



The Formation Mechanism of Iron Aluminide Phases in Fe-Al System with Different Raw Materials Ratio

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ABSTRACT: In this study, the formation mechanism of iron aluminide phases in the Fe-Al system with different raw material ratios was investigated. In aluminide systems, full consumption of aluminium or its presence have a major impact on the reaction process, the type of products and the mechanism. However these have not studied and only a defined stoichiometric ratio has been established. Therefore, the current study objective is to determine the effect of raw materials proportion on the reaction mechanism. To achieve this goal, the samples with ratios of 1:3, 1:1 and 3:1 of iron and aluminum were heat treated at 700, 800 and 900°C. It was found that the first phase formed is Fe₂Al₅. It also proved that the following reaction trend depends on the primary content of iron and aluminum. In the ratio of 3:1 of iron and aluminum, the system tends to achieve the Fe₃Al phase at higher temperatures while in the ratio of 1:1 and 1:3, FeAl is the final product. It also was found that more Al postpones the FeAl formation. In the other words, FeAl can be produced at the lower temperature in the ratio of 1:1 of iron and aluminum in the comparison with the ratio of 1:3.

Review History:

Received: 2019-07-13

Revised: 2019-10-03

Accepted: 2019-11-05

Available Online: 2019-12-02

Keywords:

Iron aluminides

Mechanism

Sintering

Phase stability

1. INTRODUCTION

Because of the attractive physical and mechanical properties, the iron aluminide compounds have attracted enormous attention. Among these properties are high temperature applications, good oxidation and sulfidation resistance, low density and low production cost [1-4]. These advantages are utilized in many different ways, such as brake disks, filtration, transfer and ethylene crackers and air deflectors [3]. Iron aluminide intermetallics suffer from low room-temperature ductility [3,5] which makes it difficult to be produced by conventional melting processes. To solve this problem, several measures have been taken one of which is utilizing the sintering and the technology of powder metallurgy of elemental powders. By the use of this method, the iron aluminides would be produced at a temperature considerably lower than the temperature in which iron and the iron aluminide phases melts [6-8]. There are some researches in which the characterization of iron aluminides and the reaction mechanism of the iron aluminide formation from the elemental powders sintering have been investigated [1, 9-15]. Gedevarishvili and Deevi [10] have investigated the sintering of the Fe and Al powders with a definite ratio (Fe-40%Al) at various heating rates between 1000–1350°C. It was found that at 600°C, Fe₂Al₅ forms. FeAl produces at 700°C and the production rate increases in a way that at 1000°C, FeAl is the main phase in the system. It was also found that the heating rate has a great impact on the mechanism of FeAl formation and its density. Chojnacki et al. [8] have explored the ratio of

34% Al-Fe with the small additions of Mg and MgH₂. It has been cleared that at first, FeAl₃ forms then Fe₂Al₅, FeAl₂ and FeAl are the phases produced in the system respectively. Gao et al. [9] have used the Fe:Al=2:3 reactive synthesis. It has been justified that FeAl is formed when the temperature is between 700 to 1000°C. Józwiak et al. [12] by using the equal molar ratio of iron and aluminum (Fe:Al=1:1) believe that compound formation happens as follows: FeAl₃- Fe₂Al₅-FeAl₂-FeAl. Józwiak et al. [12] have detected the FeAl formation at 900°C in the primary ratio of 1:1. Matysik et al. [5] also have used the equal ratio of iron and aluminum and investigated the kinetics of iron and aluminum powder mixture sintering in a non-isothermal way. The iron-aluminum solid solution formation has been detected in the first step. After that, Fe₂Al₅ forms. Then FeAl₂ produces and transforms to FeAl by increasing the temperature. Chojnack et al. [8] have achieved the same results. Novák et al. [6] believe that even before the aluminum is FeAl₂ and Fe₂Al₅ can be produced. When the temperature grows, the amount of FeAl₂ and Fe₂Al₅ definitely increases. The final phases would be Fe₃Al and FeAl. In the following, Fe₃Al has changed to FeAl. Pochec et al. [14] expressed that Fe₃Al is formed at a temperature between 400 and 500°C. At the interval of 570–580°C, FeAl₃, Fe₂Al₅ and FeAl₂ are formed. Higher temperature leads to FeAl formation. Sina et al. [11] have investigated the Fe-40at.% Al powder mixture sintering in different condition namely various reactants particle size, compaction of the powder mixtures and the heating rate.

