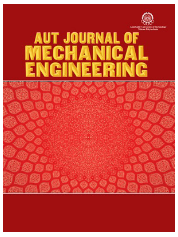

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The use of copper as an accelerator for the formation of titanium aluminide intermetallic compounds from TiO_2 and Al

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ABSTRACT: Titanium aluminides have received much attention due to their suitable properties such as lightness, maintaining strength at high temperatures, and resistance to corrosion and oxidation. Therefore, titanium aluminides and their composite play a key role in the industry, especially in urban transportation and aviation industries. The melting of aluminum is recognized as the first step of the titanium aluminide formation process from elemental powders or raw materials of TiO_2 and Al. Facilitating the aluminum smelting could be a contributing factor to accelerating the titanium aluminide-generating procedure. Selecting copper as an agent to reduce the melting point of aluminum is based on the binary phase diagram of Al-Cu in which a eutectic transformation is obvious. Different molar ratios of copper were added to the raw materials of aluminum and titania. Then the resulting compressed powder samples were subjected to heat treatment. It was found that up to a certain molar ratio, copper could reduce aluminum's melting temperature and promote the formation of titanium aluminide intermetallic compounds. The optimal amount of copper (0.2) has also greatly contributed to the uniformity and homogeneity of the composite structure. In general, in this research, the effect of copper on the production of titanium aluminide has been discussed both theoretically and practically.

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1- Introduction

Titanium aluminides have good practical properties such as high melting point and relatively low density. These compounds have maintained their strength at high temperatures and have shown good resistance to creep and corrosion. Therefore, they have received special attention in many engineering applications, including aerospace, automotive, and energy production industries [1, 2]. Titanium aluminide intermetallic compounds and their composites are also used for hypersonic structural applications [3], and high speed civil transport [4]. Although these types of materials have shown significant potential in high-temperature applications, their high production costs limit their use. The use of powder metallurgy can create a cost-effective production technique by minimizing the machining cost [5]. In addition to the above properties, titanium aluminides also have resistance to oxidation at temperatures above *500*°C [6].

These beneficial properties have made these alloys able to compete with nickel-based superalloys. Since titanium aluminides are brittle and their machining is difficult, actions are being taken to make these materials in near-net shape to reduce their production costs [1, 7]. Titanium aluminides are produced in different methods, among which the following can be referred to.Yumoto *et al*. [8] have placed the titanium aluminide layer on a surface by the supersonic free-jet PVD (SFJ-PVD) method from aluminum and titanium elemental powders. This technique consists of two stages, evaporation and deposition. Twin-wire plasma is another technique that has been used to produce this category of materials[9]. A considerable literature has grown up around reaction sintering and the quality improvement of the product to create titanium aluminides [10-12]. As an instance, Liu et al. [5] have applied different grinding methods to reduce the size of titanium aluminide particles. The speed and quality of sintering have improved. The raw materials of TiO_2 and Al powders have also been utilized to manufacture the titanium aluminide-alumina composites [13-15]. Due to similar properties, the composite of titanium aluminides-Al_2O_3 is also being applied for aerospace applications [16-19].

Since aluminum melting is the first step in the titanium aluminide production process [2, 12, 20-22], it was predicted that if the melting temperature of aluminum can be reduced in any way, the production of titanium aluminide intermetallic compounds would also be accelerated. Based thereon, the copper element, which has a eutectic transformation in the binary equilibrium diagram of Cu-Al, was used. This idea has been explored for the first time in the current study and no research has been found that surveyed this concept. To achieve this goal, different proportions of copper were added to the mixture of TiO_2 and Al powders relying on the binary diagram of copper-aluminum, and the obtained results were analyzed and discussed in detail.

