Copper's Influence on Reactions in the Fe–Al Equimolar System: Microstructure and Phase Development

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Abstract

Due to their suitable properties, iron aluminides have found numerous applications in various industries such as in the production of gas turbines, automotive combustion systems, corrosion-resistant coatings, heat exchanges, electrical and magnetic devices, and energy storage technologies. The aim of this research is to investigate the effect of copper content on the phase formation path and microstructure of products resulting from reactions that can be carried out in the Fe-Al-Cu powder system. For this purpose, samples with different copper contents (*0, 0.2, 0.32* and *0.4* atom percent) and equimolar Fe and Al were prepared from elemental powders. The powders were compressed into disk shapes after mixing. Then, they were sintered at *950*°C for 24 hours. The type of phases produced was determined using X-ray diffraction (XRD) analysis and the microstructure of the samples was determined using a scanning electron microscope (SEM) equipped with (EDX). The results showed that the type of phases produced changed depending on the amount of copper. The sample without copper only led to the formation of the FeAl phase. The addition of *0.2*% copper also resulted in the formation of the $CuAl_2$ phase alongside the iron aluminide. Further increase to *0.32*% copper led to the formation of the ω phase and at *0.4*% copper, the I phase. Also, the amount of copper affected the amount of porosity produced in the samples.

Keywords: Iron aluminide, Copper, Mechanism, Morphology, Phases.

Introduction

In the Fe-Al binary system [1], there are two types of solid solutions with the FCC structure called γ and the BCC structure called α. The α phase is formed both in a disordered form (A2) and in an ordered structure (B2 and D03). The conversion of A2 to B2 occurs at 600°C. Furthermore, it can be formed in the Fe-Al binary system. The $FeAl_2$ phase has a triclinic structure (symbol ν), the Fe_2Al_5 phase has an orthoclinic structure (symbol μ), and the FeAl3 or Fe4Al13 phase has a monoclinic structure (symbol λ). Studies have shown that various types of iron aluminide compounds can be obtained by varying the molar ratio of iron and aluminum [2-5].

Cu-Al alloy belongs to the shape memory alloy category [6]. Shape memory alloys are used in many applications, including civil structural control [7], dental wires, mending broken bones, and medical devices [8]. Many compounds form in the binary system of aluminum and copper, including the tetragonal $CuAl_2$(θ), phase which is better known than other types of compounds for its influence on the age-hardening of aluminum alloys. The remaining phase is CuAl(η), which crystallizes in a monoclinic structure, forms at low temperatures, and transforms into an orthorhombic structure at high temperatures. The ζ phase forms at temperatures below *590*°C with a copper ratio from *55.2* to *59.8*% [9]. The ε phase of the Cu_3Al_2 composition is stable at temperatures above *560*°C. The properties of Fe-Cu-Al-based shape memory alloys are believed to be strongly influenced by aluminum content [6].

Changing the molar ratio of the raw material also affects the properties. It has been reported that in the Al-Cu-Fe ternary system, increasing the amount of aluminum raises the surface tension, and increasing the amount of iron reduces the surface tension [10]. Copper can increase the corrosion resistance of $FeAl_3$. This improvement may be due to the formation of a copper-rich surface layer, which will have a higher protective effect than the intermetallic layer [11]. Akinribide et al. [12] prove that Copper has beneficial effects on the properties of aluminum alloys. Copper may also help to enhance the hardness and tensile strength of aluminum alloys in certain quantities [13, 14]. Shabestari et al. [15] showed that the best mechanical properties were obtained with about *1.5* wt.% copper in Al-Si-Mg alloys. Copper has also been employed as a sintering aid. It has been claimed that Copper can improve the porosity and capillary properties of aluminum [16].

Al65Cu20Fe15 is reported to have the highest corrosion resistance and highest hardness [17]. The Cu-Fe binary system has no intermediate phase [6]. Miscibility is also evident in the liquid state. Copper forms a eutectic with aluminum, which can facilitate aluminide-forming reactions that begin with aluminum melting [2, 4, 18-20].