Despite extensive researches have been conducted due to the use of a definite ratio there is no any comprehensive

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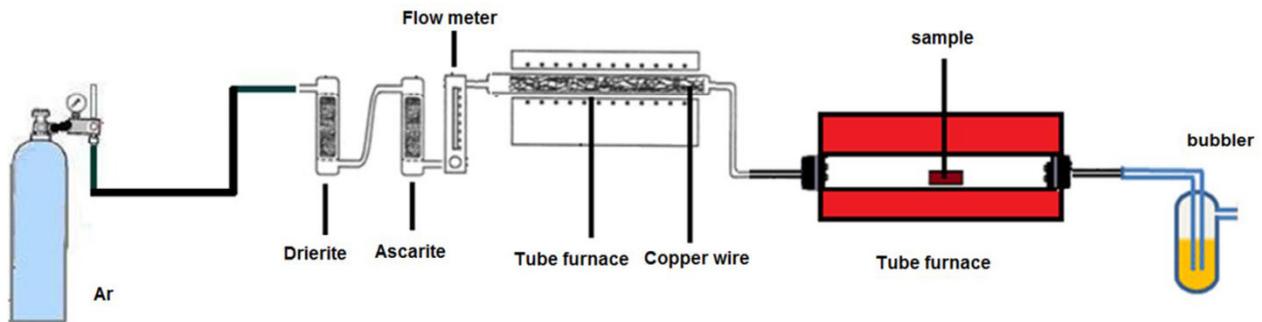


Fig. 1. The applied setup to prevent the oxidation.

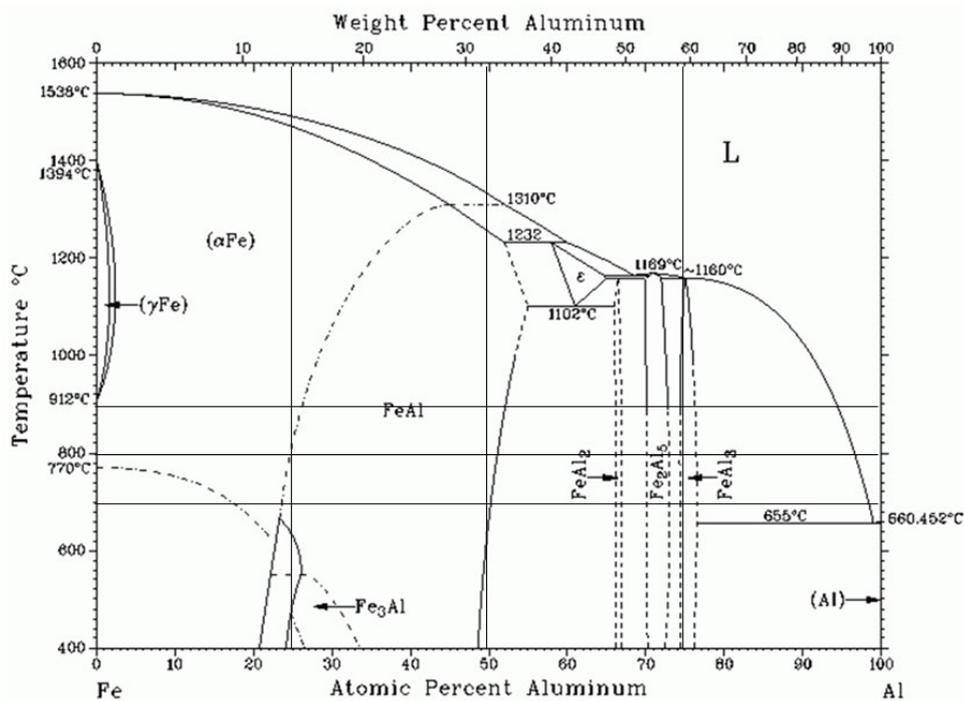


Fig. 2. Binary phase diagram of Fe and Al [16].

study available to specify the effect of primary molar ratio on the mechanism of the foresaid system. In aluminide systems, the presence or full consumption of aluminum have a major impact on the reaction process, the type of products and the mechanism. In addition, there is no agreement on the first phase of the system in each ratio and the final product. To clarify these issues in the study, three molar ratios of iron and aluminum (1:3, 1:1 and 3:1) were investigated to determine the effect of raw materials proportion on the reaction mechanism.

