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Synthesis of a pure FeAl with a density greater than 95% of theoretical density by optimizing effective factors

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ABSTRACT: In this study, the FeAl with a density greater than 95% of the theoretical density was synthesized by optimizing the compacting pressure, the particle size of iron and aluminum, and the pre-heating temperature. To do so, the products of the samples which were examined by the change of the particle size of 10μm and 50μm, the pre-heating temperatures of 25°C, 287°C and 550°C and the compacting pressures of 400MPa, 500MPa, and 600MPa, were studied. According to the appearance, composition, density, hardness, and oxidation resistance, the optimum sample was determined. The optimal FeAl had a density of more than 95% of the theoretical density, which was prepared using the particle size of 10/10 of iron and aluminum, the pre-heating temperature of 550°C, and the compacting pressure of 500 MPa. The duration of the pre-heating and the synthesis was also 5 and 3h, respectively. It was found that without pre-heating, there is no possibility of producing a compact and dense product. It was also found that the particle size of the iron is more effective than the particle size of aluminum to achieve the qualified FeAl product.

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1- Introduction

Considerable attention has been paid to intermetallic compounds during the recent five decades. That's because of their attractive physical and mechanical properties. The monolithic of iron aluminides based on FeAl will be applied as a substitution for steels and superalloys for structural applications at elevated temperatures [1]. The unique combination of the excellent resistance against the sulfiding, carburizing, and oxidizing environments at high temperatures (up to 1200°C) coupled with a low density, good mechanical properties at high temperatures (up to 800°C) and a low cost, together with have caused industrial interest in the FeAl alloys and composites for different applications such as gas-metal filters, coating for boilers and heating elements, foods industry parts, automotive parts, rails, etc. [1,2].

The keynote here is to develop economical processing methods to utilize the pure and dense attributes of intermetallic compounds in developing critical aerospace or other high-performance FeAl product. One of the significant viewpoints is based on using elemental powders [3]. This approach is known as Reaction Sintering, Combustion Synthesis, or Self-Propagating High-Temperature Synthesis, in which an exothermic reaction occurs between the elemental powders [4].

Several authors have synthesized iron aluminides by the combustion synthesis and have concluded that the main disadvantage of this process was the large porosity in the final products [3,5]. Although this method has been successfully applied, the final components' mechanical properties are still lower than those obtained from the other methods. The extent of swelling observed in such systems depends upon some processing variables, including compaction, particle sizes, heating rate, green density, and temperature [4,6]. Fig. 1 shows the iron-aluminum phase diagram. Swelling is predictable based on the phase diagram features; notably, there is a large solubility for aluminum in iron, low reverse solubility, and a large melting point difference, suggesting imbalanced diffusion rates. Systems that exhibit a large driving force for compound formation are particularly susceptible to porosity formation during the alloying [4].

Gedevanishvili et al. [3] investigated the sintering behavior of Fe and Al's elemental powders in the range of 1000-1300°C using the dilatometric method. According to the results, it was found that the formation of FeAl leads to an expansion due to the volume change during the formation of the intermediate phase of Fe_2Al_5. The FeAl formation mechanism and the final density depend on the heating rate. During the slow heating rate (0.5 °C min−1), the expansion rate was 0.0018 mm/min and 0.34 mm/ min at a higher heating rate of 1 °C min−1. Reducing the heating rate (0.5°C min−1) leads to the lower expansion and subsequently to a higher density of the produced FeAl up to 94.5% of theoretical density. Jia et al. [7] studied the mechanical properties and the related densities of the prepared FeAl samples

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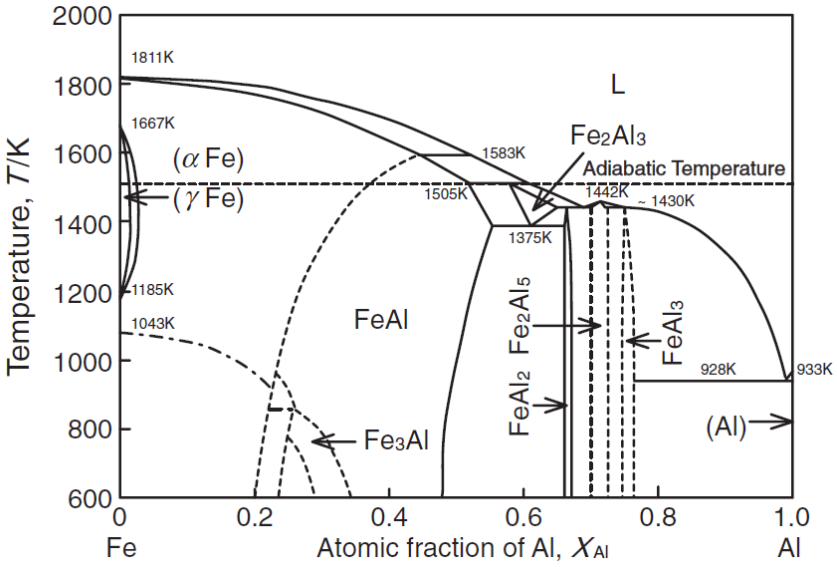


