Journal of Nano- and Electronic Physics
Vol. **15** No 1, 01005(5pp) (2023)

Журнал нано- та електронної фізики
Том **15** № 1, 01005(5cc) (2023)

A Comparative Study of the Structural and Magnetic Properties of Arc-Melted and Ball-Milled $Fe_{1-x}Al_x$ Alloys

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(Received 09 October 2022; revised manuscript received 16 February 2023; published online 24 February 2023)

In this study, the authors prepared a series of technologically significant $Fe_{1-x}Al_x$ ($0.2 \le x \le 0.6$) alloys by two distinct methods: (a) arc melting and (b) ball milling and compared their structural and magnetic characteristics using XRD and VSM. Regardless of the synthesis method, structural analyses show that a FeAl alloy phase forms in both situations. Although FeAl alloy is formed using both processes, the diffraction patterns are indeed very different. In samples prepared by ball milling, the peaks are substantially wider than in samples obtained by arc melting. This is mostly due to the development of nanostructured disordered FeAl alloy during ball milling of the material. Aside from this, the existence of an Aluminum peak in a sample obtained by arc melting shows an unequal distribution of Al into the Iron matrix, whereas in a sample prepared by ball milling, Al is completely dissolved into the Fe lattice. Magnetic data indicate that the arc melted process favors the nonmagnetic FeAl alloy phase, whereas the ball milled method favors the weakly magnetic FeAl alloy phase. The existence of weak magnetism in a ball-milled sample is explained by considering the system's degree of nanocrystallization and disorder.

Keywords: Arc melting, Mechanical alloying, Fe-Al phase, XRD, Magnetism.

DOI: 10.21272/jnep.15(1).01005 PACS numbers: 81.20.Ev, 81.07.Bc, 75.75. + a, 71.20.Lp

1. INTRODUCTION

In recent years, the study of intermetallics has garnered a significant amount of attention from the scientific community. The reason for this is because intermetallics possess unique and highly desirable physical features [1, 2]. Over the past few decades, researchers have been exploring the possibility of using ordered intermetallics, which are based on the aluminides of transition metals, particularly iron, as high-temperature structural materials [4-6]. Because of its superior physical, chemical, and mechanical characteristics, such as low density, exceptional corrosion, oxidation resistance, and high strength at both room and increased temperatures [1, 7, 8], FeAl intermetallics are gaining popularity in the field of materials engineering. The density of FeAl alloys decreases as their Al content rises, while the alloys' resistance to oxidation and sulphidization improves [9]. FeAl intermetallics with high Al concentrations are promising options for structural materials under adverse situations. However, FeAl intermetallics' limited applicability has been caused by their intrinsically low ductility and toughness, especially at ambient temperatures. In FeAl intermetallics, increasing Al content decreases ductility [10, 11]. Strong augmentation of diffusivity in nanophase materials [12], attributable to grain-boundary mechanisms, is said to have significant effects on the ductility at low temperatures. Therefore, two main approaches were generally followed to improve the ductility: The first includes careful control of grain-boundary cohesion by micro-alloying and the second includes the improvement of the suitable grain refinement processing, such as inoculation, rapid solidification, and mechanical alloying (MA) techniques. When metals are alloyed mechanically, the solubility limits of the constituent solids shift, and phase transformations are triggered. Important alloying methods include ball milling and arc melting. Both methods have their benefits and drawbacks, so it is important to consider them both before making a decision. As a result of their advantageous mechanical and corrosion resistance properties [13-15], FeAl based alloys are prepared using both methods.

In this paper, the author has taken these considerations into account and meticulously prepared FeAl alloys via Ball milling and Arc Melting methods and performed comparative structural and magnetic characterizations in order to collect pertinent information about the various probable phases of FeAl in order to determine when to apply which technique when working with this system. The author has also made an effort to provide an analysis of the findings, including consideration of potential explanations and justifications.

2. EXPERIMENTAL DETAILS

A series of intermetallic $Fe_{1-x}Al_x$ alloys with composition in the range ($0.2 \le x \le 0.6$) have been prepared by arc melting and high energy MA process.

Arc Melted Sample: Fe and Al metals of high purity (more than 99.9 %) were used to prepare FeAl alloys using arc melting process in Argon atmosphere. Care was taken to avoid oxygen contamination and to achieve the same Titanium (Ti) was evaporated inside the chamber before melting of the metals started. The prepared alloys were cut into small pieces and annealed at 600 °C for 120 h under the UHV condition.