2. EXPERIMENTAL PROCEDURE

The molar ratios of 1:3, 1:1 and 3:1 of iron (99.5%, <math><10\mu\text{m}</math>, from Merck company) and aluminum powders (90 %, <math><10\mu\text{m}</math>, from Merck company) were used in this study. A fast mill at 400 rpm with an alumina jar for 5 minutes was applied in order to mix the powders. The alumina balls had 2 cm diameter. The ball to powder ratio was (5:1). Next, a 1.5 cm diameter pellet was prepared including 2 g of the mixed powder. Subsequently, 700, 800 and 900°C were used for heat treating the samples for 5 h. The setup shown in Fig. 1 was

utilized to prevent the oxidation. To eliminate any O_2 , CO_2 and H_2O present in the argon gas the heated Cu at 550°C, Ascarite and Drierite have been applied, respectively.

The X-Ray Diffraction (XRD) technique (PHILIPS model PW 1800 machine with Cu $\text{K}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$)) was used for the phase analysis of the products under a voltage of 40 kV and the current of 30 mA, respectively.

3. THE RESULTS AND DISCUSSION

To investigate the reaction mechanism of Fe-Al system, three molar ratios of 1:3, 1:1 and 3:1 of iron and aluminum powder were selected. The pressed samples heat treated for 5 h at 700, 800 and 900°C.

Fig. 2 shows the binary phase diagram of Fe and Al. Accordingly, if samples with the foresaid ratios cool from a liquid state, it will be anticipated to achieve the stable phases presented in Table 1.

To be studied which phases would be formed as the equilibrium phase in the Fe-Al powder system, three molar ratios of 1:3, 1:1 and 3:1 of iron and aluminum have been

Table 1. Predicted equilibrium phases in the Fe-Al system with the molar ratios of 3:1, 1:1 and 1:3 based on the Fe/Al binary phase diagram.

	Fe:Al=3:1	Fe:Al=1:1	Fe:Al=1:3	Temperature (°C)
Predicted Phases	FeAl	FeAl	FeAl ₃	700
	Fe or FeAl	FeAl	FeAl ₃	800
	Fe or FeAl	FeAl	FeAl ₃	900

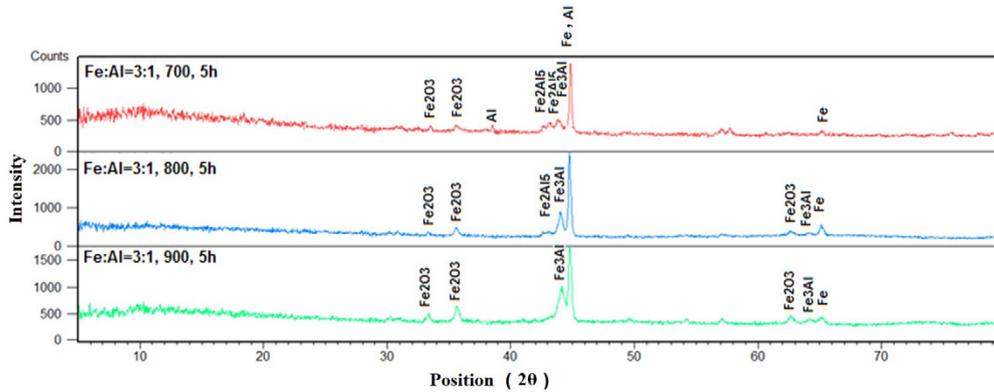


Fig. 3. XRD results for the samples heat treated for 5 h at 700, 800 and 900°C containing the molar ratio of 3:1 of iron and aluminum.

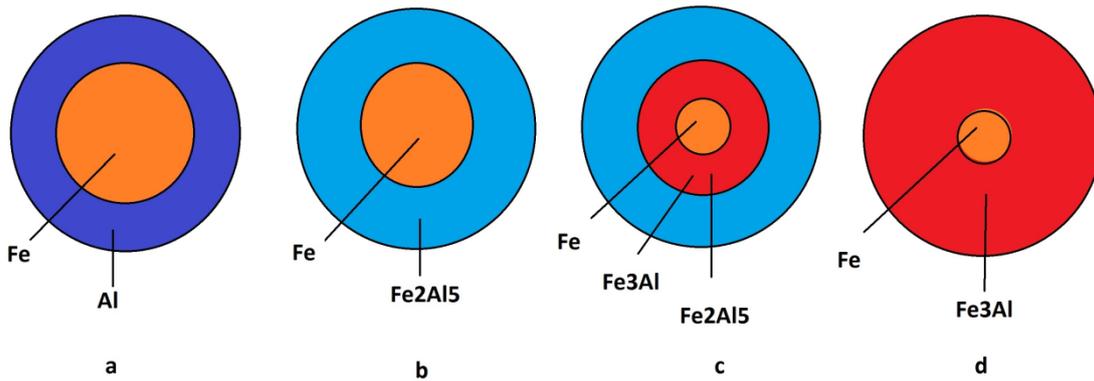


Fig. 4. Schematic of the proposed mechanism in the ratio of 3:1 of iron and aluminum.

considered.

