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AN INVESTIGATION OF AMORPHIZATION IN Fe-Al ALLOYS DURING MECHANICAL ALLOYING

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ABSTRACT: *In order to determine the amorphization time, Fe and Al elemental powder mixtures with composition of $Fe_{10.8}Al_{89.2}$, $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ were mechanically alloyed in a Spex 8000D Mixer/Mill for up to 96 h. The characterization of Fe-Al powder mixtures was studied as a function of mechanical alloying time by X-ray diffractometry and scanning electron microscopy. A fully amorphous phase was not observed by X-ray diffraction for $Fe_{10.8}Al_{89.2}$ and $Fe_{80}Al_{20}$ even after 96 h mechanical alloying. However, X-ray diffraction observation showed that 50 h mechanical alloying of $Fe_{50}Al_{50}$ alloy resulted in a fully amorphous phase formation.*

KEYWORDS: *Mechanical alloying, amorphous alloys, Fe-Al.*

Fe-Al ALAŞIMININ MEKANİK ALAŞIMLANDIRILMASINDA AMORFLAŞMANIN İNCELENMESİ

ÖZET: *Amorflaşma süresinin belirlenmesi amacıyla, $Fe_{10.8}Al_{89.}$, $Fe_{50}Al_{50}$ ve $Fe_{80}Al_{20}$ bileşimlerinde Fe ve Al tozları Spex 8000 D öğütücü kullanılarak mekanik olarak 96 saate kadar alaşımlandırılmıştır. Mekanik alaşımlama süresine bağlı olarak Fe-Al toz karışımlarının karakterizasyonu X-ışını difraksiyonu ve taramalı elektron mikroskobu ile gerçekleştirilmiştir. X-ışını difraksiyonunda, $Fe_{10.8}Al_{89.2}$ ve $Fe_{80}Al_{20}$ alaşımının 96 saat mekanik alaşımlandırılmasında bile tam amorf yapı gözlenememiştir. Diğer yandan, $Fe_{50}Al_{50}$ alaşımının 50 saat mekanik alaşımlandırılmasında tam amorf yapı oluşumu X-ışını difraksiyon incelemeleri ile görülmektedir.*

ANAHTAR KELİMELER: *Mekanik alaşımlandırma, amorf alaşımlar, Fe-Al.*

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I.INTRODUCTION

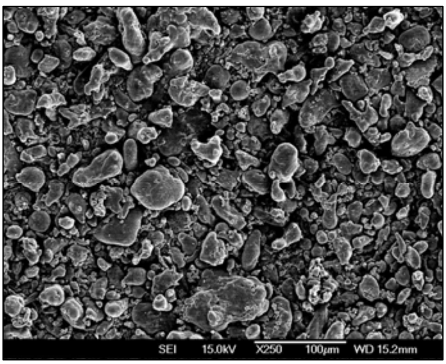
Mechanical alloying, first developed by Benjamin and co-workers in 1966 to produce oxide dispersion-strengthened alloys [1-3], is a solid-state powder processing technique that allows to synthesize materials including oxide dispersion-strengthened alloys [4], extended solid-solutions, amorphous and/or nanocrystalline alloys, intermetallics and composites [5-8]. Iron aluminide alloys are very attractive structural materials because of their superior mechanical properties, good corrosion and oxidation resistance and relatively low cost. A number of studies have been reported on where mechanical alloying has been successfully applied to produce Fe-Al alloys with various types of structures including nanocrystalline, amorphous and intermetallic [9-20]. It is well known that formation of these structures strongly depends on process conditions such as type of mechanical alloying device, mechanical alloying time and temperature, ball-to-powder weight ratio, *etc*. Due to these reasons, a knowledge on the mechanical alloying of Fe-Al systems need improvement. With the aim of the extends the investigations reported early, the structural transformations induced by mechanical alloying of $Fe_{10.8}Al_{89.2}$, $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ alloys were investigated in this work. In order to determine the effect of mechanical alloying time on formation of different phases, results are compared with previous report on similar compositions of mechanically alloyed Fe-Al system.