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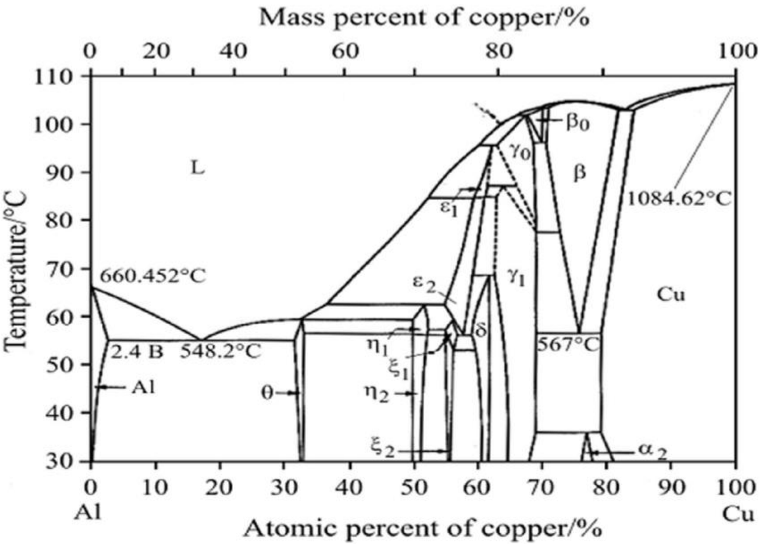


Fig. 1. Aluminum-copper binary diagram [29]

2- Materials and Methods

At first, a mixture with the molar ratios of 1 to 3 of TiO_2 (98%, <45 µm) and Al (99.5%, <45 *µm*) powders were prepared. Then, according to the binary aluminum-copper diagram, different ratios of 0.1, 0.2 and 0.3 to the ratio of aluminum in the system, copper powder (99%, <20 *µm*) was added to the foresaid powders. The achieved powders were mixed. Cylindrical samples of *2* grams with a diameter of 1 cm were prepared from this powder mixture under *300* Psi pressure. Pressing was to create a suitable contact surface and perform better reactions. The obtained samples were heat treated at 900*°C* in pure argon atmosphere. A FESEM microscope (KYKY, EM8000) equipped with EDS was used to survey the microstructure of the samples and the elemental map in each of the samples was also determined. XRD analysis (Bruker, D8 advance) was used to explore the obtained phases. Also, with the help of DSC analysis (Jupiter STA), the critical temperatures of evolutions were determined. This analysis was performed under an argon atmosphere at a rate of *20*°C/min in an alumina crucible.

3- Results and discussion

In the binary equilibrium diagram of Cu-Al, on the left side of the diagram (Fig. 1), the eutectic transformation can be observed, which has caused a decrease in the melting temperature of aluminum. The minimum of this melting point happened around the molar ratio of *0.2* mol of copper and *0.8* mol of aluminum. On the other hand, the melting of aluminum has been determined as the first step of titanium aluminide formation process [23-28].

Based on these two facts, it was predicted that copper may be able to facilitate the production of titanium aluminide intermetallic compounds from TiO_2 and Al raw materials. Therefore, according to the ratio of TiO_2:Al=1:3, which lead to the $TiAl_3$ production, 0.1, 0.2, and 0.3 moles of copper were added according to the molar ratio of aluminum. The aim of this study is to evaluate the effect of copper on the production of titanium aluminide intermetallic compounds.

Since the first goal of the current research was to examine the effect of copper on aluminum melting temperature, a DSC analysis was used to identify the thermal behavior of each sample (containing copper and copper-free). For this purpose, 0.1, 0.2, and 0.3 moles of Cu (according to the aluminum molar ratio) were added to the samples prepared with molar ratios of 1 to 3 of TiO_2 and Al. Then, DSC test was performed on the mixed powders in alumina crucibles under an argon atmosphere with a heating rate of 20*°C/min*.

The results of this analysis are given in Fig. 2. The positive part of the vertical axis determines the heat absorption and the negative part of the axis determines the heat release. As can be seen in this figure, the first peak is an endothermic peak that belongs to the melting of aluminum, and then an exothermic peak occurs, which represents the exothermic reactions of titanium aluminide formation. The blue curve, which does not contain copper and contains only a 1 to 3 molar ratio of TiO_2 and Al, has the highest melting temperature of aluminum, and with the addition of copper to the system, the melting temperature of aluminum has decreased. It also is illustrated that with the addition of copper, the heat content released from titanium aluminide formation (the exothermic peak) increases at 0.1 and 0.2 mol of copper and then decreases while the amount of copper reaches *0.3* moles.

By adding copper, the intensity of the melting peak of aluminum has also declined due to the diminishing proportion of aluminum in the sample. When copper was added (Fig. 2, gray, red, and yellow curves), the melting temperature of aluminum reduced as well. It seems that the exothermic reactions of titanium aluminide production have been carried out more intensively. In general, it can be considered from this graph that copper has been able to reduce the melting temperature of aluminum.