Fig 1. Fe–Al phase diagram system [8]

using the mechanical alloying and the vacuum hot-pressing. The results show that the hot-pressed FeAl intermetallic has a reasonably good strain at break of 3.2%, while the addition of 0.5 at.% B (Boron) reduces the peak temperature for hot-pressing from 1180°C to 1100°C and increases the compacts' density from 95% to 96.3% of the theoretical density. Durejko et al. [10] studied the influential factors on the Fe-Al system. They have used the sintering method under a cyclic loading. Nosewicz et al [11] presented a numerical and experimental survey of powder metallurgy techniques for production of intermetallic ceramic composites.

1]The current study aims to obtain pure, stiff, oxidation resistant, and dense FeAl by optimizing the compacting pressure, the particle size of iron and aluminum, and the pre-heating temperature.

To do so, the products of the samples which were examined by the change of the particle size of 10µm and 50µm, the pre-heating temperatures of 25°C, 287°C and 550°C and the compacting pressures of 400MPa, 500MPa, and 600MPa, were studied. The optimum sample was determined according to the appearance, composition, density, hardness, and oxidation resistance. The optimal FeAl had a density of more than 95% of the theoretical density, which was prepared using the particle size of 10/10 of iron and aluminum, the pre-heating temperature of 550°C, and the compacting pressure of 500 MPa. The duration of the pre-heating and the synthesis was also 5 and 3h, respectively.

2- Experimental Method

Iron powders (99.5%, 10 µm and 50 µm, from Merck company) and aluminum powders (99.99%, 10µm, and 50µm, from Merck company) were mixed with the molar ratio of 1:1 and milled for 2 minutes in a fast mill at 400 rpm in an alumina jar, including 16 alumina balls of 2cm diameter. The Ball ration to Powder Ratio (BPR) was equaled (5:1). After the milling procedure, the mixed powder was then pressed in a mold to obtain a pellet with a diameter of 0.8 cm. Fig. 2 demonstrates the schematic of the mold used in this research.

The experiment's conditions (how the parameters have changed during the experiment) are given in Table 1. The different compacting pressures (400, 500, and 600 MPa), the different particle sizes of aluminum and iron (10 and 50µm), and the different pre-heating temperatures (25, 287, and 550ºC) were applied to investigate their effects on the quality of the final FeAl product. The duration of pre-heating and the heating was 5 hours each.

To prevent the oxidation of the products, heat treatment was performed under an argon atmosphere. The applied setup is shown in Fig. 3. The argon gas was passed through a heated pure Cu at a temperature of 550°C to eliminate O_2. Ascari and Drierite have also been utilized to eliminate CO_2 and H_2O, respectively.

The dimensions and the weight of the final samples were measured to obtain the density. The hardness of the samples was measured using a digital hardness tester. The oxidation of the samples was performed at 850°C in the tube furnace (Fig. 3). The samples were exposed to the air for 48 hours. The extent to which oxidation occurred was measured by calculating the extent of gain weight per cm^2 of sample area (g/cm^2).

3- Results and Discussion

As previously stated in the section 2, the purpose of this study was to synthesize the FeAl with high quality by optimizing the compacting pressure, the particle size of iron and aluminum, and the pre-heating temperature. To achieve this goal, the particle size of 10 and 50µm, the pre-heating temperatures of 25, 287, and 550ºC, and the compacting pressures of 400, 500, and 600 MPa were chosen based on the previous published researches results [3-9]. Therefore, the experiments were designed based