It was also found that during the friction welding process, a discontinuous reaction layer of $FeAl_3$ composition is formed between aluminum and iron, which does not occur between aluminum and copper [21]. Various compounds have been reported in the Fe-Al-Cu ternary system. The Cu_3Al and $AlFe_3$, Al_3Fe, $AlTi_2$, and $Al_{65}Cu_{20}Fe_{15}$ phases were prepared using a magnetron sputtering process by applying a coating containing a specific proportion of the Fe-Al-Cu system and annealing the coating at *700* °C for *2* h [22]. The presence of this layer on the Ti-6Al-4V substrate improves its corrosion resistance [15]. Among the phases formed through the interaction of Fe, Al, and Cu are the I-phase and ω-phase. The I-phase is an intermetallic compound within the iron–aluminum–copper system that exhibits quasi-crystalline characteristics, with properties that can be modified through heat treatment [23]. Quasicrystals (QCs) are intermetallic structures distinguished by their non-periodic atomic arrangement. Specifically, the I-phase displays a unique five-fold symmetry that defies conventional crystallographic principles [24, 25]. This phase has been reported across various compositional ranges, such as Al64Cu24Fe12 to Al61.75Cu25.5Fe12.75, although its composition and thermal stability are known to vary with temperature [26], and reported ranges differ among studies [27-29]. The ω-phase, on the other hand, possesses a hexagonal crystal structure and typically forms in systems with body-centered cubic (bcc) arrangements [30]. It is considered a metastable phase, emerging under conditions such as rapid cooling, elevated pressure, or low temperatures [31, 32]. This phase is more likely to form in compositions with high aluminum content [33] and is characterized by its extreme hardness and brittleness [34]. The ω-phase is generally represented by the composition Al_7Cu_2Fe [35-37], while the I-phase corresponds to $FeCu_2Al_6$ [38]. Chromium and nickel are also added to the Fe-Al-Cu ternary system. It was found that chromium alloys provide the greatest thermal stability, while the addition of nickel forms a homogeneous alloy structure [39]. The quasicrystalline phase Al60Cu30Fe10 was produced by mechanical alloying and spark plasma sintering. Other phases such as Al_7Cu_2Fe, FeAl, Fe4Al13, and $CuAl_2$ are also found to be formed [40]. Barua et. al [41] studied different ratios of Al70−x(Cu, Fe)30+x (x=*0*, *5*, *7*) by a mechanical alloying method. They found that increasing x reduces the tendency to form the i phase. They also reported that the β-phase cubic solid solution is one of the major phases in all the above alloy compositions. This study also addresses the formation of the $CuAl_2$ phase. Silicon has also been an additive that has been able to create desirable properties in iron aluminides [42, 43].

Based on the present study the influence of copper on the formation of various phases, the synthesis pathway of the iron aluminide phase, and the resulting microstructural characteristics were investigated.

Experimental Method

Elemental powders of iron (Merch, *10* µm, *99.5*%), aluminum (Chempur, *-100*, *+325* mesh, *99.95*%), and copper (Pourian-Chemical, *99*%) were used in this study. First, iron and aluminum were mixed in an equal molar ratio. Then, depending on the amount of aluminum present in each sample, three samples containing *0.2*, *0.32*, and *0.4*% copper were prepared based on the aluminum-copper phase diagram. After mixing the powder, disk samples weighing *2* g were taken from each sample using a mold with a diameter of *1* cm and a pressure of 2.41×10^{6} Pa.

According to the outlined procedure, the molar proportions of each element in the samples are presented in Table 1.

Table 1: Molar percentages of Fe, Al, and Cu for each sample.

Sample	%Fe	%Al	%Cu
0	**50**	**50**	**0**
0.2	**45.45**	**45.45**	**9**
0.32	**43.1**	**43.1**	**13.8**
0.4	**41.66**	**41.66**	**16.68**

Copper-free samples were also used as witness samples. The samples were then synthesized in a tube furnace at a temperature of *950*°C for *24* h. he argon atmosphere was passed through an Ascarite and Drierite media, and the furnace contained copper at a temperature of *550*°C, all of which are used to remove CO_2, H_2O, and O_2 from the argon atmosphere, respectively.

XRD analysis (Bruker, D8 Advance) with CuKα radiation (λ = *1.54* Å) was used to determine the type of phase formed in the samples. The data were collected employing a step size of *0.02°* and a time per step of *2* seconds.

SEM (KYKY, EM8000) was used to determine the structure of the product. The stages of the experiment are schematically shown in Fig. 1.

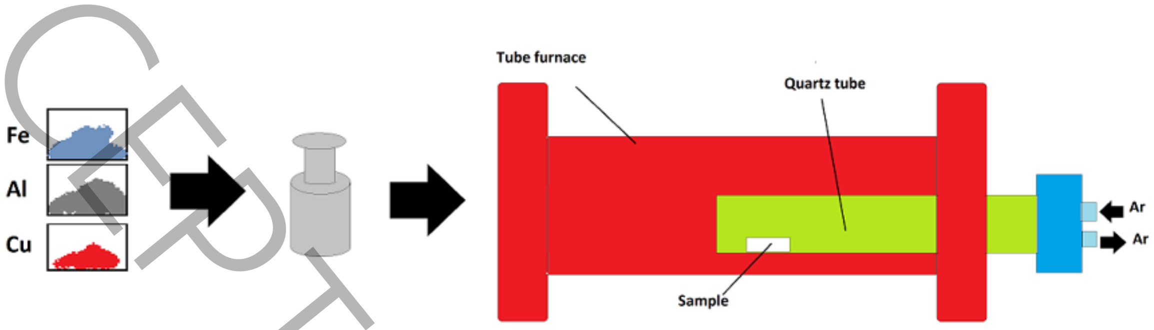


Fig. 1. Sample preparation steps and used set-up.