3-1- The result of the samples heat treated at 700, 800 and 900°C, containing the molar ratio of 3:1 of iron and aluminum.

Fig. 3 shows the XRD results for the samples heat treated for 5 h at 700, 800 and 900°C containing the molar ratio of 3:1 of iron and aluminum. Accordingly, at 700°C the sample contains Fe, Fe₂O₃, Fe₂Al₅, Al and Fe₃Al. It is not clear exactly which iron aluminide phase forms before another, because Fe₂Al₅ and Fe₃Al formed simultaneously. Since with increasing the temperature, Fe₂Al₅ disappears gently and Fe₃Al formation increases, it seems that Fe₂Al₅ is the first phase formed in this ratio. Also, there are some Fe₂O₃ which is not detected in the other ratios.

It probably relates to the iron aluminide formation reaction rate. If the aluminide formation progress was intense in this ratio, most of the iron particles surface would be covered with the aluminide. Since aluminides are the

compounds with the oxidation resistance ability, the iron particles could be protected against the oxidation by a layer of iron aluminide. Based on this justification, it can conclude that at this ratio, the iron aluminide formation was not fast and intense enough, thereupon iron oxidation is observed more than the other ratios. Remaining of raw materials after 5 h at 700°C can also confirm this claim.

By increasing the temperature, the peak intensity of Fe₂Al₅ reduces and its sharp mode is changed to a hill form at 800°C while the Fe₃Al sharp peaks form at this temperature. It is necessary to mention that aluminum content has been finished in this temperature. Probably, Fe₃Al has been produced as the result of the reaction between Fe₂Al₅ and iron.

Finally, at 900°C, Fe, Fe₂O₃ and Fe₃Al phases remained as the stable phases while Fe₂Al₅ disappeared completely.

Fig. 4 illustrates the proposed mechanism of the reactions in the ratio of 3:1 of iron and aluminum. As can be seen, Fe₂Al₅ is the first phase forms based on the following reaction:

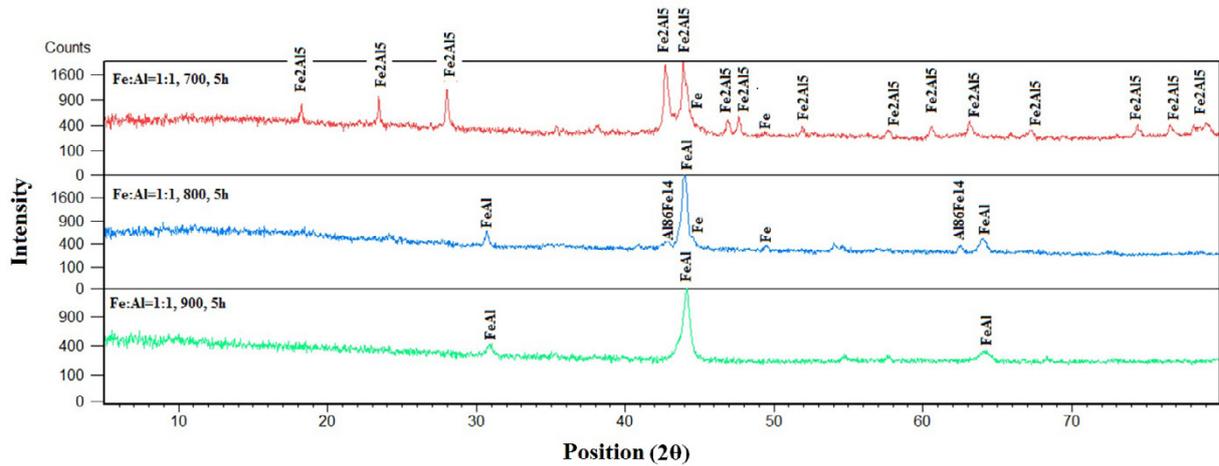


Fig. 5. XRD results for the samples heat treated for 5 h at 700, 800 and 900°C containing the molar ratio of 1:1 of iron and aluminum