Ball Milled Sample: The second series of FeAl alloy sample were prepared by ball milling method. Initial mixing was done by pestle and mortar and then for further milling the SPEX 8000M high energy mill machine was used to serve the purpose. The analytical grade Fe and Al powders with a purity of 99.9 % were used. A ball to powder ratio of 20:1 was maintained for

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better milling results. In order to form alloy the sample was milled for 5 h under Argon atmosphere.

X-Ray diffraction (XRD) technique was employed to determine the structure and particle size of the samples. The XRD measurements were carried out at a wavelength (λ) of 1.542 Å, operated at 40 kV and 30 mA. The average crystallite size D was calculated from the broadening of X-Ray diffraction peaks using Scherrer's formula. The *M-H* curve was recorded using vibrating sample magnetometer (VSM Lakeshore-7300 model).

3. RESULTS AND DISCUSSION

Fig. 1 shows the comparison of XRD patterns of $Fe_{1-x}Al_x$ alloys prepared using arc melting (Fig. 1a) and ball milling (Fig. 1b) methods. It shows that the peaks obtained from arc melted samples are very sharp whereas the peaks obtained from ball milled samples are broadened and relatively lower in intensity. The observed differences in the two cases are mainly because of the large differences in their crystallite size.

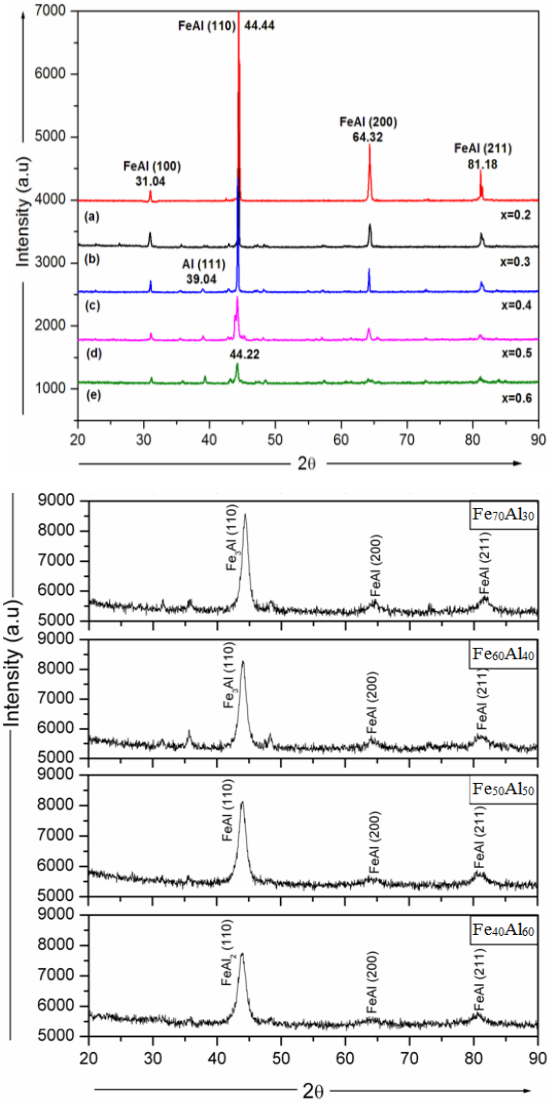


Fig. 1 – Comparison of XRD patterns of $Fe_{1-x}Al_x$ alloys prepared using (a) arc melting and (b) ball milling methods

It is to be noted that the diffraction spectra of arc melted samples (especially for $Fe_{0.6}Al_{0.4}$, $Fe_{0.5}Al_{0.5}$, and $Fe_{0.4}Al_{0.6}$ samples) shows a small peak at 39.04° corresponds to Al (111). It indicates that whole Al does not intermix completely with Fe to form uniform FeAl alloy. Whereas in case of ball milled samples more uniform alloy formation takes place as compared to arc melted samples.

Further the intensity of arc melted samples is very high as compared to milled samples. This can also be attributed to difference in particle size and phase changes during alloying process. It is also seen that the intensity decreases with increase in Al content. The XRD graph of both cases indicate that major peak at $2\theta = 44.6°$ is shifted towards the lower angles with increasing Al content. This can be attributed to the expansion of the lattice and presence of internal strain occurring as a result of non-uniform alloy formation during sample preparation.