Results and discussion

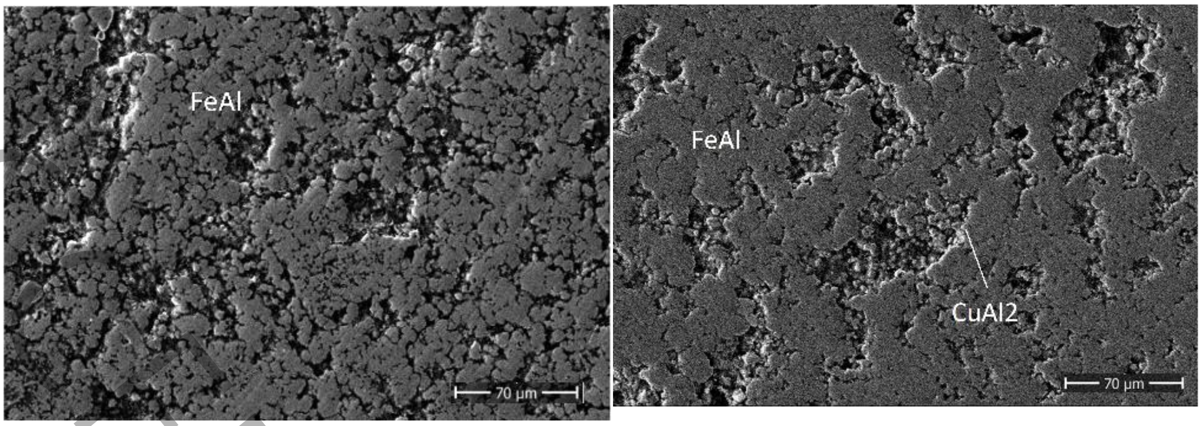
In the binary phase diagram of aluminum and copper, an eutectic transition is observed at *32.7%* Cu [44]. Therefore, an attempt was made to study three ratios from the hypo-eutectic, eutectic, and hyper-eutectic regions. Accordingly, the mole percentages of each element (Fe, Al, and Cu) in samples were shown in Table 1.

Fig. 2 presents electron microscopy (SEM) images of the samples along with EDX analysis results for the identified compounds. In the sample without copper, only the FeAl phase is observed. Upon introducing *0.2%* copper, the $CuAl_2$ phase emerges, predominantly located at the particle boundaries. As the copper content increases to *0.32%*, the ω phase begins to form. Finally, at *0.4%* copper, the I phase is also detected among the resulting products.

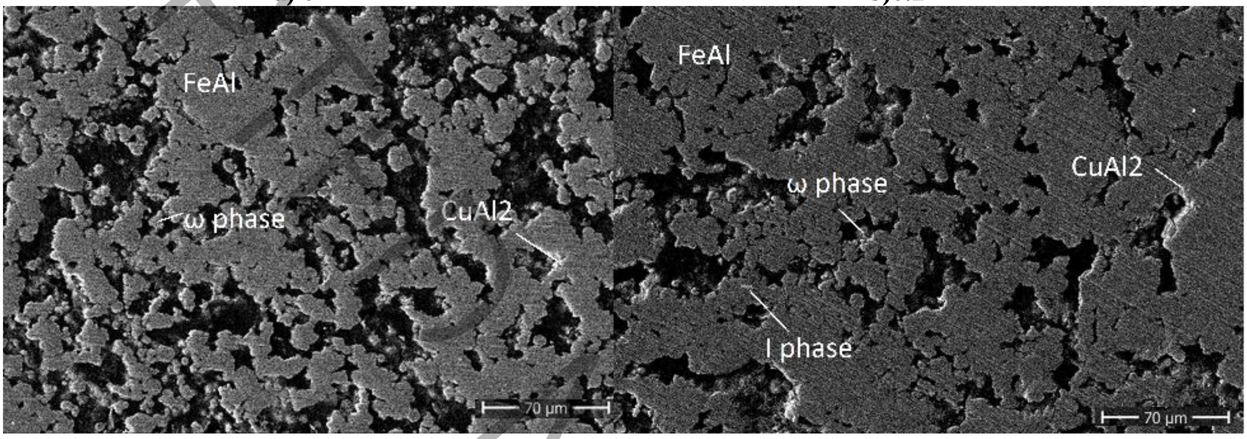
The porosity percentages were quantified for each image, revealing void ratios of *28.11%*, *19.01%*, *31.87%*, and *22.15%* for images a, b, c, and d, respectively.

“Imag J” software was used to obtain the porosity. Based on the images, the percentage of areas without pores was calculated. The percentage of the remaining surface area is reported as pores.