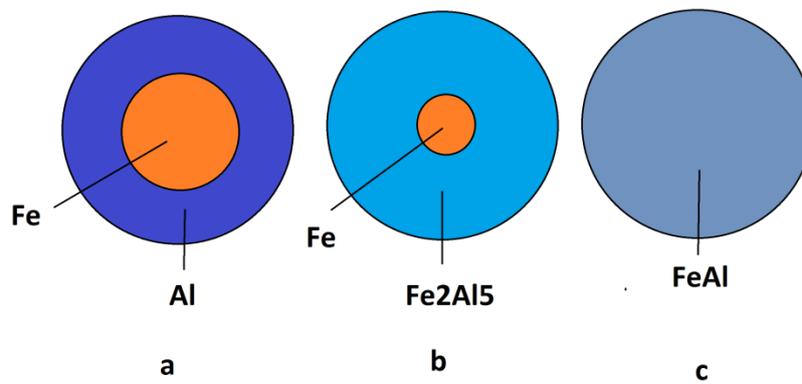


Fig. 6. Schematic trend of the reactions in Fe:Al=1:1 ratio.



Since the Fe content of the raw materials is more than needed for the formation of Fe_2Al_5 , (Eq. (1)) some Fe remains unchanged. In the following, unchanged Fe and produced Fe_2Al_5 react (Eq. (2)):



Because of the iron wasting as the result of the oxidation, all of the raw materials have not been consumed. Accordingly, there should be a small amount of Fe_2Al_5 in the final product that is not detectable by the XRD analysis. Accordingly, there should be a small amount of Fe_2Al_5 in the final product that is detectable in a very small quantity in XRD analysis result at 42° and 44° (Fig. 3 at 900°C).

3-2- The results of the samples heat treated at 700, 800 and 900°C for 5 h containing the molar ratio of 1:1 of iron and aluminum.

As can be seen in Fig. 5 which shows the XRD results of the samples heat treated for 5 h at 700, 800 and 900°C containing the molar ratio of 1:1 of iron and aluminum, at 700°C the sample contains Fe_2Al_5 , some Fe and no remained aluminum. At 800°C , the Fe_2Al_5 transformed to the FeAl while lots of the

Fe_2Al_5 disappeared. Finally, the FeAl is the major phase at 900°C . It seems that by increasing the temperature up to the 800°C , the Fe_2Al_5 react with the remained Fe to produce the FeAl (Eq. (3)).



It seems that the first phase in the Fe-Al powder system is Fe_2Al_5 . Gedevanishvili et al. [10] and Novák et al. [6] believe that Fe_2Al_5 forms even before the aluminum melting and changes to other phase in higher temperatures. Based on the previous discussions, the proposed mechanism of the reactions in the ratio of 1:1 of iron and aluminum is illustrated in Fig. 6. As can be seen, the formation of Fe_2Al_5 occurs by the reaction of Fe and the molten Al (Eq. (1)). Some Fe remains unchanged the amount of which is less than the first step of the previous ratio. In the following, remained Fe reacts with Fe_2Al_5 to form the FeAl phase (Eq. (3)): This mechanism was discussed by details in the previous publication [17].

Identifying the compounds created and the phases transformed at the ratios of 3:1 and 1:1 of iron and aluminum, the following trend detected:

At the ratio of 3:1: $\text{Fe, Al} \rightarrow \text{Fe}_2\text{Al}_5 \rightarrow \text{Fe}_3\text{Al}$

At the ratio of 1:1: $\text{Fe, Al} \rightarrow \text{Fe}_2\text{Al}_5 \rightarrow \text{FeAl}$