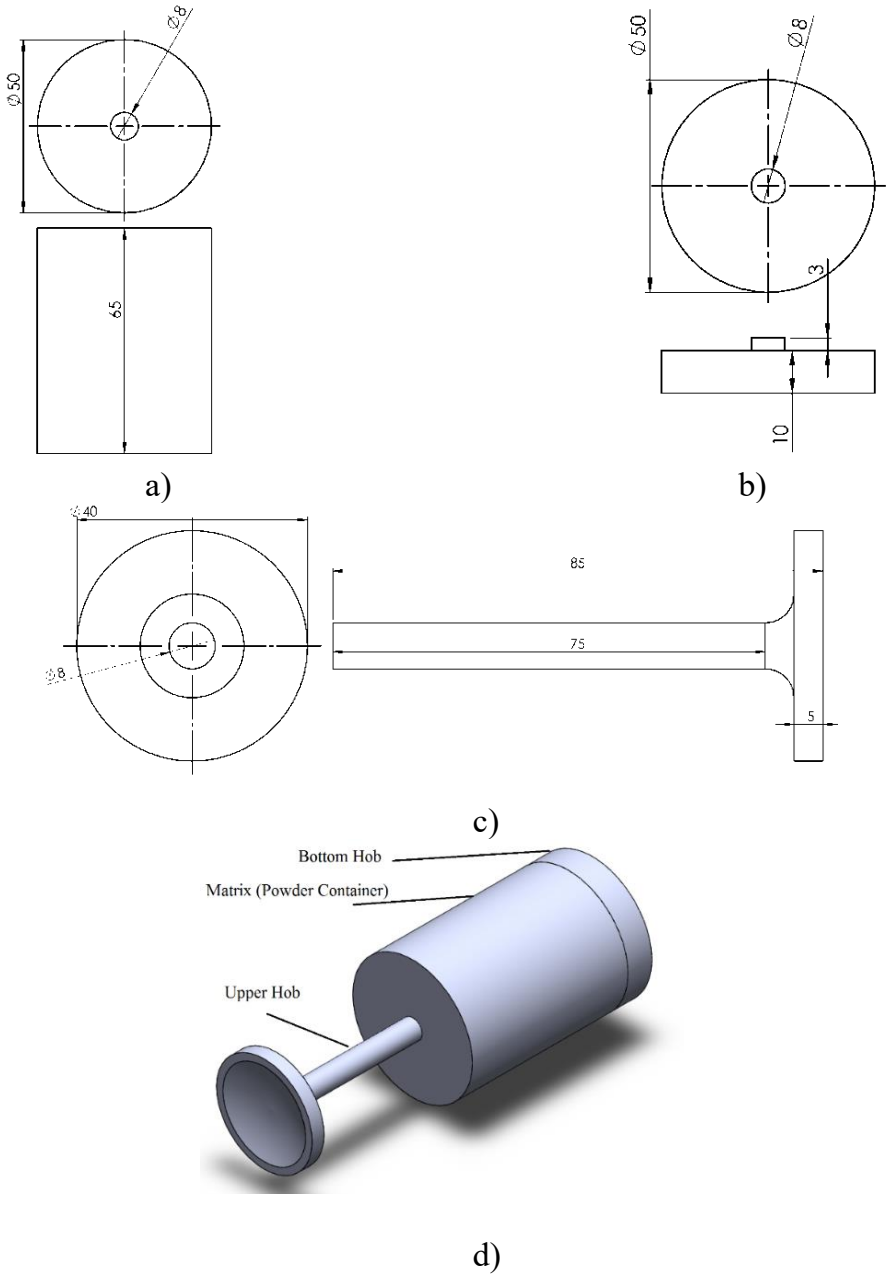


Fig 2. Schematic of the mold, a) Powder container (matrix), b) Bottom hob (fixed hob), c) Upper hob (pressing hub) and d) 3D view of the mold

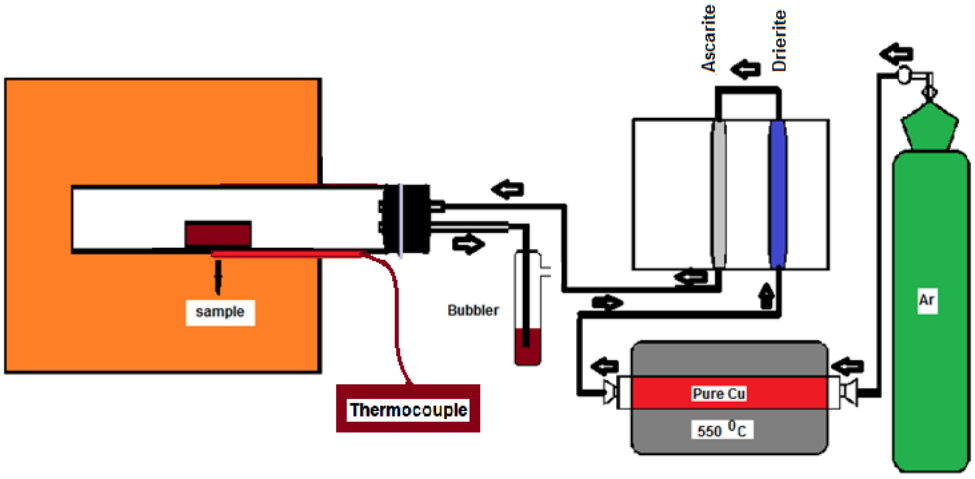


Fig. 1. Phase velocity dispersion curves for a steel pipe with outer diameter of 220 mm and wall thickness of 4.8 mm

Table1. The arrangement of the influential factors (particle size of aluminum and iron, compacting pressure, pre-heating temperature, and final synthesis temperature) in the experiments

Al Particle Size/Fe Particle Size	Pre-heating Temperature (ºC)	Compacting Pressure (MPa)	Synthesis Temperature (ºC)	Appearance
10/10	25	500	950	Dense
	287	500	950	Dense
	550	500	950	Dense
10/50	25	500	950	Swelled, Porous, and Brittle
	287	500	950	
	550	500	950	
50/10	25	500	950	Crooked
	287	500	950	Crooked
	550	500	950	Dense
50/50	25	500	950	Swelled, Porous, and Brittle
	287	500	950	
	550	500	950	

on Table 1.