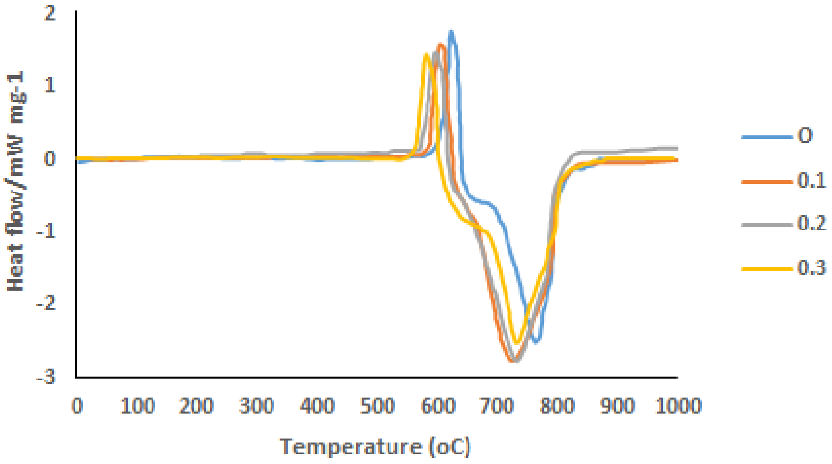


Fig. 2. The result of the DSC test performed on the samples containing 1 to 3 molar ratios of TiO_2 and Al, which have 0, 0.1, 0.2, and 0.3 moles of copper (based on the molar ratio of aluminum)

Table 1. Gibbs standard free energy of formation of TiO2-Al-Cu system compounds.

HSC software							[31]			
Compound	**Ti_3O_5**	**Ti_2O_3**	**TiO**	**Cu_2O**	**CuO**	**TiAl**	**$TiAl_3$**	**Al_2Cu_3**	**Al_3Cu_4**	**AlCu**	**Al_2Cu**
G^o (kJ/mol)Δ	-2820.7	-1725.8	-606.8	-259.5	-228.2	-62.4	-108.1	-18.2	-17.9	-18.6	-13.1

For the thermodynamic investigation of the TiO_2, Al, and Cu system, all possible phases were considered at the temperature of *900*°C which is the temperature applied in the present research (Table 1). As reported in this table, the first phases that can be formed are titanium oxides. It is expected that as the reactions proceed, titanium aluminide intermetallic compounds will also appear. It is worthwhile noting that the system's tendency to form intermetallic compounds between aluminum and copper are low. The thermodynamic data on the formation of Ti-Cu compounds was not available even in the new version of the HSC chemistry software database [30].

Now that the possible phases of the system were checked, HSC Chemistry 5.11 software was used to additionally verify the reactions procedures. To extract the equilibrium composition of this system, it was assumed that one mole of TiO_2 and 0.75 moles of copper (based on *3* moles of aluminum) exist in the system. Based on the binary diagram of copper-aluminum, the extent of copper was calculated according to the ratio of 0.2:0.8 of Cu:Al. Another assumption that was taken into consideration was that *0.142* moles of aluminum were added to the system in *21* steps at the temperature of 900°*C*. Fig. 3 displays the calculated equilibrium phases. As illustrated in Fig. 3, when aluminum and TiO_2 react, titanium oxide phases with a lower percentage of oxygen are produced and subsequently intermetallic compounds are created. As the amount of aluminum increases, more $TiAl_3$ is produced compared to TiAl.

Despite the thermodynamic prediction of the presence and possibility of formation of other titanium aluminide phases(Fig.3), researchers have reported that as long as the aluminum is present in the system, no phase other than $TiAl_3$ is formed [32, 33]. With the completion of available molten aluminum, it is possible to produce other phases such as TiAl or Ti_3Al.

The next step was to consider the experimental samples obtained based on the heat treatment process described in the Experimental section of the current study. Fig. 4, shows SEM images and the elemental maps of Ti, Al, O, and Cu, for the samples containing *1* to *3* molar ratio of TiO_2 and Al without copper, containing 0.1, 0.2, and 0.3 *mol* copper (based on the proportion of aluminum) that have been heated for *3* hours at a temperature of 900°*C*.