13 moles of Fe is needed to produce the 5 moles of Fe_3Al

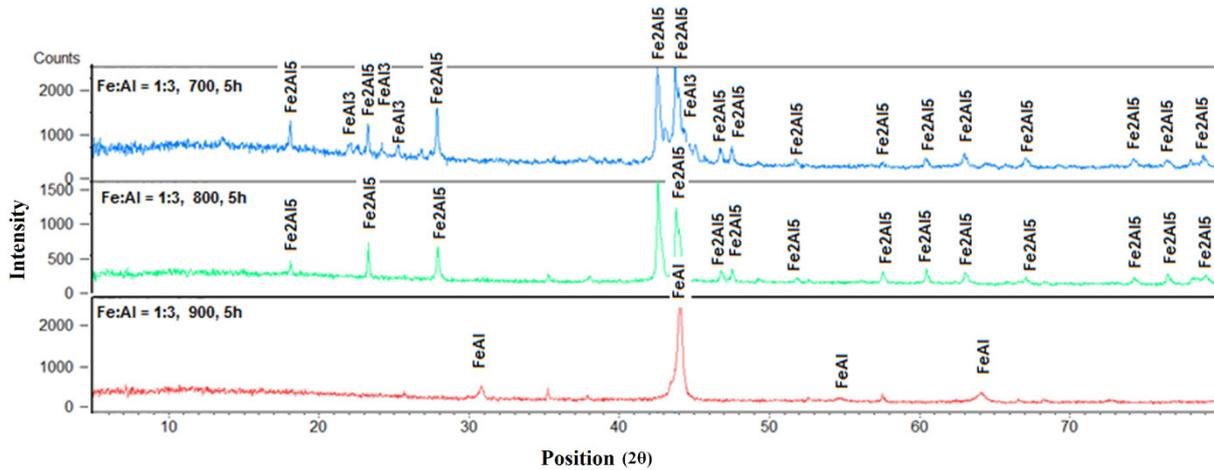


Fig. 7. XRD result for the samples heat treated for 5 h at 700, 800 and 900°C containing the molar ratio of 1:3 of iron and aluminum.

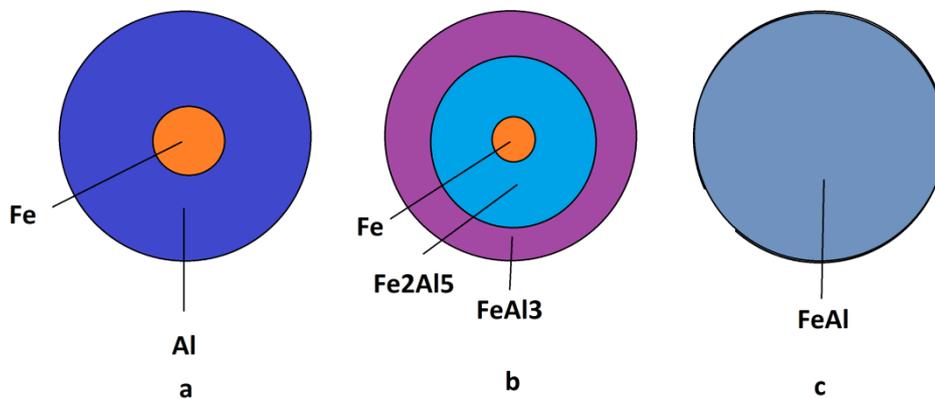


Fig. 8. Schematic trend of the reactions in Fe:Al=1:3 ratio.

(Eq. (1)) while just 3 moles of Fe is needed for the production of the 5 moles of FeAl (Eq. (2)). Accordingly, it is expected that FeAl be detected as a transient phase in the ratio of 3:1 but it does not happen.

Since the target final product in the ratio of 3:1 is Fe₃Al, if FeAl forms in the first step, it will be necessary for FeAl to react with Fe afterwards. Hence, the amount of energy needed to form FeAl is more than the energy needed for the Fe₃Al direct formation; system prefers that FeAl not to be formed.

As a result of the previous discussion, the first phase in the Fe-Al system is Fe₂Al₅.

Since most researches have used the equal ratio of iron and aluminum, the results of this section can be compared with their findings. In some researches [6, 12, 14] FeAl₂ has been detected as the transient phase which has not detected in this study. There is not any agreement on the FeAl phase formation temperature. For example, Gedevarishvili et al. [10] and Gao et al. [9] have reported that the formation of FeAl occurs at 700°C while Józwiak et al. [12] have shown that FeAl forms at 900°C. This research has proven that the temperature in which FeAl can be formed depends on the raw material ratio.

Novák et al. [6] have reported the Fe₃Al as the latest phase before the FeAl formation while Pochec et al. [14] stated that

Fe₃Al forms between 400 and 500°C and changes to other phases at elevated temperatures. In this study, it has been observed that Fe₃Al is appeared just in the ratio of 3:1 of iron and aluminum.

In fact, the final product depends on the primary molar ratio of the raw material. It also depends on the speed of the reactions, which can be determined by some of factors such as the particle size of the elemental powders and the heating rate.