Considering the synthesized samples' appearance, the samples containing the aluminum and iron with the particle size of 50/50 and 10/50 had a swelled, brittle and porous structure. In contrast, the samples with the particle size of 50/10, which was not pre-heated (pre-heated at 25ºC) and pre-heated at 287ºC, were crooked. Thus, all of these conditions were eliminated from further experiments. The swelling, brittle and porous appearance of the samples with the bigger particle size of iron suggests that the iron particle size is more critical than the aluminum size to achieve a high-quality FeAl. Both smaller and larger particle sizes of aluminum showed a suitable appearance. The result of which would be the size of the aluminum particles has less effect on the quality of FeAl. The reason can be the flexibility and the formability of aluminum, leading to change in the aluminum powder shape under pressure and helping the aluminum to surround the iron particles and fill the narrow and empty places.

In order to obtain the optimum compacting pressure, the three pressures were applied (400, 500, and 600 MPa) on the samples containing the particle size of 10/10 of aluminum and iron-

Table 2. The experiment conditions to investigate the effect of the compacting pressure

Al Particle Size/Fe Particle Size	Pre-heating Temperature (ºC)	Compacting Pressure (MPa)
10/10	550	400
		500
		600

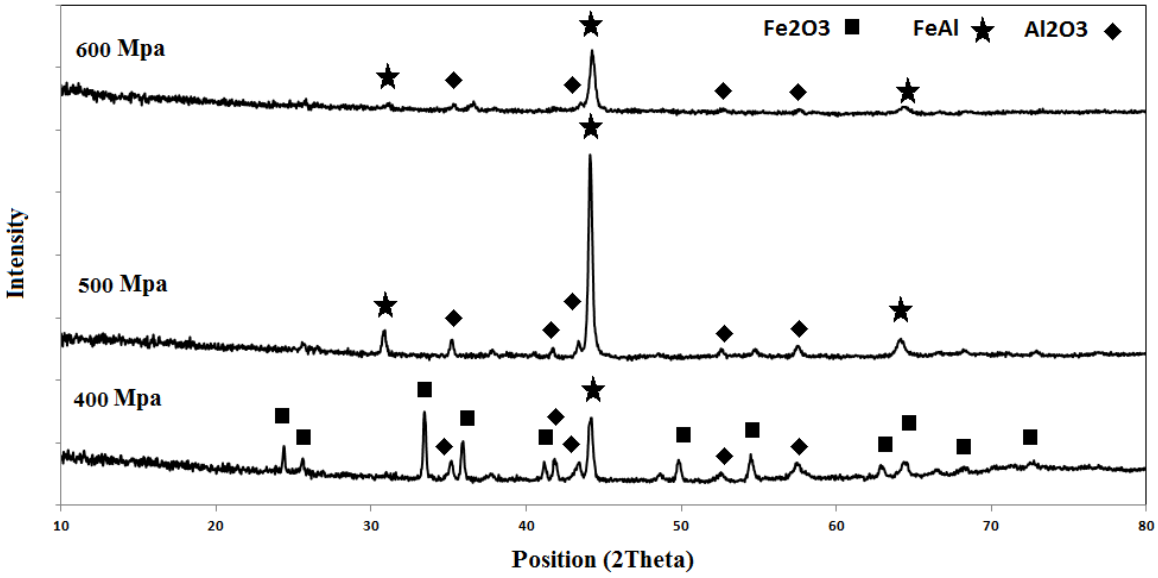


Fig 4. XRD patterns for the samples containing the particle size of 10/10 of aluminum and iron compressed under the different pressures (400, 500, and 600 MPa), pre-heated at 550ºC and heated at 950ºC

based on the conditions mentioned in Table 2. The samples with the particle size of 10/10 of aluminum and iron were selected because their appearance was dense at all conditions, as shown in Table 2. That's because the appearance of all the synthesized samples under the conditions mentioned in Table 2 was dense and similar. It was not possible to identify the optimal sample from the appearance. Thus the XRD analysis was performed on the sample products. Fig. 4 shows the XRD results for the samples with the particle size of 10/10 compressed under the different pressures (400, 500, and 600 MPa), pre-heated at 550ºC and heated at 950ºC. As illustrated in Fig. 4, the sample which has been prepared with the minimal compacting pressure (400 MPa) contains iron oxide (Fe_2O_3) and alumina (Al_2O_3)—reducing the compacting pressure results in more porosity and internal surface formation. As a result of the particle's susceptibility to react with the oxygen, the porosities lead to the oxides formation (Fe_2O_3 and Al_2O_3). Also, due to the reduction of the contact surface of the primary powders, the reaction speed is slowed down, and the raw materials are exposed to the argon gas for a longer period of time. Although the gas is refined, it has a small extent of oxygen that can react with the powders. With increasing the compacting pressure, FeAl was formed sooner and only a very small amount of alumina was observed in the X-ray pattern as the result of the FeAl oxidation.