II. EXPERIMENTAL METHODS

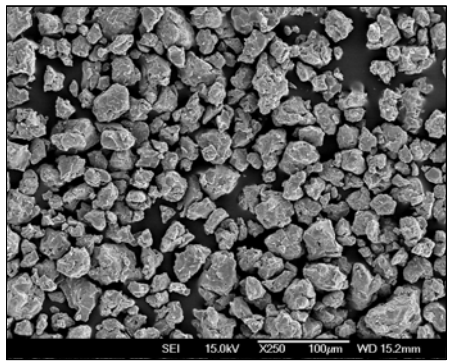
Alloys of the compositions $Fe_{10.8}Al_{89.2}$, $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ were prepared by mechanical alloying of Al powder (purity 99.97%, average particle size <8 μm) and Fe powder (purity 99.9%, average particle size <10 μm) from Alfa Aesar. The mechanical alloying was carried out in a Spex 8000 D mixer/mill. The ball to powder weight ratio was 10:1. The vial and the balls (2x½ inch and 4x¼ inch diameter) were of hardened steels. All samples were handled in a glove box filled with purified argon to minimize oxidation. The duration of the mechanical alloying was varied from 24 to 96 hours. Structure properties of the mechanically alloyed were studied by X-ray diffraction (XRD) in a Shimadzu XRD-600 diffractometer with Cu Kα radiation. The morphology of the alloys was observed by Jeol JSM 6335f Scanning Electron Microscopy.

III. RESULTS AND DISCUSSION

The evolution of the $Fe_{10.8}Al_{89.2}$ powder mixtures particle morphology as a function of the mechanical alloying time is shown in the SEM micrographs displayed in the Fig.1. As seen in Fig. 1(a), the as-received powder has an irregular morphology. At the early stage of mechanical alloying, there is an increase in the average particle size of powders due to cold welding of the flattened particles (Fig. 1b). With further mechanical alloying up to 96 h (Fig. 1e), agglomerated particles composed of much finer particles are observed. A similar morphological evolution was obtained in $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ alloys.



(a)



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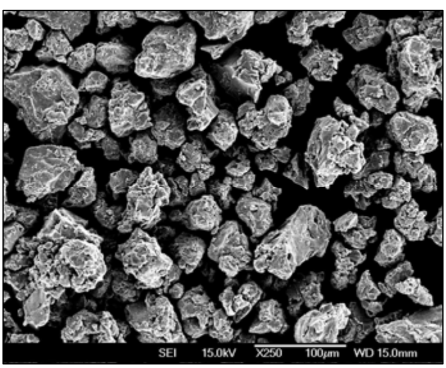

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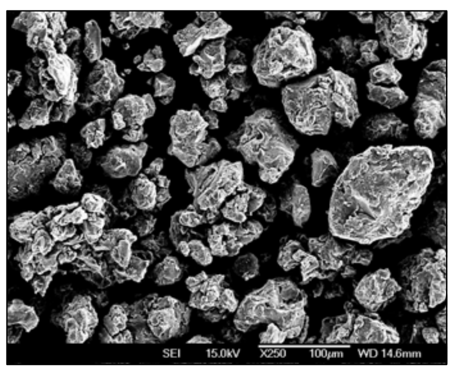

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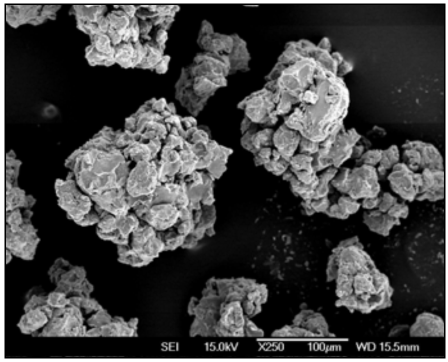

(e)

Fig. 1. SEM micrographs of $Fe_{10.8}Al_{89.2}$ powder mixtures after different mechanical alloying times (a) 0 h, (b) 24 h, (c) 48 h, (d) 72 h and (e) 96 h.