The XRD results of the samples were also illustrated in Fig. 5. In general, according to Figs. 4 and 5, it was found that the addition of *0.1* to *0.2* of copper led to the formation of more $TiAl_3$. The format of the produced $TiAl_3$ structure, which has caused many points sputtering in the samples containing 0.1 and 0.2 of copper, is another reason for the higher production rate of the $TiAl_3$ component. The formation of the $TiAl_3$ phase requires an increase in volume and consequently, leads to a rupture in the microstructure. Some research has focused on the same issue. For example, Bohm *et al*. [34] have pointed out that in the first stage of reactions between titanium and molten aluminum, $TiAl_3$ is created. $TiAl_3$ formation causes

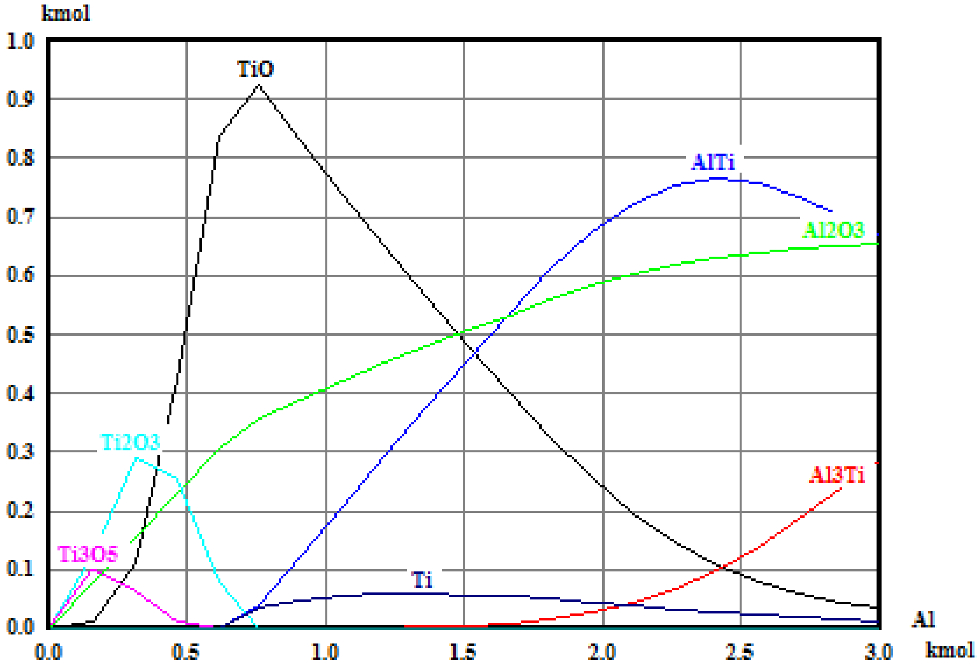


Fig. 3 Prediction of the equilibrium composition of the system containing one mole of TiO2 and 0.75 moles of copper, to which aluminum was added in 21 steps at a temperature of 900°C, 0.142 moles in each step (HSC chemistry software)

an increase in volume and, as a result, swelling of samples. Sina et al. [23] have also reported this rupture that occurs due to the generation of $TiAl_3$, which they named the "spongy structure". They have also stated that if the molar ratio of 1 to 3 of Ti and Al is used, the only phase that can be produced when the sample is heated at 1113 *k* would be $TiAl_3$. It should be noted that they used titanium and aluminum elemental powders. In the present research, TiO_2 was used as a source containing Ti and oxygen. Since aluminum is also present in the raw materials and it intensively tends to oxidize, besides $TiAl_3$, some Al_2O_3 was also observed in the samples.