Due to the formation of iron oxide in the sample with the lowest compacting pressure (400 MPa), it is evident that some of Fe are wasted. So this cannot be desirable. Under the pressure of 500 and 600 MPa, FeAl and Al_2O_3 are the dominant phases, but since the sample is compacted under the 500 MPa, the central peak of FeAl is much longer than the other peaks. It means that the ratio of FeAl is the most in this sample. It also confirms that the optimum compacting pressure is 500 MPa.

Table 3. The experimental conditions to investigate the effect of pre-heating temperature

Al Particle Size/Fe Particle Size	Pre-heating Temperature (ºC)	Compacting Pressure (MPa)	Synthesis Temperature (ºC)
10/10	25	500	950
	287	500	950
	550	500	950

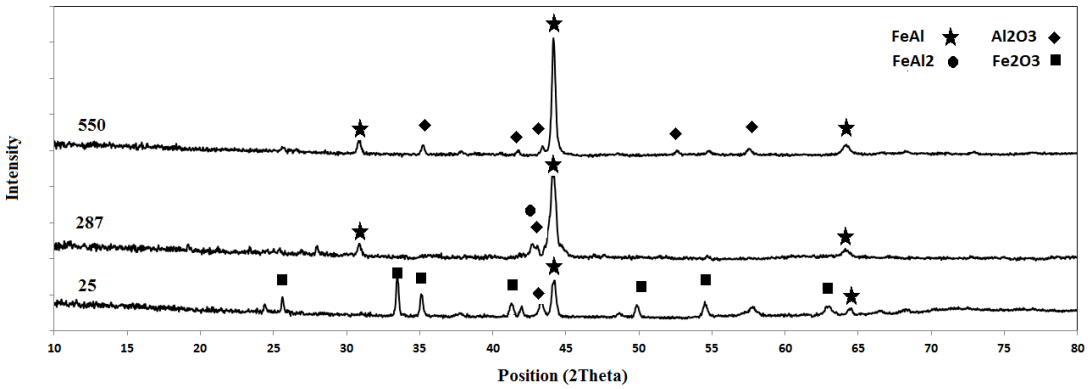


Fig 5. XRD patterns for the samples contain the particle size of 10/10 of aluminum and iron compressed under the 500 MPa pressure, pre-heated at 25, 287, and 550ºC and heated at 950 ºC

For the third step, applying the optimum compacting pressure (500 MPa), the optimum pre-heating temperature is investigated using the experiments' arrangement based on Table 3. Considering the appearance of the final product, all of them were dense. Thus XRD analysis was applied for the samples to determine the produced phases. Fig. 5 shows the XRD results for the samples containing the particle size of 10/10 compressed under the 500 MPa, pre-heated at 25, 287, and 550ºC, and synthesized at 950ºC. As shown in Fig. 5, in the sample pre-heated at 550ºC, FeAl produced as the dominant phase while there is a little amount of Al_2O_3 formed. This is the result of the use of the optimized pre-heating temperature of 550ºC. This result could also be deduced from the results of Table 1. The results of the obtained products in Table 1 showed that the sample contained the particle size of 50/10 of aluminum and iron pre-heated at 550ºC has the qualified appearance compared to the similar particle size powders, pre-heated at 25 and 287ºC. The result of this step strongly confirms that pre-heating is necessary to achieve a dense product.

To determine the revolutions made during the pre-heating process at 550ºC, the sample's composition with the particle size of 10 and 10 of aluminum pressed under 500 MPa pressure and heated for 5h at 550ºC, the XRD results were analyzed (Fig. 6). As illustrated in this Fig., the FeAl production process started, but it was not completed, since $FeAl_2$ remained the transient phase. This is due to the FeAl formation mechanism. The formation mechanism of FeAl is based on the formation of a FexAly intermetallic composition, which consequently progresses by the diffusion of aluminum through the FexAly layer and FeAl forms [8]. Since the pre-heating temperature (550ºC), more time is required for FeAl to form, some of the transient phases ($FeAl_2$) remained unchanged.

Pre-heating the samples before the melting point of aluminum causes the reactions to start in the solid-state. Since the reaction speed in the solid-state is much slower than the liquid state, the system has enough time to minimize its dimensional variation by diffusing the elements (Al). Although FeAl production is not completed in this temperature, small amounts of transformation remain for the higher temperatures (950ºC). On the other hand, since the pre-heating process at 550°C has been extended for 5 hours and takes 5 hours to react at 950°C, in total, the sample took 10 hours to react. Therefore, the reaction occurred gradually, and the least distortion was created in the sample.

After determining the optimum pre-heating temperature, the optimal conditions were applied to the samples with the particle size of 10/10, 10/50, 50/10, and 50/50 of aluminum and iron. The experimental conditions, final products (based on the XRD analysis), and the product's appearance are shown in Table 4.