3-3- The results of the samples heat treated at 700, 800 and 900°C for 5 h containing the molar ratio of 1:3 of iron and aluminum.

Fig. 7 shows the XRD results of the samples heat treated at 700, 800 and 900°C for 5 h containing the molar ratio of 1:3 of iron and aluminum. As can be seen at 700°C, FeAl₃ and Fe₂Al₅ form. By increasing the temperature to 800°C, the dominant phase is Fe₂Al₅. The notable point is that FeAl₃ is detected here which never appeared in the previous ratios.

Fig. 8 shows the proposed mechanism of the reactions occurs in a sample containing the ratio of 1:3 of iron and aluminum. As can be seen, besides the Fe₂Al₅ formation, FeAl₃ is formed. Chojnacki et al. [8] have also reported FeAl₃ as the first phase in the system with Fe-34%Al of course in the presence of Mg and MgH₂. Józwiak et al. [12] by using the

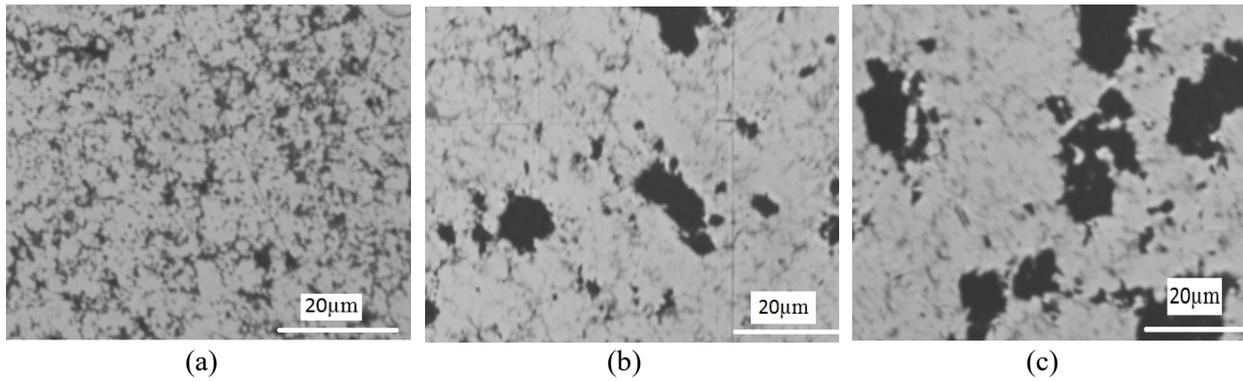


Fig. 9. OM images of the samples with the molar ratios of a) 3:1, b) 1:1 and c) 1:3 of iron and aluminum heat treated for 5 h 900°C.

Table 2. Thermodynamic data of intermetallic phases in the Fe–Al system reported by Ivanov [18].

Iron aluminide	$(933-1500\text{ K})\Delta G^\circ \left(\frac{\text{cal}}{\text{mole}}\right)$	calculated ΔG° at 973 K	calculated ΔG° at 1073 K	at 1173 calculated ΔG° K
Fe ₃ Al	-16160+21.68T	4934.64	7102.64	9270.64
FeAl	-24210+11.81T	-12718.9	-11537.9	-10356.9
FeAl ₂	-33680+15.29T	-18802.8	-17273.8	-15744.8
Fe ₂ Al ₅	-45290+19.97T	-25859.2	-23862.2	-21865.2
FeAl ₃	-49060+21.78T	-27868.1	-25690.1	-23512.1

equal molar ratio of iron and aluminum (Fe:Al=1:1) believe that the order of compound formation is as follow: FeAl₃-Fe₂Al₅-FeAl₂-FeAl. In other words, they have also identified the first production phase as FeAl₃. The reason is the increasing in impact of aluminum compared to the previous stoichiometric molar ratios. This is due to its higher content or because of additives that have lowered the melting temperature. The difference in the result of current molar ratio with the previous one can be found justified by the amount of available molten aluminum. By increasing the Fe₂Al₅ thickness, the iron diffusion is limited; therefore, the condition is suitable for the FeAl₃ formation. When Al content finishes, Fe reacts with FeAl₃ at the first step and secondly, with Fe₂Al₅ to form the FeAl (Eqs. (3) and (4)).