Figure 2 shows the XRD patterns of the $Fe_{10.8}Al_{89.2}$ powders after various mechanical alloying durations. The intensities of the diffraction peaks of the crystalline Fe and Al decreased and the widths of the peaks increased with the increasing mechanical alloying time as a result of the tendency of a nanocrystalline microstructure and the heavily deformation of the powder particles. The peaks of the Al phase, compared with the peaks of the Fe phase, decreased rapidly. At the early stage of the mechanical alloying, the Bragg peaks of Al are slightly shifted towards higher angles indicates the formation of amorphous phase coexisting with a nanocrystalline Al(Fe) solid solution. Further mechanical alloying up to 72 h, there are still remaining Al peaks in XRD pattern. Although the mechanical alloying has been prolonged to 96 h, a fully amorphous phase in the $Fe_{10.8}Al_{89.2}$ alloy does not occur, and the $Fe_{10.8}Al_{89.2}$ alloy is only partially amorphized. The similar results was reported for $Fe_{10}Al_{90}$ by Pekala *et al.* [9] and for $Fe_{10.7}Al_{89.3}$ by Mukhopadhyay *et al.*, using conventional horizontal low energy ball mill (800 h mechanical alloying) and Spex mixer/mill (50 h mechanical alloying), respectively. Considering that performed mechanical alloying conditions, it can be concluded that the formation of amorphous structures strongly depends on the alloy composition. Unless this alloy composition is changed, an increase in the mechanical alloying time and energy induced by mechanical alloying, amorphous structures cannot obtain.

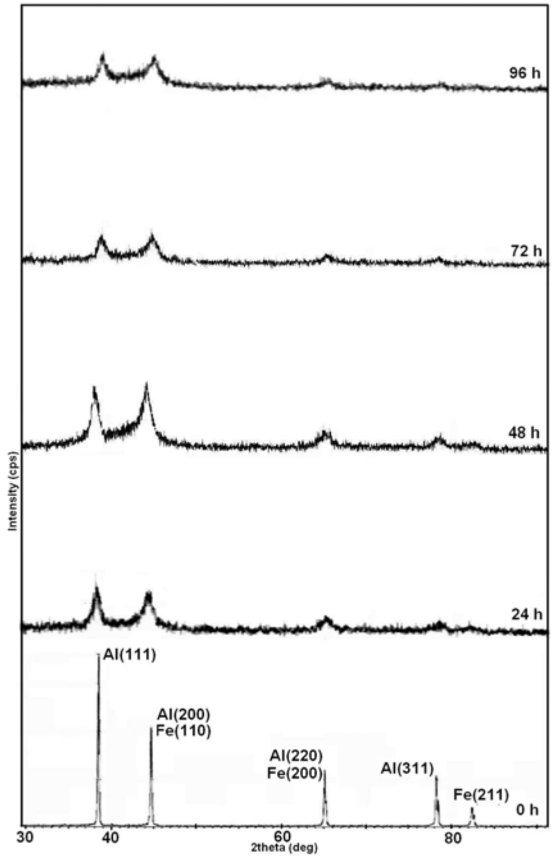

Fig. 2. X-ray diffraction patterns of $Fe_{10.8}Al_{89.2}$alloys after different mechanical alloying times.