The XRD results for the samples containing the particle size of 10/10, 50/10, 10/50, and 50/50 of aluminum and iron compressed under 500 MPa, pre-heated at 550ºC and heated at

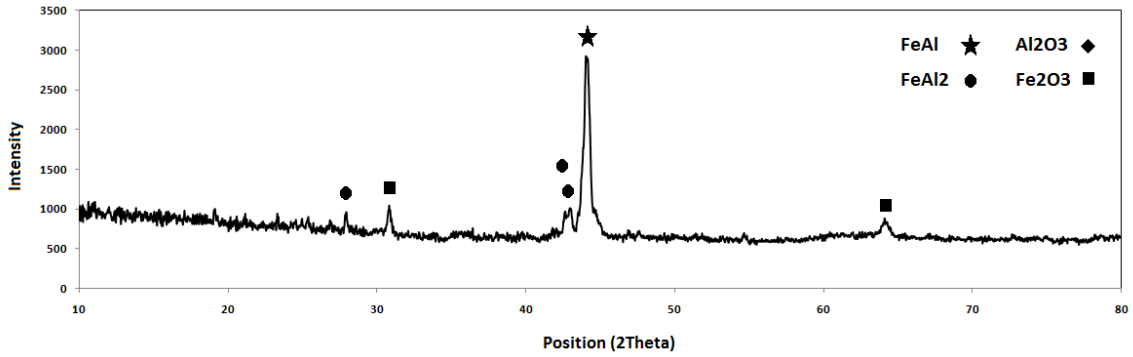


Fig. 6. XRD patterns for the samples containing the particle size of 10/10 of aluminum and iron compressed under the 500 MPa pressure and pre-heated at 550°C for 5h

Table 4. The experimental conditions to investigate the effect of the particle size of aluminum and iron and the appearance and compositions of the final product

Al powder size (μm)	Fe powder size (μm)	Pre-heating Temperature	Compacting Pressure	Synthesis Temperature	The Appearance	The Product composition
10	10	550	500	950	Dense	FeAl, Al_2O_3
50	10	550	500	950	Dense	FeAl
10	50	550	500	950	Swelled, Porous, and Brittle	FeAl, Al_2O_3
50	50	550	500	950	Swelled, Porous, and Brittle	FeAl, Al_2O_3

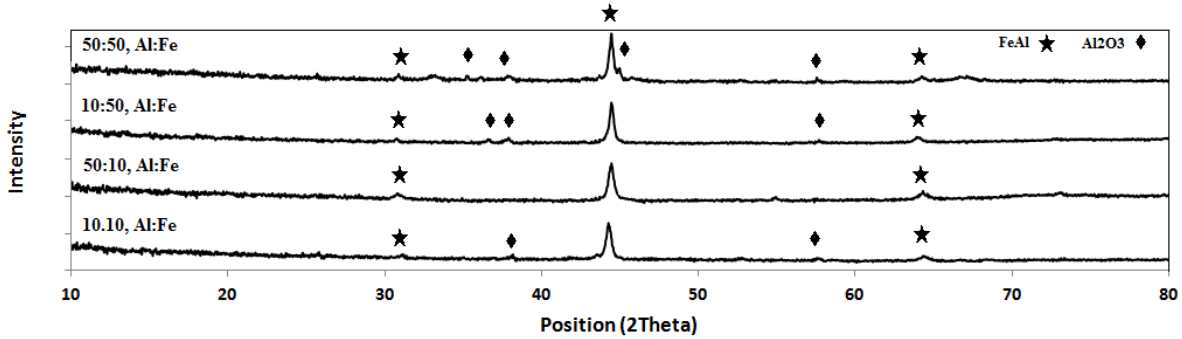


Fig 7. XRD patterns for the samples containing the particle size of 10/10 of aluminum and iron compressed under the different pressures (400, 500, and 600 MPa), pre-heated at 550°C and heated at 950°C

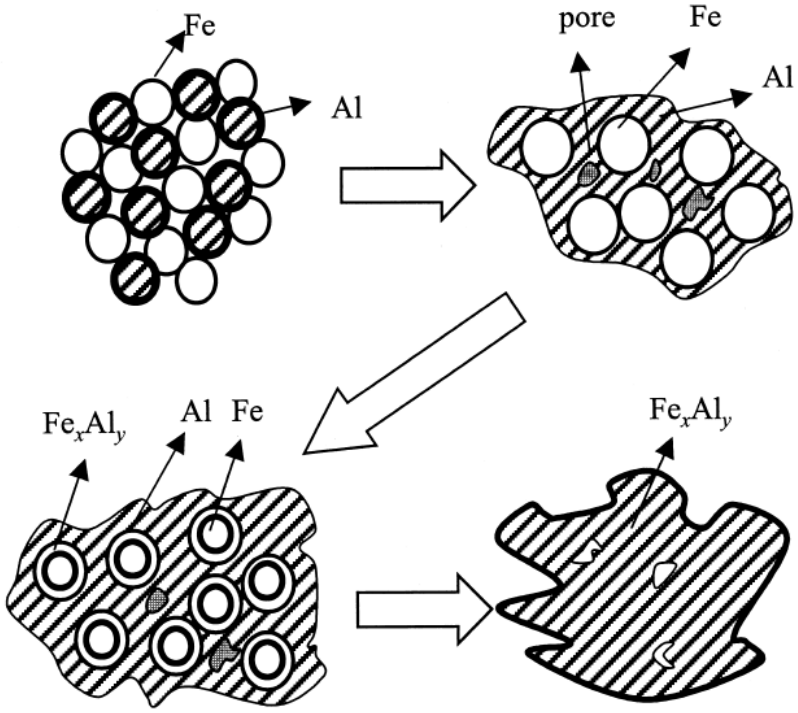


Fig 8. The formation mechanism of FeAl intermetallic [9]