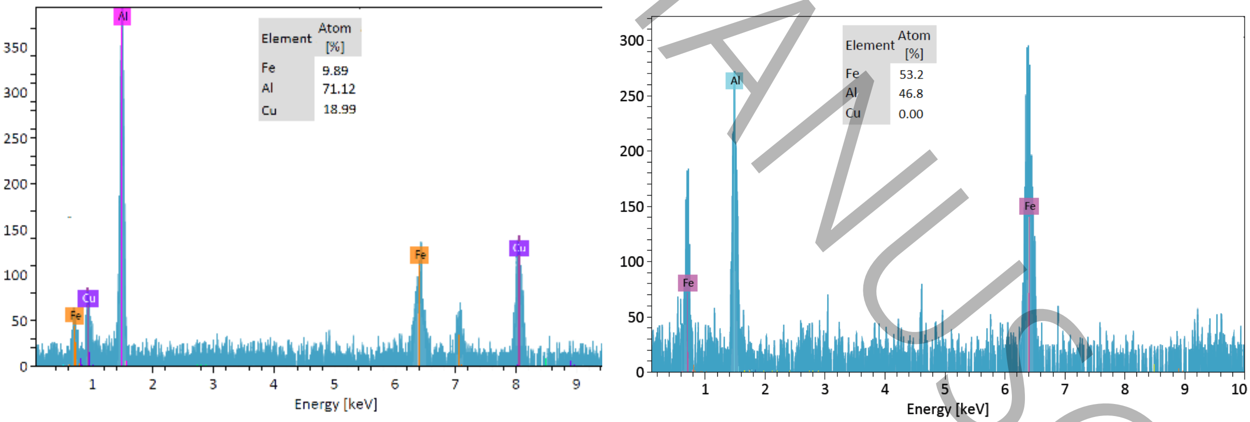
These results indicate that the addition of *0.2%* copper contributed to a favorable structural correlation and led to decreased porosity relative to the copper-free sample. Moreover, the sample with 0.4% copper exhibited a notably more compact and uniform structure.



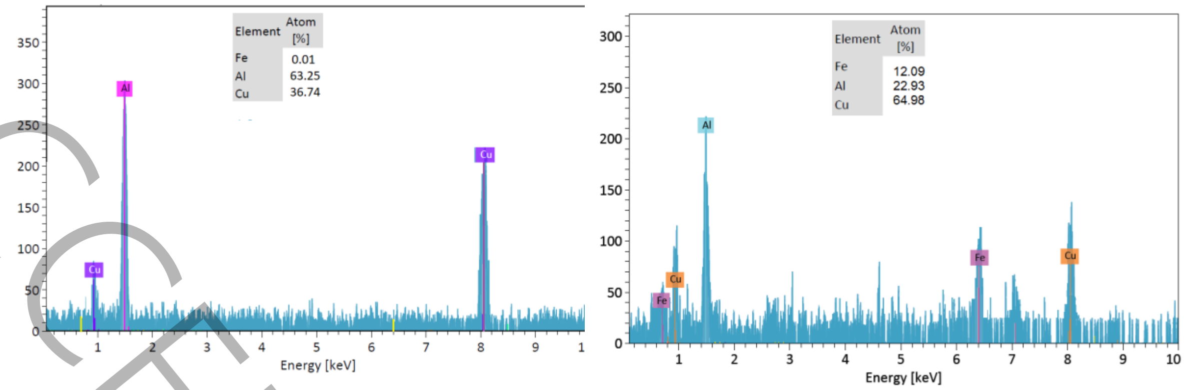
a) 0 b)0.2



c)0.32 d)0.4



e) ω f) FeAl



g)$CuAl_2$ **h) I**

Fig. 2: Electron microscope images (SEM) images of samples with a) *0*%Cu, b)*0.2*% Cu, c)*0.32*%Cu, and d)*0.4*% Cu as well as the results obtained from EDX analysis for the compounds e) ω, f)FeAl, g)$CuAl_2$, and h)I.

Thermodynamic studies were performed on the Fe-Al-Cu ternary systems. The results of one of these studies are presented in Fig. 3. It should be noted that reference [46] derived its information from references [9, 38, 45]. As shown in this figure, increasing the copper content allows the formation of ω and I phase.