3.1 Comparison of the Crystallite Size and Lattice Parameters of FeAl Alloys

Fig. 2 shows the comparison of crystallite size and lattice parameter of the corresponding samples prepared by (a) arc melting and (b) ball milling methods, respectively. The study shows that the crystallite size decreases with increasing Al content in both the cases whereas the crystallite size is very much small in case of ball milled samples. The average crystallite size of milled samples is nearly in the range of 8 nm to 6 nm and that of arc melted samples is nearly in the range of 110 nm to 28 nm with variation of Al content.

The lattice parameter shows opposite behavior than crystallite size. In both cases, the lattice parameter (a_0) increases with increasing Al concentration. It can be due to the migration of Al atoms in to lattice of Fe producing a local dilatation because of their larger size. The lattice expansion of ball milled samples is more than that of arc melted samples. This can be attributed to smaller crystallite size of ball milled samples. In case of arc melted samples, the lattice parameter increases from 2.87 to 2.89 whereas in case of milled samples it increases from 2.89 to 2.92 which is exactly equal to JCPDS data value.

3.2 Comparison of Magnetic Properties

The magnetic study of the corresponding samples was made through VSM. The hysteresis loops observed for arc melted and ball milled samples are shown in Fig. 3, respectively. In arc melted samples, the Fe rich samples show saturation magnetization and other samples do not show saturation magnetization even after maximum applied field of 1.4 Tesla. However, all the ball milled samples show the saturation magnetization. In both the cases, the saturation magnetization decreases with increasing Al content. This characteristic reflects the gradual development of alloying process of Fe with Al. Aluminum atoms reduces the direct ferromagnetic (FM) interaction between Fe-Fe sites and at the same time increase in anti-ferromagnetic (AFM) interaction could take place, which reduces the magnetic moment of Fe.

The hysteresis loop is also shifted in case of arc

melted samples and this shift increases with increase in Al concentration. This loop shift can be explained on the basis of exchange bias phenomenon associated with the exchange anisotropy created at the interface between AFM-FM phases. The loop is further shifted indicating the increase in AFM-FM coupling with more addition of Al. But in case of ball milled samples there is no loop shift. This can be attributed to more uniform alloying in case of ball milled samples.

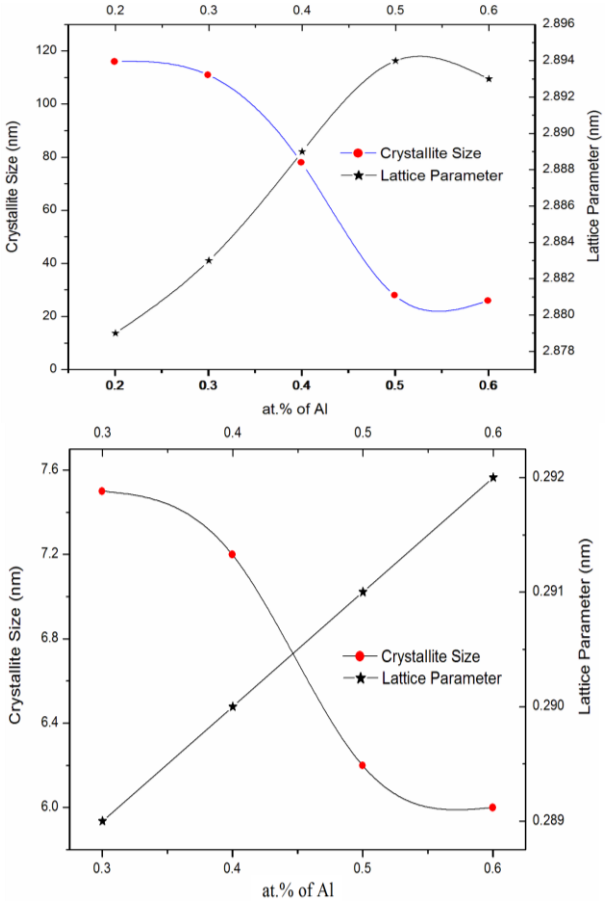
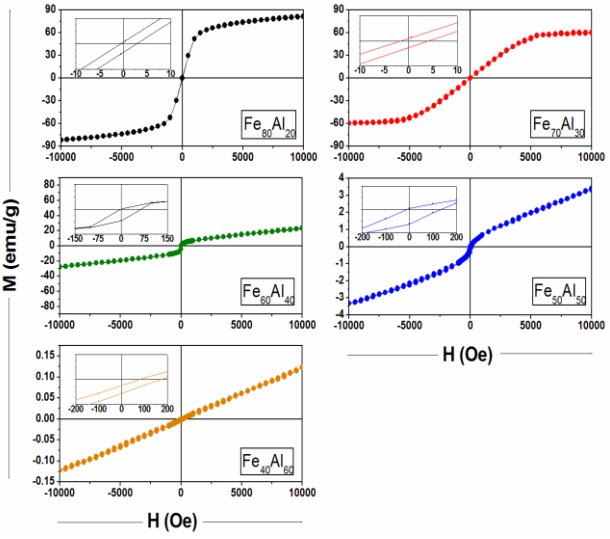


