RESEARCH PAPER

Study on the Ti-C/nano-ceramic additives reaction due to sintering of elemental powders

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A B S T R A C T:
The goal is to contribute towards well understanding the solid state reaction between Ti-C base ceramic and nano additives. A mixture of elemental powders of Ti and Graphite doped with a range of ceramic nano additives of each Al₂O₃ and CuO was pressed and heated up to 1100 °C. Phase evolution was then investigated using X-ray diffraction (XRD) and Differential thermal analysis (DTA). TiC is identified to be the most dominant phase through the reaction between Ti and Graphite at 1100 °C. TiO₂ is the most detected oxide due to the reaction of Ti-C/nano Al₂O₃ and also for Ti-C/nano CuO reaction. No evidence of both elemental Al and Cu was found due to structural analysis in the final products. On the other hand, energy dispersive X-ray spectroscopy (EDS) results confirm the appearance of Al and Cu in the produced microstructure. Further investigation may believe that Al and Cu ingress within TiC structure causing a fluctuation in its measured lattice constant.

KEY WORDS: Nano ceramic composites; nano additives; Titanium Carbide; Lattice constant.

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1. INTRODUCTION

The progress of ceramic-matrix composites is of increasing importance due to the strengthening of the fracture resistance, which is famous for ceramic materials (Vallauri et al 2008). Transition metal carbides and nitrides composites are of much interest due to their unlimited combination of mechanical and electrical properties (Vallauri et al 2008; Fu & Koc 2016). Ceramic composites appear to have better properties as compared with carbide materials like TiC (Fu & Koc 2016; Zhang et al 2007). In most cases secondary additives found to enhance the properties of ceramics especially transition metal carbides (Park et al 2016). Beside improving mechanical properties, additives or secondary phases have been added to improve densification and reduce the sintering temperatures come from strong covalent bonds of some carbides (Cheng et al 2012; Fu & Koc 2016; Xue et al 2016). Basically these carbides are highly effective for many applications as they present some physicochemical features of ceramics and electronic properties of metals (Baillet et al 2016). Titanium carbide (TiC) is attracted wide range of interest for many structural applications according to the properties like high melting temperature, hardness, chemical resistance and great electrical conductivity. TiC is used for magnetic recording heads, cutting tools, grinding wheels, corrosion
and wear-resistant coatings, high-temperature heat exchangers, turbine engine seals, and bullet-proof vests, and as gas-cooled fast nuclear reactor matrix materials (Niyomwas 2011; Fu & Koc 2017). On the other hand, TiC is very difficult to be sintered as mentioned before (Hassan et al 2007). Many additives or secondary phases have been added to carbides in order to improve sintering effects however, rare researches go through the reaction directions. In this work, the effect of Al₂O₃ and CuO nano additives on the stability of Ti-C base composite has been investigated. A more precise focus was placed on the reaction path and the resulting phases. Some consequences were based on a link between the results of the obtained phases and the amount of change in lattice constant of the early formed TiC phase.

2. EXPERIMENTAL

Titanium (>99%, 10 μm) and Graphite (>99%, 1 μm) were used as a raw material. The mixture composed of two groups: group 1 with 2%, 4%, and 6% Al₂O₃, respectively. The second group was mixed with 2%, 4%, and 6% CuO, respectively, which preparing with titanium and graphite for making the samples. The samples from each one synthesized by the technology of powder technology. First of all each group in the given percentage were mixed together by mechanical mixer for 2 h, then pressed using uniaxial pressing at loads of 5, 10, and 15 ton, respectively and sintering in an Ar. atmosphere at 1100 °C for 1 h. The used heating chamber was automatic to achieve the temperature of 500 °C with the rate of 40 °C / min and then to 1100 °C with the rate of 20 °C / min. The samples were then cooled down gradually inner the heating chamber. The phase composition of the sintered samples were studied by X-ray diffraction analysis (XRD-6000 Shimadzu) employing Cu Ka radiation in the variety 20-80° of 2θ, the measuring settings was 60 Kv and 80 mA. The prepared samples was examined by using SEM (inspect S50 SEM, Japan made) (high-energy beam of electrons in a raster scan pattern to investigate the sample's surface topography and composition.