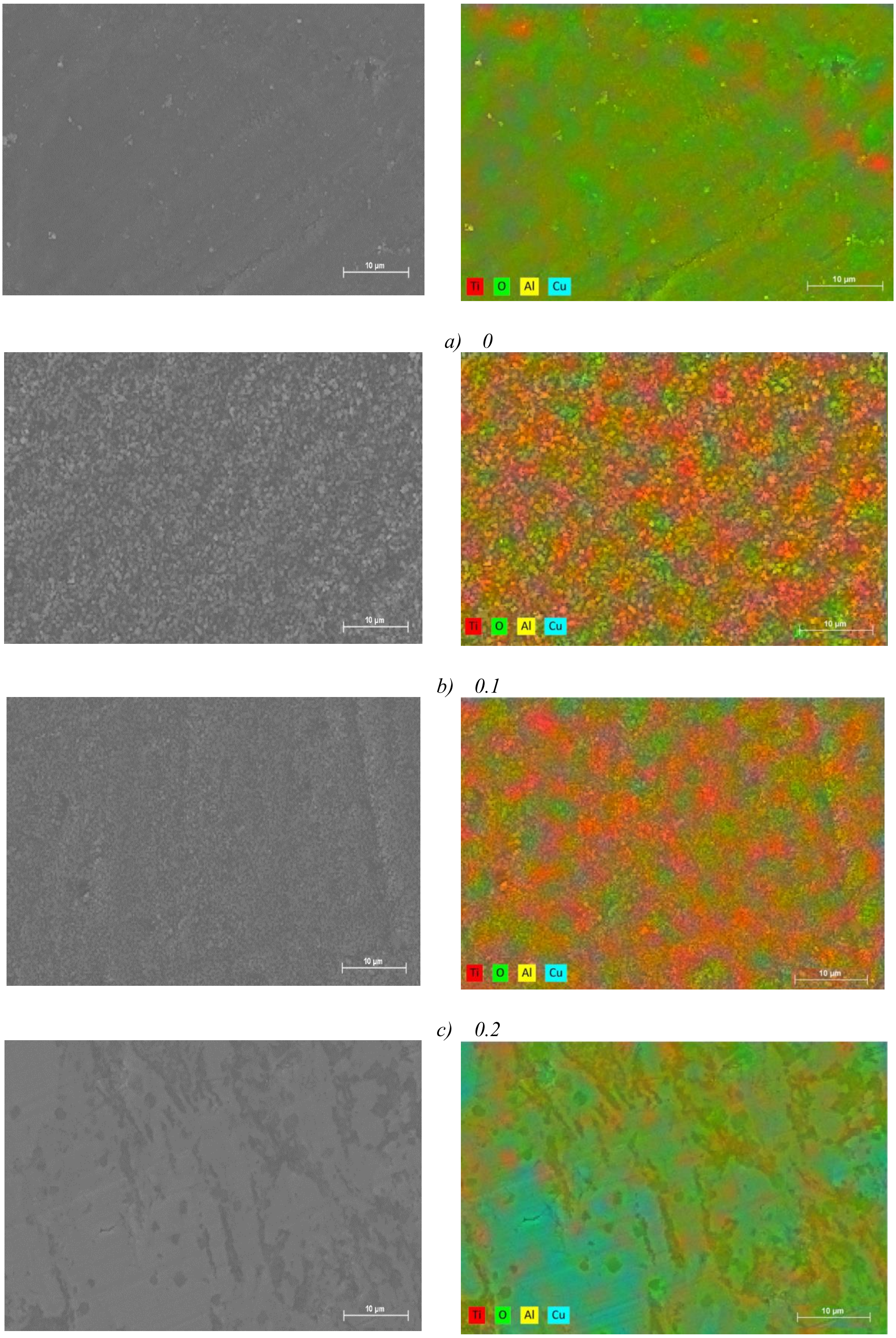
It has been found that at higher temperatures, the amount of expansion that occurred out of $TiAl_3$ formation, decreases [23]. They attributed this contraction to the integration of some components. This can be seen properly when comparing two samples containing 0.1 and 0.2 of copper. As illustrated, Fig. 4b shows more expansion, while Fig. 4c has less contraction. This issue is directly relevant to the reduction of the aluminum melting point in these two samples. The melting temperature of aluminum in the sample containing 0.2 of copper is lower than the melting point of aluminum in the sample containing 0.1 of copper. The temperature used in this research was 900*°C*. This temperature is higher than the melting temperature of the reaction and the start of reactions in the sample containing 0.2 of copper compared with the 0.1 of Cu sample.

Verdian[35] has shown that by grinding powders and heating them to 800*°C*, the final product Al_2O_3-$TiAl_3$ can be obtained without the presence of even small amounts of raw materials of TiO_2 or Al. In the current research, even at a temperature of 900*°C*, some raw materials were detected in the structure because the powder of the raw materials had just been pressed and sintered and no mechanical activation had been applied to it. Therefore, the speed of reactions in the current system is lower compared to the activated powders.