Table 5. The density, hardness, and the amount of the oxidation for the samples containing the particle size of 10/10 and 50/10 of aluminum and iron, compressed under the 500 MPa pressure, pre-heated 550ºC, and heated at 950ºC

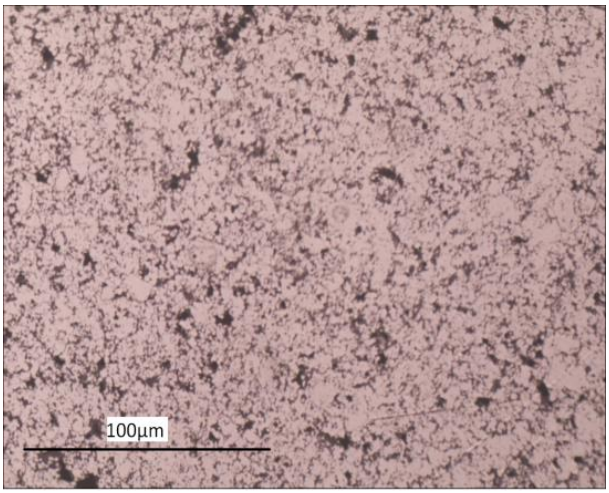
Al Particle size (µm)	Fe Particle size (µm)	Pre-heating Temperature (°C)	Compacting Pressure (MPa)	Synthesis Temperature (°C)	(Apparent Density/ real density*)×100	Vickers Hardness	Oxidation (g/cm^2) After 48h
10	10	550	500	950	95.3%	495	0.011
50	10	550	500	950	89.2%	472	0.016

* The real density of FeAl is 6.06 g/cm^2 [3]

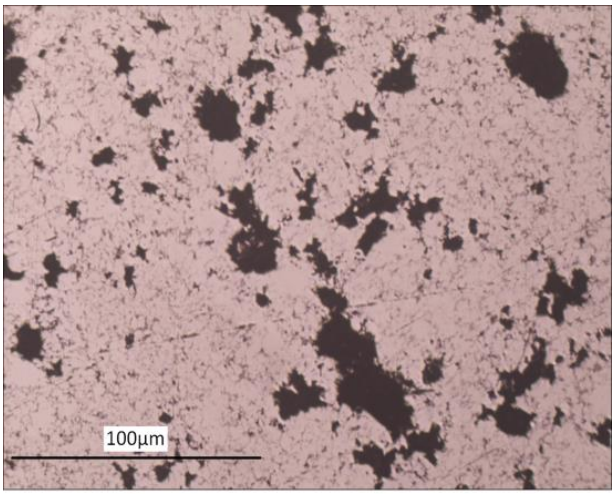
950ºC are shown in Fig. 7.

Based on the obtained product appearance and the previous discussion, two samples were obtained as the optimized products. The samples contain the particle size of 10/10 and 50/10 of aluminum and iron, compressed under the 500 MPa pressure, pre-heated 550ºC and heated at 950ºC for 5h. The finer size particle of Fe leads to a better product. Since the FeAl formation process is a diffusion process, by forming the layer of FexAly between the aluminum and iron particles, aluminum should diffuse through the iron aluminide layer to precede the following reaction. The kinetic of the aluminum diffusion is much less than the kinetic of aluminum and iron [8,9]. Thus the Fe size plays an essential role after the formation of the FexAly layer. The smaller the size of the iron particles causes the shorter the aluminum diffusion path. Thus the less Fe particle size increases the rate of the FeAl formation. Fig. 8 shows the formation mechanism of FeAl intermetallic, schematically.

To determine the most optimal sample, the optical micrograph, density, resistance to the oxidation, and the products' hardness were considered. Checking the factors of density, hardness, and oxidation is necessary to control the applicability and the quality



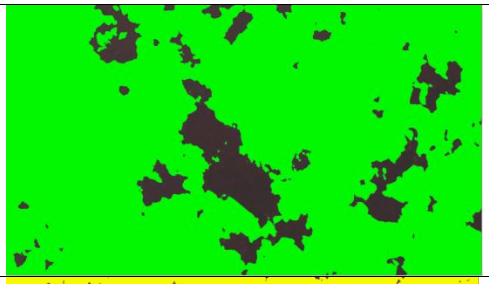
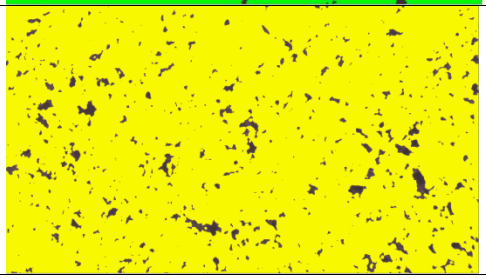
the particle size of 10/10



the particle size of 50/10

Fig 9. The optical micrographs of the samples containing the particle size of 10/10 and 50/10 of aluminum and iron compressed under the 500 MPa pressure, pre-heated at 550ºC and heated at 950 ºC

