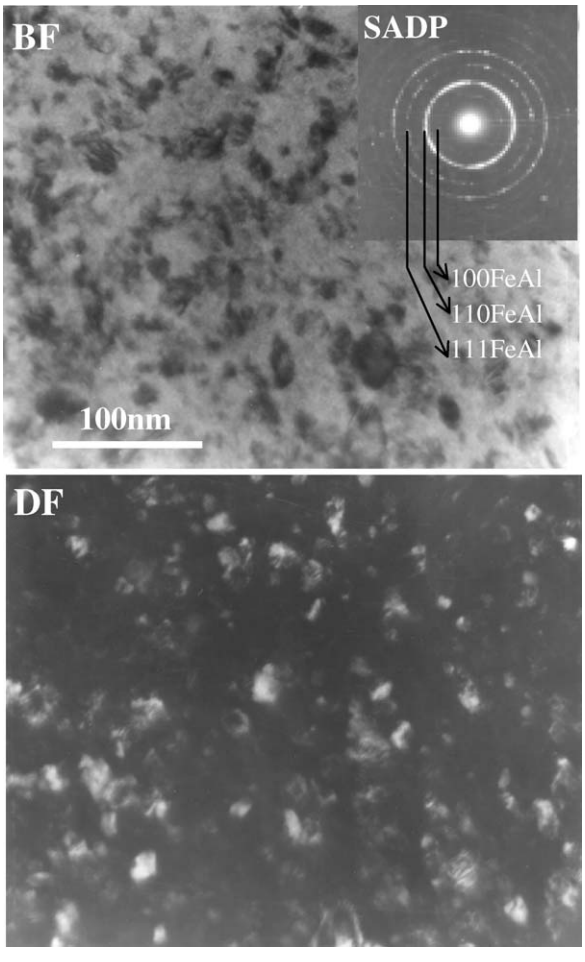

Figure 7 The bright field transmission electron micrograph and corresponding dark field image and selected area diffraction pattern of hot pressed Fe-40Al-20Mn alloy.

The micro-hardness measurements have shown that the alloy with the Ni addition possesses the hardness of about 1200 HV_{20}, whereas the hardness of the alloy with the Mn addition is 1100 HV_{20}. The higher value of the micro-hardness (about 1400 HV_{20}) was reported by Krasnowski *et al.* [7] for nanocomposite FeAl-TiN produced by reactive milling and hot pressing under 8 GPa.

4. Conclusions

MA process (for 40 h) leads to the formation of ordered B2 structure in Fe-40Al-40Ni-20 alloy, and to the formation of the disordered Fe(Al) solid solution in Fe-40Al-40Mn-20 alloy. The differences in structure after milling are connected with amount of dissolved elements in Fe(Al) solid solution. For both alloys, chemically homogenous rounded particles (with the size of

about 5 μm) were formed after 40 h of milling. The hot pressing process of milled powders allowed to obtain compacts with density of about 87 and 89% of the theoretical one for Fe-40Al-40Mn-20 and Fe-40Al-40Ni-20 alloys, respectively, and with micro-hardness of about 1200 HV_{20}. The compacts posses a fully ordered B2-FeAl structure with mean grain size below 50 nm.

Acknowledgements

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