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Figure 3 shows the XRD patterns of the $Fe_{80}Al_{20}$ powders after different mechanical alloying times. As seen, the mechanical alloying behaviour of $Fe_{80}Al_{20}$ alloy was in similar to $Fe_{10.8}Al_{89.2}$ alloy. The Al(111) and Al (311) peaks shifted to higher angles after 24 h of mechanical alloying, indicating diffusion of Al in Fe. The Al(111) and Al(311) disappear, after 24 h mechanical alloying. However, a longer mechanical alloying time led to no change in structure of powder. Fe(200) and Fe(211) peaks is still observed, after 96 h mechanical alloying. From the presence of these peaks, it may be concluded that a fully amorphous phase can not be obtained in this composition.

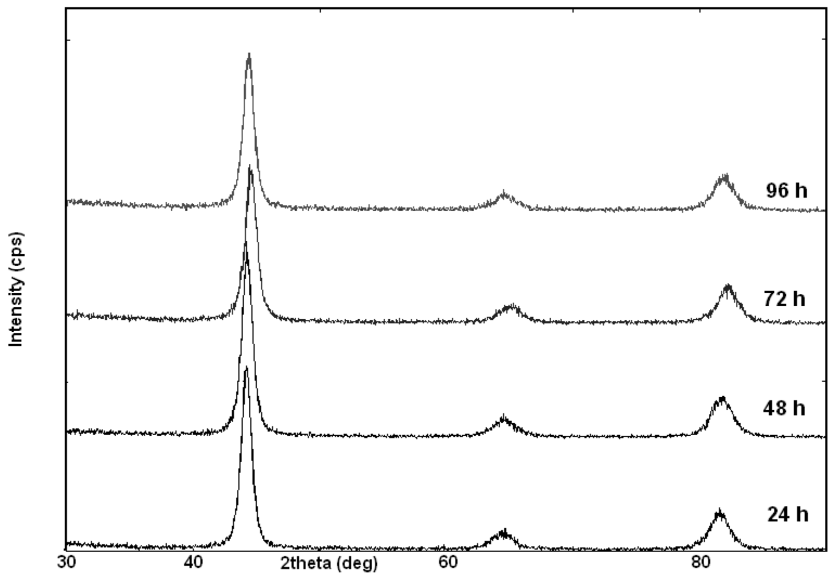

Fig. 3. X-ray diffraction patterns of $Fe_{80}Al_{20}$ alloys after different mechanical alloying times.

Figure 4 shows the XRD patterns of the $Fe_{50}Al_{50}$ powders after different mechanical alloying times. After 24 h of mechanical alloying, a broad peak overlapping with the major Bragg-peaks of Al(111) and Fe(110), indicating the existing of a major amorphous phase. All the minor Bragg-peaks of the Al(220), Al(311) and Fe(211) have completely disappeared and a second diffuse halo pattern has appeared, after 44 h of mechanical alloying. As seen in Fig.4, a single homogenous amorphous phase is obtained, characterized by smooth diffuse halos, after 50 h of mechanical alloying. Further mechanical alloying, for up to 96 h, causes crystallization of the amorphous phase.