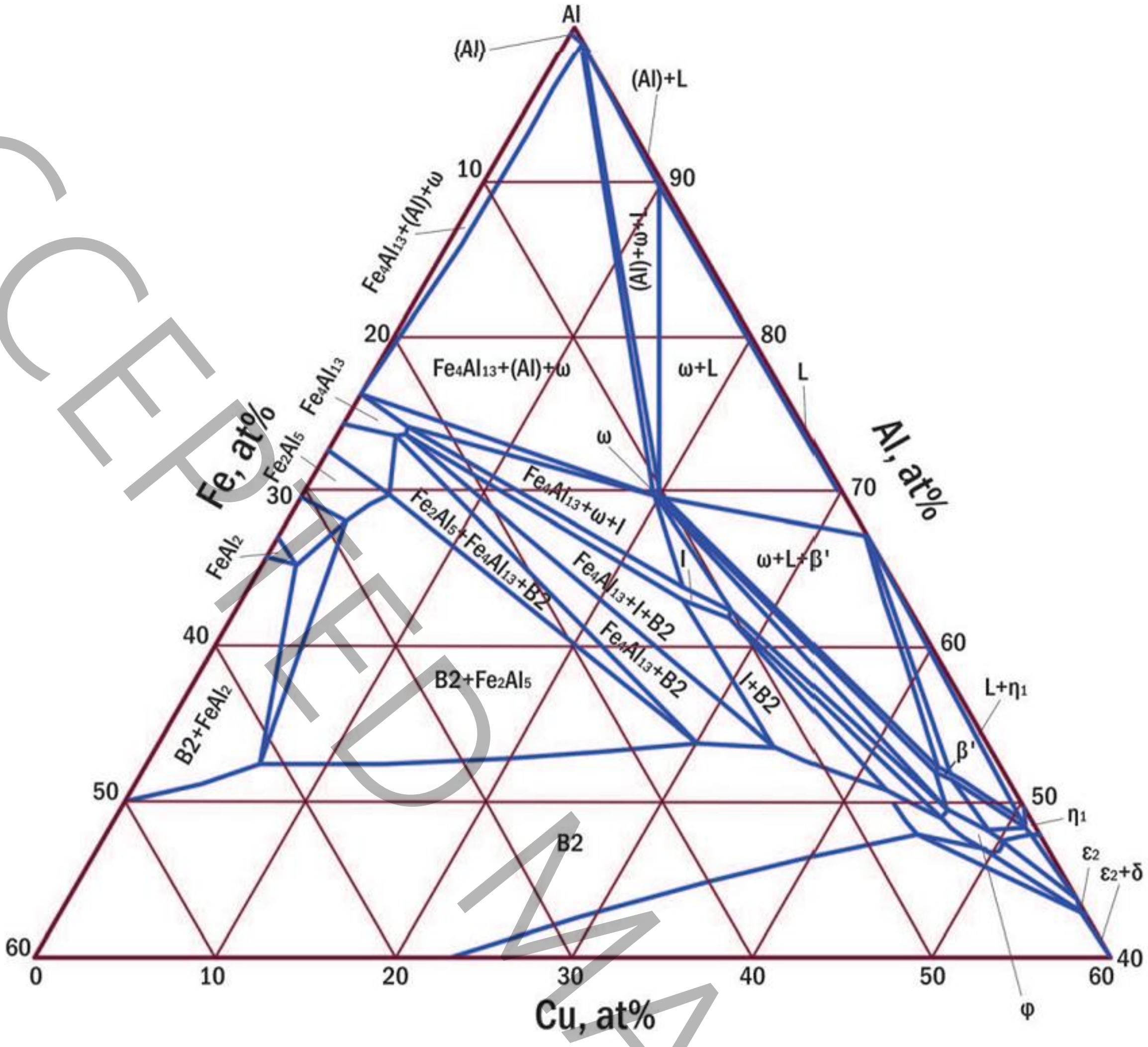


Fig. 3. An isothermal section of the Cu-Fe-Al system at *600*°C was extracted and summarized by Makarenko [46].

According to the predicted graph in Fig. 3, the composition used in this study should yield only the B2 phase. However, experimental results revealed the presence of both I and ω phases. This deviation can be explained by the differences in reaction conditions: the mechanism illustrated in Fig. 3 start from high-temperature processing where all three elements are fully molten, allowing for optimal mixing and contact. In contrast, our study involves solid-state compression of powders. Although molten aluminum forms during intermediate stages, its volume is minimal compared to the fully molten scenario. Consequently, the resulting products tend to be richer in aluminum and poorer in iron, as reflected in Fig. 3. This imbalance arises from aluminum's partial melting and iron's solid nature. Additionally, the variation in processing temperatures may contribute to the formation of different phases.

It has also been found that as the temperature increases, the expansion of the equilibrium region of the β and α phases increases, and the I and ω phases can also appear [47]. The phase of "I" has a triangular structure and may contain $Al_{62.4}Cu_{24.4}Fe_{13.2}$, $Al_{65}Cu_{23}Fe_{12}$, and $Al_{61}Cu_{28.4}Fe_{10.6}$ compounds [48].