Fig. 2 – Variation in crystallite size and lattice parameter of (a) arc melted and (b) ball milled $Fe_{1-x}Al_x$ ($0.3 \le x \le 0.6$) alloy samples as a function of x



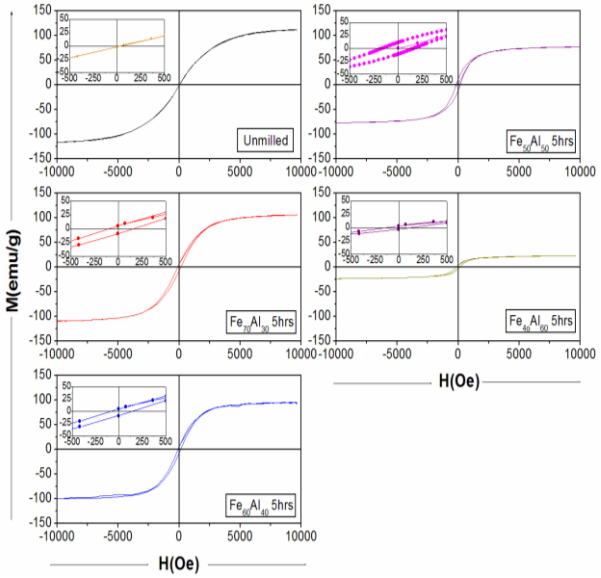


Fig. 3 – *M-H* curves of (a) arc melted and (b) ball milled $Fe_{1-x}Al_x$ ($0.3 \le x \le 0.6$) alloy samples as a function of x

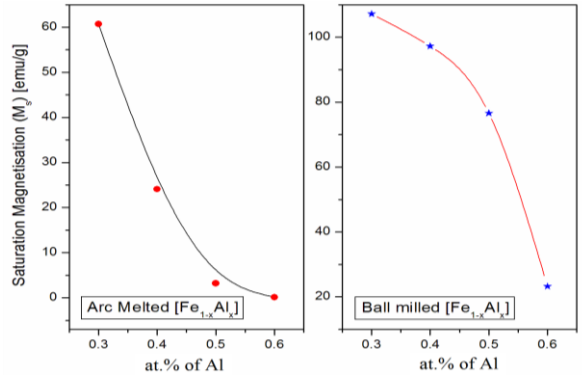


Fig. 4 – The comparison of saturation magnetization (M_s) of arc melted and ball milled $Fe_{1-x}Al_x$ ($0.3 \le x \le 0.6$) samples as a function of x

3.3 Comparison of Saturation Magnetization Results

In both the cases, the saturation magnetization decreases with increase in Al concentration (see Fig. 4). As the paramagnetic behavior dominate in Al rich samples and magnetism is due to Fe cluster. The observed magnetic behavior is mainly attributed to the formation of different FeAl phases and increase in anti-ferromagnetic interlayer coupling with addition of Al. The value of saturation magnetization in case of milled samples varies from 111 to 28 emu/g whereas, in case of arc melted samples it varies from 60.7 to 0.13 emu/g only.

3.4 Comparison of Coercivity Results

The coercivity in arc melted samples increases with increase in Al concentration (see Fig. 5) which can be attributed to enhancement in the anisotropy as a result of non-uniform and disordered formation of non-magnetic FeAl phases. In case of ball milled samples it decreases first and then again increases and after that again decreases which can be attributed to uniform alloying process and small particle size.