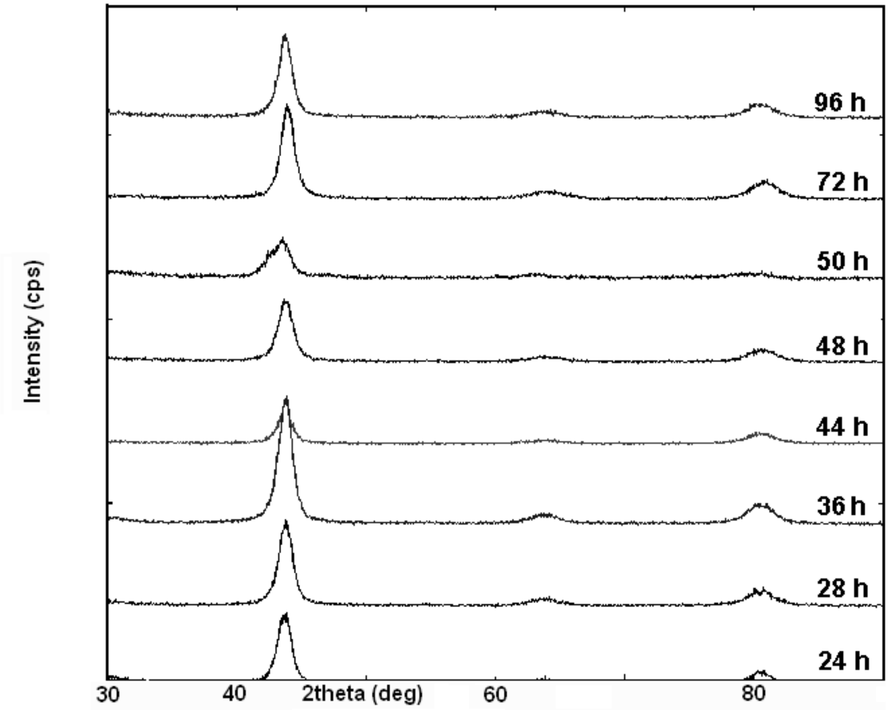

Fig. 4. X-ray diffraction patterns of $Fe_{50}Al_{50}$ alloys after different mechanical alloying times.

The formation enthalpies of solid solution or an amorphous phase can be calculated using Miedema's model (Fig. 5). According to calculated Miedema's semi empirical model of Fe-Al system by Mukhopadhyay *et all.*, mechanical alloying of Fe and Al powder mixtures with composition $Fe_{10.8}Al_{89.2}$, $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ resulted in amorphous + solid solution, single amorphous and amorphous + solid solution phases formation. The phases obtained with mechanical alloying of Fe-Al powders with composition $Fe_{10.8}Al_{89.2}$, $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ shows, in agreement with Miedema's model.

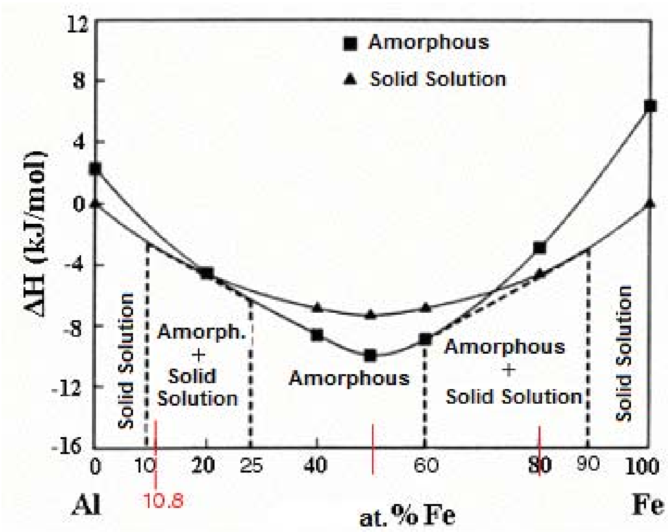

Fig. 5. Enthalpy-composition diagram of Fe-Al sytem (Miedema's model) [21].

IV. CONCLUSION

Elemental Fe-Al powders with compositions of $Fe_{10.8}Al_{89.2}$, $Fe_{50}Al_{50}$ and $Fe_{80}Al_{20}$ were mechanical alloyed in a Spex 8000D mixer/mill and the structural change was observed by SEM and XRD. The results show that:

(1) The powders of composition both $Fe_{10.8}Al_{89.2}$ and $Fe_{80}Al_{20}$ were found to form a mixture of amorphous and solid solution. A fully amorphous phase could not be achieved even after 96 h of mechanical alloying.

(2) A fully amorphous phase was obtained by mechanical alloying of $Fe_{50}Al_{50}$ alloy powders. The required time for amorphization is 50 h for 10:1 ball to powder weight ratio. Further mechanical alloying upon time need for amorphization, resulted in crystallization of amorphous phase.

(3) Good agreement with the Miedema model for amorphization was obtained in this study.

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