The I-phase is a quasicrystalline solid that displays long-range icosahedral orientational order without any periodic translational symmetry. Its microstructure and properties can be tuned through different heat-treatment procedures [23]. As a member of the quasicrystal family, it features a non-repeating, fivefold rotational symmetry that falls outside the scope of classical crystallography [24, 25]. Experimentally, this phase has been reported over compositions from Al64Cu24Fe12 to Al61.75Cu25.5Fe12.75, with its exact stoichiometry and thermal stability shifting as a function of temperature [26]. Various studies note slight variations in these composition ranges[27-29]. Under both ambient and moderately elevated pressures, the I-phase maintains stability between *750*°C and *800*°C [49]. In some reports, its formula is also given as FeCu$_2$Al$_6$ [38].

The ω-phase adopts a hexagonal crystal lattice that typically forms in alloys with an original body-centered cubic (bcc) matrix [30]. It is a metastable structure that can be induced by rapid quenching, application of high pressure, or exposure to low temperatures[31, 32]. It also appears in alloys with high aluminum content [33]. This phase is characterized by very high hardness but limited ductility[34]. It's ideal stoichiometry is often expressed as Al$_7$Cu$_2$Fe [35-37]. Under equilibrium conditions, ω-phase has been observed around *550*°C in alloys containing *50–70* at. % Al and up to ~*9* at. % Fe[30]

With a higher percentage of aluminum [41] than the content used in the present study, it was reported that after 20h of mechanical alloying, both B phase, and Al$_2$Cu phase were observed. When the milling time is increased, Al50Fe50 and B2 types will form. In another study, in addition to the Al$_2$Cu phase, the Al3Fe and I phases were also formed. [50]. The Al$_2$Cu phase appears to be prepared under different conditions and with different stoichiometry. The reason for this may be related to the diffusion rate of copper in aluminum. It has been reported that copper atoms diffuse five times faster in aluminum than aluminum atoms in the copper layer. This allows the production of compositions containing more aluminum [50]. In the current study, the formation of the Al$_2$Cu phase is predicted considering the copper content. The solubility limit of copper in Al at ~*723* K is about *0.15*at% [51], while the maximum solubility of Al in copper at the same temperature is about 18at%. The solubility limit of copper in Al is lower than the solubility of Al in copper, so the solid solution of Al(Cu) is saturated first. Therefore, Al$_2$Cu becomes the first phase at the interface. As the diffusion becomes stronger, the Al$_4$Cu$_9$ phase and AlCu phase may appear.

The production of the CuAl$_2$ phase supports this conclusion. If aluminum were the faster diffuser, only trace amounts of Al would diffuse into the copper lattice, shifting the stoichiometry toward a copper-rich intermetallic. However, the fact that CuAl$_2$—an aluminum-majority compound—forms indicates that copper diffuses more rapidly into the aluminum matrix.

Al$_3$Cu is another potential compound in the Al–Cu system, yet it was not identified among the phases formed in this study. This absence is addressed using Fig. 4, which is adapted from the work of Souissi et al. [52]. In the study of Souissi et al. [52], using thermodynamic calculations, it was shown that the θ-Al2Cu phase is the ground state phase of the Al-Cu structure (Fig. 4).

In mechanical alloying methods, non-equilibrium phases can often be produced due to the application of high energy and reaching high local temperatures. However, the method used in the present study is a constant temperature synthesis method in which the entire assembly is subjected to thermal treatment at a constant temperature and the phases are closer to the thermodynamically predicted phases. In some of the mentioned methods, the phases formed in the liquid phase of the ternary system were also investigated, which could again give different results from the method of this research because in the molten state reaction, particle contact and penetration are much greater than in the solid powder state.

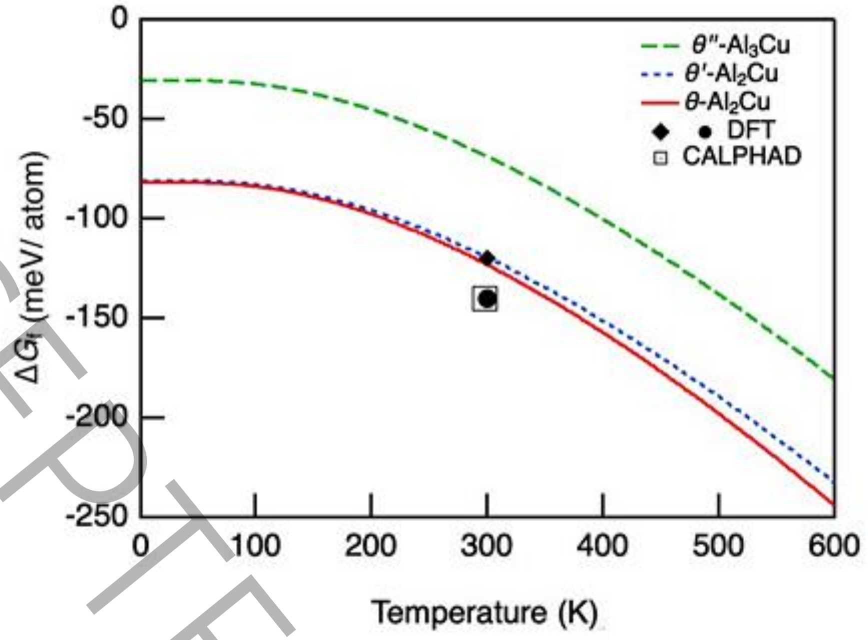


Fig. 4. The standard free energy of formation for aluminum and copper compounds [52].