By increasing the copper content to 0.3, some copper is left without participating in the reactions. On the other hand, it has reduced the amount of $TiAl_3$ phase formation, probably, due to the reduction of the interaction surface of TiO_2 and molten aluminum.

One of the main goals of composite making is to produce a uniform and homogeneous microstructure, which is more visible in samples containing *0.1* and *0.2*. The structure of these two samples (Figs. 4b and c) show that the presence of copper has been able to create a homogeneous structure of the $TiAl_3$/Al_2O_3 composite in addition to reducing the melting temperature and increasing the tendency of $TiAl_3$ intermetallic compound formation.

It has been stated that the diffusion coefficient of aluminum in Ti solid solution and inversely is low [36-38]. But titanium can be dissolved in copper to a limited quantity [39], so copper could provide an appropriate substrate for the movement of atoms in the system. Copper also delivers a suitable surface for Al and Ti atoms to meet each other. It presumably plays a catalytic role in the production of titanium aluminide compounds. However, small amounts of Al_2Cu, and Cu_3Ti were also detected, which from another point of view, confirms the above claims about the dissolution of titanium in copper, i.e., the presence of a higher molar ratio of copper in the obtained Cu_3Ti composition may prove the existence of more copper atoms available.



a)   0

b)   0.1

c)   0.2

d)   0.3

Fig. 4 Scanning Electron Microscope images and elemental map of Ti, O, Al, and Cu in the samples with a molar ratio of 1 to 3 of TiO2 and Al, in which a) 0, b) 0.1, c) 0.2, and 0.3 moles of copper (as described in experimental section) have been added and heated at 900°C for 3 hours

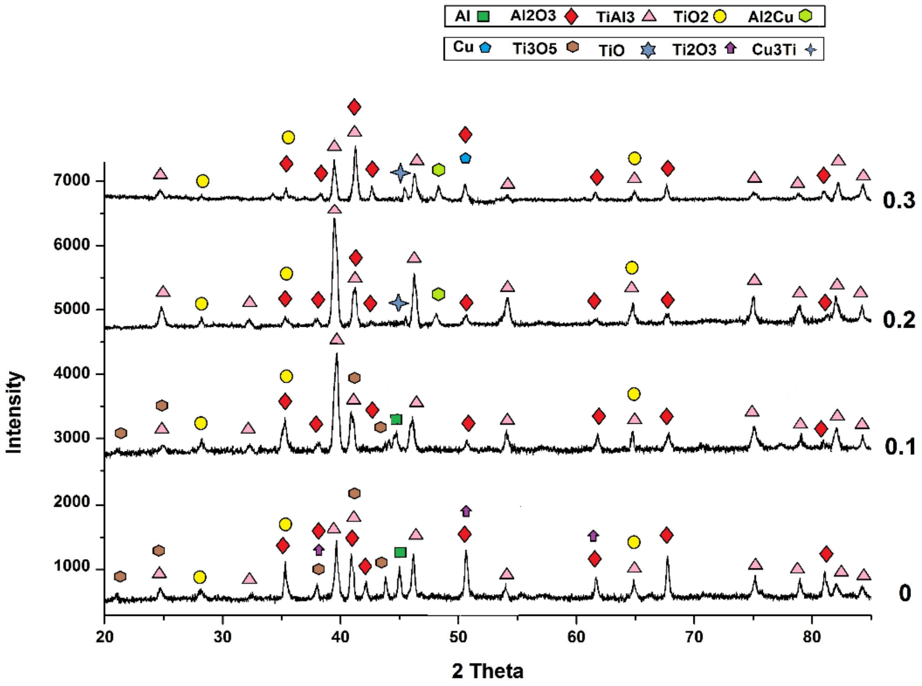


Fig. 5 The XRD results of samples containing the molar ratio of 1 to 3 of TiO2 and Al, to which a) 0, b) 0.1, c) 0.2, and 0.3 moles of copper have been added (according to the aluminum molar ratio) and heated at 900°C for 3 hours