Table 6. The selected parts to calculate the area percent using the Clemex software

Al Particle size (µm)	Fe Particle size (µm)	Pre-heating Temperature (°C)	Compacting Pressure (MPa)	Synthesis Temperature (°C)	The selected parts to calculate the area percent using the Clemex software	The calculated area percent (%)
50	10	550	500	950		91.09%
10	10					97.19%

of FeAl. These factors are reported in Table 5.

The samples' optical micrographs containing the particle size of 10/10 and 50/10 of aluminum and iron compressed under the 500MPa pressure, pre-heated at 550ºC and heated at 950ºC are shown in Fig. 9. The density as the area percent was also calculated using Clemex software (Table 6).

An optical microscope equipped with an image analyzer system was used to calculate the volumetric percentage of porosities.

The density calculated by the area percent measurement is a

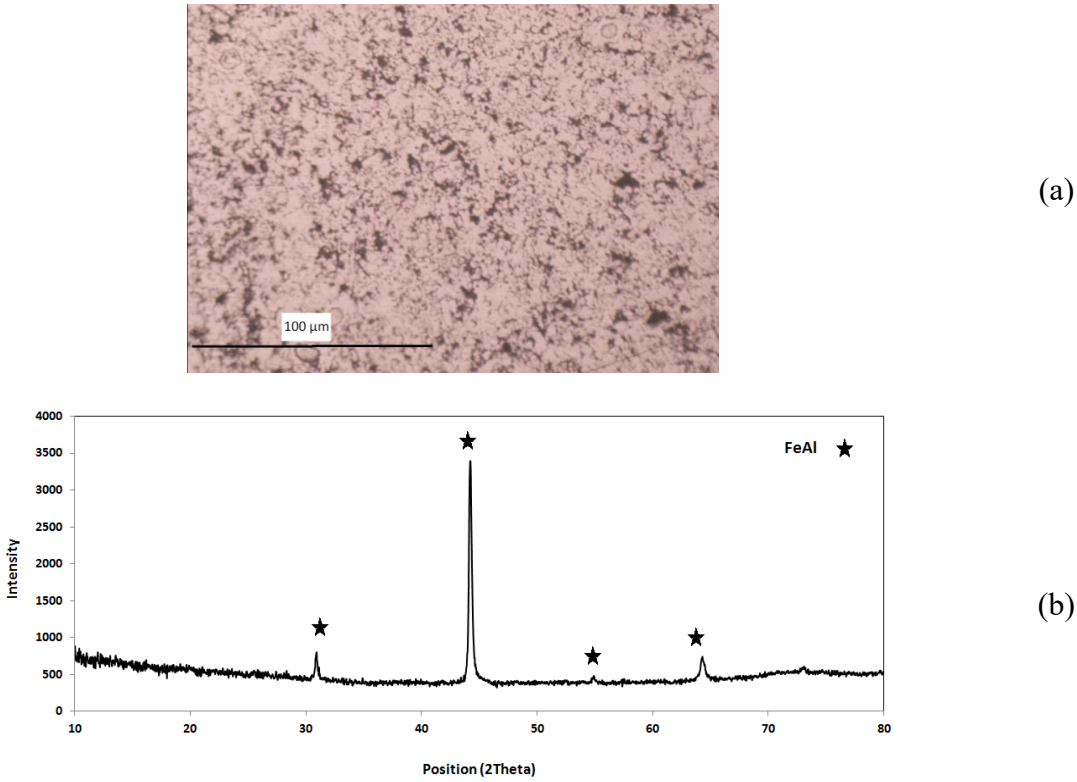


Fig 10. The optical micrograph (a) and the XRD result (b) of the sample containing the particle size of 10/10 of aluminum and iron compressed under the 500 MPa pressure, pre-heated at 550ºC and heated at 950ºC for 3h

little more than the calculated density based on the weight and the volume. This is due to the elimination of the grain boundary in the area percent measurement method.

The presence of the alumina in the sample containing the particle size of 10/10 leads to more hardness and more oxidation resistance than the sample containing the particle size of 50/10.

Although alumina's presence improves the oxidation resistance and increases the hardness of produced FeAl, if needed to remove the alumina, the heating time at 950ºC should be reduced from 5h to 3h. The XRD result and the optical micrograph of the sample containing the particle size of 10/10 of aluminum and iron compressed under the 500 MPa pressure, pre-heated at 550ºC and heated at 950 ºC for 3h (shorter time than 5h, before the initiation of the oxidation of FeAl) are shown in Fig. 10.

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