The type of phases produced in the samples were determined using XRD analysis (Fig. 5). As can be seen in this figure, in the absence of copper, the FeAl phase is formed because the number of moles of iron and aluminum is equal. The Al_2Cu phase is produced by adding a small amount of copper (*0.2*%). Increasing the copper content (*0.32*%) produces the “ω” phase. Finally, when the copper content reaches 0.4%, the “I” phase also appears in XRD analysis.

The reason why the ω phase is formed before the I phase is considered to be because the ω phase has an ordered hexagonal structure. Aluminum, copper, and iron also have a cubic crystal structure, so the ω phase is easier to form than other phases.

Using the Debye–Scherrer method, the crystallite size of FeAl was determined for all four samples. The calculated values for samples with *0, 0.2, 0.32,* and *0.4%* copper were *415, 386, 354,* and *307* Å, respectively. The results indicate that increasing the copper content leads to a reduction in crystallite size, which may be attributed to the lattice strain introduced by the added Cu element.

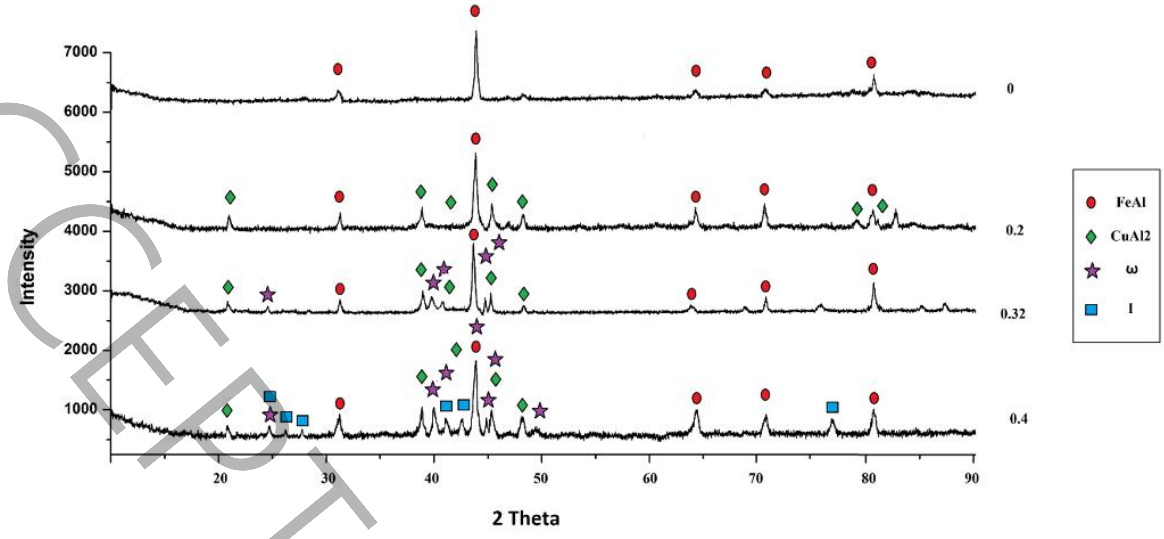


Fig. 5. XRD results of free copper and samples containing *0.2*, *0.32*, and *0.4*% of copper.

Based on the XRD patterns, SEM observations, and EDX analysis, the mechanism illustrated in Fig. 6 appears to provide a general representation of the reactions that took place and the resulting products. When copper is introduced into the iron–aluminum system in small amounts (*0.2*), the Al_2Cu phase is formed. Owing to its stability and the abundance of aluminum, this phase readily develops. With a higher copper content (*0.32*), the ω phase also emerges within the structure, and at an even greater concentration (*0.4*), the I phase appears in certain regions. Based on the Al_2Cu crystal structure[53], this phase may serve as a favorable site for the nucleation of the FeAl phase. The ω phase can also form due to its hexagonal structure[54] and its close resemblance to the cubic arrangement.

The recognized phases, FeAl, $CuAl_2$, the ω-phase (Al_7Cu_2Fe), and the I-phase ($Al_{64.3}Cu_{23.5}Fe_{12.2}$) detected using ICDD PDF card numbers of 03-065-0985, 00-002-1309, 03-065-1685, and 00-049-1729, respectively.