Fig. 9 shows the Optical Microscope (OM) images of the samples with the molar ratios of a) 3:1, b) 1:1 and c) 1:3 of iron and aluminum heat treated for 5 h at 900°C. It is clear that with increasing the aluminium content, there are more porosity detectable on the surface. Melting and consuming the aluminium content lead to the porosity generation. The uniform structure of the samples with molar ratio of b) 1:1 and c) 1:3 of iron and aluminum heat treated for 5h at 900°C was predictable based on the results of XRD (Figs. 3, 5 and 7).

Limited thermodynamic data is available to predict the iron aluminide phase stability. Among the few, the standard

Table 3. Thermodynamic data of intermetallic phases in the Fe–Al binary system reported by Richard [19]

Intermetallic compound	$\Delta G^\circ 700^\circ\text{C} \text{ (J/mol)}$ [19]
FeAl ₃ (θ)	-22869
Fe ₂ Al ₅ (η)	-19636
FeAl ₂ (ζ)	-16999
FeAl($\beta 2$)	-11090
Fe ₃ Al($\beta 1$)	-4827

Gibbs free energy formation of iron aluminides has reported in 2 references which contradict each other in some parts. Tables 2 and 3 show the standard Gibbs free energy formation of iron aluminides which reported by Ivanov and Richards [18,19]. As can be seen, the standard Gibbs free energy formation of Fe₃Al at 700°C (973 K) is positive in Table 2 while it is negative in Table 3. According to the study results, the standard Gibbs free energy formation cannot be positive because it forms at 700°C and it remains stable at 800°C.

Although there are some contradictions in these two reports, there are also some agreements. For example, in both reports, FeAl₃ has the lowest standard Gibbs free energy formation at 700°C. Nevertheless, why this phase has not

Table 4. Final products detected in the Fe-Al system with the molar ratio of 3:1, 1:1 and 1:3 heat treated at 700, 800 and 900°C for 5 h.

	Fe:Al=3:1	Fe:Al=1:1	Fe:Al=1:3	Temperature (°C)
Predicted Phases	Fe, Al, Fe ₂ Al ₅ , Fe ₃ Al, Fe ₂ O ₃	Fe ₃ Al	FeAl ₃ , Fe ₂ Al ₅	700
	Fe, Fe ₂ Al ₅ , Fe ₃ Al, Fe ₂ O ₃	Fe ₂ Al ₅	Fe ₂ Al ₅	800
	Fe, Fe ₃ Al, Fe ₂ O ₃	FeAl	FeAl	900

been detected in the ratios of 3:1 and 1:1. This study confirms that the amount of available elemental powder of Fe and Al is effective in the reaction process. Therefore, it is due to enough amount of aluminum that is not provided for the ratios of 1:1 and 3:1 of iron and aluminum, the FeAl₃ does not form.

Another issue that must be discussed here is the stable aluminide phase type at the higher temperatures. FeAl and Fe₃Al have the structure of CsCl (B2) [19] and (D03) [19] respectively which B2 structure is the ideal for the ratio of 1:1 of iron and aluminum and D03 is ideal structure for the ratio of 3:1. Because both of them are symmetric structures, the system prefers to achieve these phases. It also is proved that despite of thermodynamic data, FeAl₃ is not more stable than FeAl at the temperature of 800°C and 900°C because even though the formation of FeAl₃ at the lower temperature (700°C) occurs, it disappears at elevated temperatures (800°C and 900°C).

The compounds which detected as the stable phase of the powder system with different ratios at different temperatures are summarized in Table 4. It can conclude that stable phase of Tables 4 and 1 are not the same; it probably is the result of the kinetic limitation for diffusion and reaction in the solid state.

4. CONCLUSION

The mechanism of the reactions in the iron - aluminum powder system was investigated with different raw material ratios. It was found that the first phase formed in the system is Fe₂Al₅. The following reactions were also influenced by the raw material ratios. If Fe content is enough, system prefers to form the Fe₃Al in higher temperatures. In the equal proportion of iron and aluminum and in the ratio of 3:1, FeAl will be preferred phase at the higher temperatures. It also was found that FeAl can be produced at the lower temperature in the ratio of 1:1 of iron and aluminum in the comparison with the ratio of 3:1. It means more Al postpones the FeAl formation.

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HOW TO CITE THIS ARTICLE

R. Khoshhal, A. Hosseinzadeh, *The Formation Mechanism of Iron Aluminide Phases in Fe-Al System with Different Raw Materials Ratio*, *AUT J. Mech Eng.*, 4(3) (2020) 407-414.

DOI: [10.22060/ajme.2019.16739.5831](https://doi.org/10.22060/ajme.2019.16739.5831)