Various types of research conducted on Ti-Cu or Al-Cu systems have reported different compositions. For instance, Bozik et al. [36] have disclosed the formation of Cu_3Ti and Cu_4Ti phases. Alshammari *et al.* [37] have reported the formation of the $CuTi_2$ phase by the addition of 0.5, 2.5, and 5% *wt* of copper to titanium. The reason for this could be the limitation of available copper atoms for the production of copper-rich compounds. In the welding of copper and titanium plates, the formation of Cu, $TiCu_2$, Ti_2Cu, and Ti_3Cu_4 phases have been identified [38]. Fowler et al. [39] have proved that if the weight percentage of copper exceeds *10*%, the Ti_2Cu phase will be created next to titanium, and in the case of exceeding *5*%, the Ti_3Cu phase will be created. Wei et al.[40] have detected the formation of Al Cu and Al_4Cu_9 in the weld metal. They believed that the standard Gibbs free energy of formation of copper aluminide phases is dependent on the ratio of available copper. This article may be a good justification for the aforesaid claim made.

As illustrated in Fig. 6, based on the molar ratio of aluminum and copper, various compounds have the minimum Gibbs free energy of formation. On that account, it is reasonable to produce the Al_2Cu compound in the current research, in which the molar ratio of copper to aluminum is 0.2 to 0.8.

Another noteworthy point that can be checked from the XRD results of the samples (Fig. 5) is that no copper oxides were formed. The reason for this may be explained by Ellingham Richardson's diagram [41]. Due to the presence of aluminum in the system and its strong tendency to react with oxygen, it is not possible to produce copper oxide despite the availability of oxygen released from TiO_2. Therefrom, extra copper can be identified in the elemental map of structure (Figs. 4 and 5).

Following the stoichiometric ratio required for the reaction of TiO_2 and Al to produce $TiAl_3$ (Eq. (1)), the amount of aluminum in this formula is more than the amount of aluminum used in the raw materials of the current research. Therefore, some amount of TiO_2 may remain unreacted (Fig. 5). However, the remaining aluminum can be seen in the copper-free sample, which proves that the reaction was not completed in three hours.

$$1.5\ TiO_2 + 6.5\ Al = 1.5\ TiAl_3 + Al_2O_3 \quad (1)$$

4- Conclusion

This study set out to explore the influence of copper on the formation of titanium aluminide intermetallic compounds from TiO_2 and Al powder raw materials. The findings indicate that the presence of copper decreases the melting temperature of aluminum. The molar ratio of 0.2 to 0.8 of copper and aluminum can also cause the $TiAl_3$ to be formed easier in a greater amount. It was also found that the addition of 0.2 mol of copper led to a uniform structure of the obtained $TiAl_3/Al_2O_3$ composite.

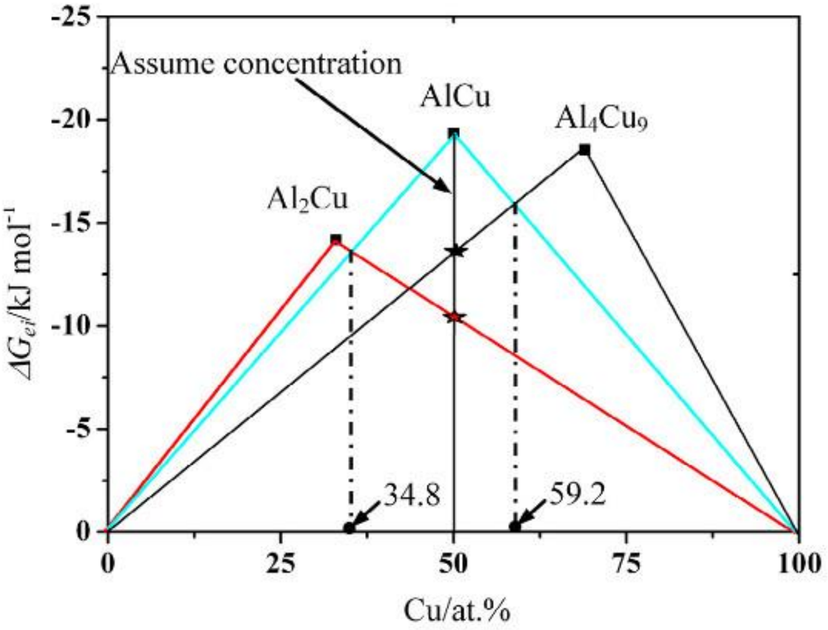


Fig. 6 The effective Gibbs free energy of formation for Cu-Al compounds in different molar ratios of copper [43]

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