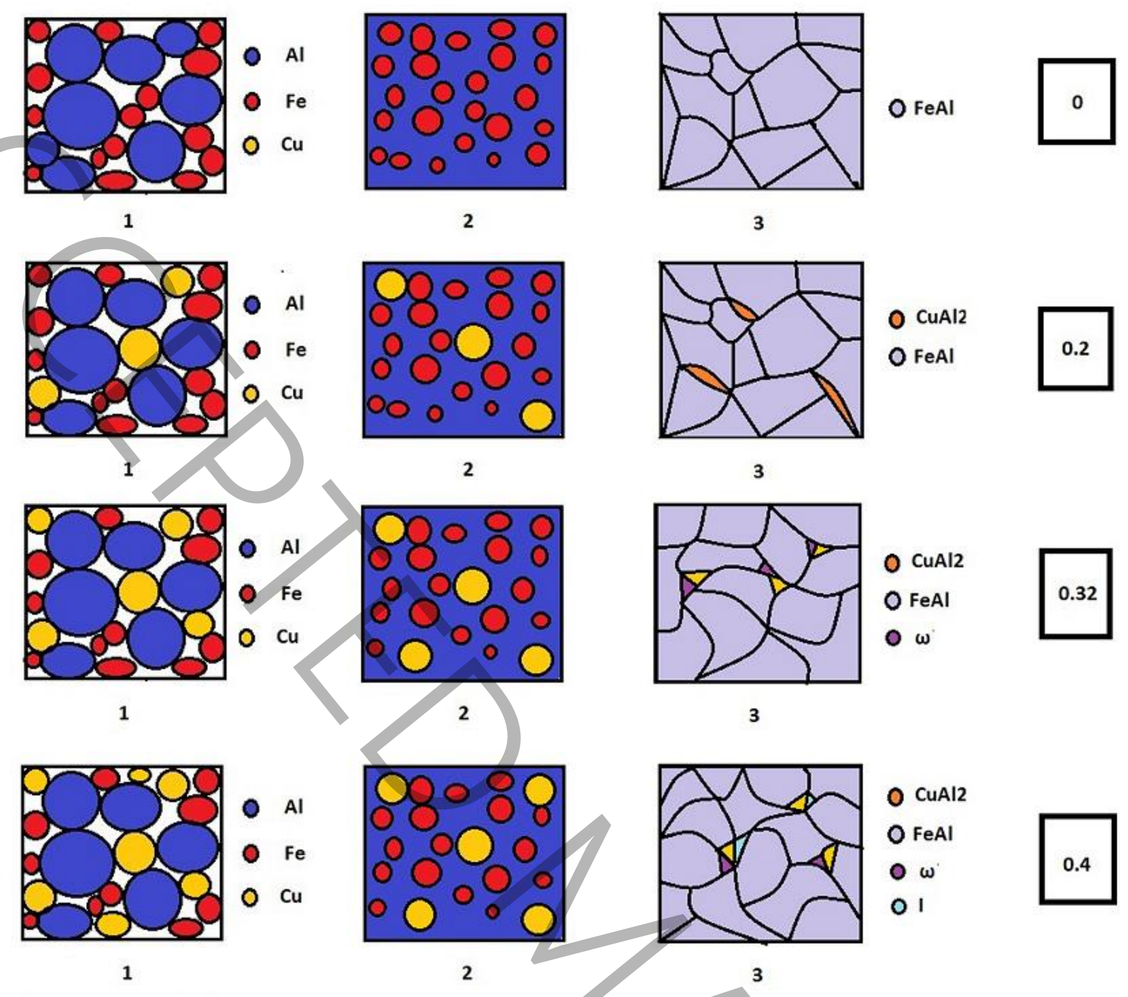


Fig. 6. Schematic representation of products obtained from copper-free samples and samples containing different amounts of copper (*0.2*, *0.32*, and *0.4*).

Conclusion

In this study, the effect of copper on the formation of iron aluminide from an equimolar powder mixture of iron and aluminum was determined. The results showed that the presence of copper changes the type and path of phase formation and microstructure in the equimolar Fe-Al system. In the absence of copper, the only phase that could be produced under these conditions was the FeAl phase. Based on the results of this research, adding *0.2* mol% copper causes the formation of a $CuAl_2$ phase in addition to the dominant FeAl phase. Increasing the copper content to *0.32*% results in the production of a quasi-stable ω phase with a hexagonal structure and high hardness. When the copper content reaches *0.4*%, phase I, which has non-periodic symmetry, is formed. As the results of this study showed, copper can affect the type of phases that can be produced from Fe and Al elemental powders. Specifically, the sample containing *0.2*% copper showed lower porosity compared to the copper-free sample. However, with increasing the copper content, the porosity increased. This issue highlights the importance of determining the optimal amount of copper to achieve a microstructure with minimal porosity.

There are also changes in the crystallite size that are likely due to lattice strains caused by the addition of copper.

In the future, different ratios and temperatures can be used to study the same system. Research programs can also be defined on modifying the microstructure and mechanical properties of this system.

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