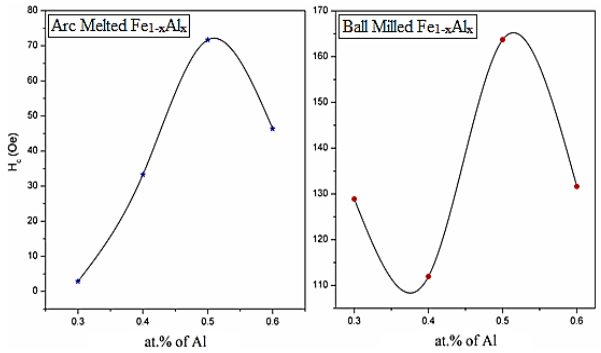


Fig. 5 – The comparison of variation in coercivity (H_c) of arc melted and ball milled $Fe_{1-x}Al_x$ ($0.3 \le x \le 0.6$) samples as a function of x

4. DISCUSSION

The comparison of structural and magnetic characteristics demonstrates that these disparities arise from the formation of different phases and non-equilibrium structures as a result of two distinct fabrication methods. Compared to arc-melted samples, ball-milled samples show more homogeneous alloy formation. Due to the repetitive welding, fracturing, and re-welding of powder particles in high energy ball milling, the crystallite size decreases, and once the structure is sufficiently small, solid-state interactions between the initial phases are activated, resulting in mechanical alloying. Second, there is adequate time during the process to develop ordered and balanced structures. In the case of arc melting, the arc is struck on the material to be melted while argon gas flows continuously. In order to create a uniform and homogenous melt, the stingers are moved over and around the material. In order to ensure the homogeneity of the sample, the specimen is repeatedly re-melted and rotated. Unlike the above, the arc melting process is a non-equilibrium process that lacks adequate time to generate stable and ordered phases, resulting in the formation of metastable and disordered phases. Due to the fact that Al atoms are more mobile than Fe atoms, it is anticipated that highly mobile Al atoms will diffuse into the more static lattice of Fe, resulting in the production of disordered FeAl intermetallic alloys. For the aforementioned reasons, the characteristics of samples collected using two different approaches are slightly distinct from each other. Additionally, the crystallite size differs between the two situations. (28 nm for materials melted by an arc and 6 nm for samples milled by a ball mill). The variation in saturation magnetization and coercivity is attributable to a difference in crystallite size and an increase in anisotropy resulting from disordered and non-uniform phases. Lastly, the differences between the two series can be explained by differences in particle size and the way the phases form (ordered or disordered).

5. CONCLUSIONS

The following inferences can be made after considering all of the aforementioned measurements described above.

- The potential Phases of alloys to be formed are significantly affected by the synthesis process.
- The nanostructure and the amount of disorder present in the system have a direct bearing on the structural and magnetic characteristics of these systems.
- The Arc Melting process encourages the formation of the non-magnetic FeAl phase, whereas the Ball Milling process encourages the formation of the disordered FeAl magnetic phase.

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Порівняльне дослідження структурних і магнітних властивостей сплавів $Fe_{1-x}Al_x$, виготовлених за допомогою дугового плавлення та кульового помелу

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У дослідженні автором підготована серія технологічно значущих сплавів $Fe_{1-x}Al_x$ ($0{,}2 \le x \le 0{,}6$) за допомогою двох різних методів: дугового плавлення та кульового помелу та порівняні їх структурні та магнітні характеристики за допомогою XRD та VSM. Незалежно від методу синтезу, структурний аналіз показує, що в обох ситуаціях утворюється фаза сплаву FeAl. Хоча сплав FeAl утворюється за допомогою обох процесів, дифракційні картини дуже відрізняються. У зразках, отриманих методом кульового помелу, піки значно ширші, ніж у зразках, отриманих дуговим плавленням. Це здебільшого пов'язано з формуванням наноструктурованого невпорядкованого сплаву FeAl. Окрім цього, наявність піку алюмінію у зразку, отриманому дуговим плавленням, показує нерівномірний розподіл Al у матриці заліза, тоді як у зразку, отриманому кульовим помелом, Al повністю розчинений у решітці Fe. Магнітні дані вказують на те, що процес дугового плавлення сприяє немагнітній фазі сплаву FeAl, тоді як метод кульового помелу сприяє слабомагнітній фазі сплаву FeAl. Існування слабкого магнетизму в змеленому зразку пояснюється врахуванням ступеня нанокристалізації та безладу системи.

Ключові слова: Дугове плавлення, Механічне легування, Фаза Fe-Al, XRD, Магнетизм.