The result of X-ray diffraction for Ti-C mixture sintered at 1100 °C is shown in figure 1. As can be seen, almost all diffraction peaks much the formation of cubic structure for transition metal carbide of TiC. Hence, the TiC cubic phase is been considering as the most stable phase through the sintering procedure mentioned in this work. It is well known that TiC needs very high sintering temperature to be formed (Cheng et al 2012; Xue et al 2016; Fu & Koc 2017). Hence, the temperature range presented here for the formation of almost pure TiC phase may be one of the lowest.

Figure 2 presented the XRD results of Ti-C base powders mixed with 2% weight percentage of nano Al₂O₃ pressed at different pressures and sintered at 1100 °C. As can be detected, TiO₂ oxide is the most dominant phase as a sintering product. No clear evidence for the effect of pressure value is been confirmed and TiC maintains his presence in most cases. Mehrizi obtained the formation of TiO₂ during the ball-milling process to form (TiC/Al₂O₃) nanocomposite (Zarezadeh et al 2016). Assuming that the TiC is formed first, it is reasonable to believe that titanium reacts with the oxygen withdrawn from the nano Al₂O₃ to form TiO₂ oxide that may be the most stable. This is supported by the presence of the remaining graphite in the XRD pattern. No evidence for the appearance of elemental Aluminum via structural examination. For the purpose of promoting this belief, EDX was utilizing to study the possibility of element appearance.

Figure 3 shows the EDX results of Ti-C base powders mixed with 2% weight percentage of nano Al₂O₃ sintered at 1100 °C. A clear proof is shown for the presence of Al in the microstructure. Thus, it can be said that the presence of aluminum may be concentrated within the crystalline network of TiC.

Figure 4 presented the XRD results of Ti-C base powders mixed with 2% weight percentage of nano CuO pressed at different pressures and sintered at 1100 °C. As can be detected, TiO₂ oxide is the most dominant phase as a sintering product. No clear evidence for the effect of pressure value is been confirmed and TiC maintains his presence in most cases together with some Cu₂O. Assuming that the TiC is formed.
first, it is reasonable to believe that titanium reacts with the oxygen withdrawn from the nano CuO to form TiO$_2$ oxide that may be the most stable. This is supported by the presence of the remaining graphite in the XRD pattern. No evidence for the appearance of elemental Cooper via structural examination. For the purpose of promoting this belief, EDX was utilizing to study the possibility of element appearance.

Figure 5 shows the EDX results of Ti-C base powders mixed with 2% weight percentage of nano CuO sintered at 1100 °C. A clear proof is shown for the presence of Cu in the microstructure. Thus, it can be said that the presence of copper may be concentrated within the crystalline network of TiC. In order to be able to detect the effect of both Al and Cu on the microstructure, lattice constant of formed TiC has been determined and compared. Figure 6 show the effect of increasing Al$_2$O$_3$ and CuO on the lattice constant of appeared TiC in both cases of Ti-C/nano Al$_2$O$_3$, and Ti-C/nano CuO, respectively. It is assumed to increase the effect of both resultants Al and Cu on the lattice constant of TiC by increasing the source of both elements. It is confirmed that by increasing the percentage of Al$_2$O$_3$ lattice parameter of TiC tends to be decreased. CuO percentage causes in further decreasing of lattice constant of TiC. Hence, a clear proof may be put here to confirm the effect of elemental Al and Cu on the lattice constant hosted TiC structure. The fluctuating of lattice value in both cases may be refer to the atomic size of the different elements.

4. CONCLUSIONS

The following conclusions can be set from the results presented in this paper

1- TiC phase found to be the most stable from the direct reaction between Titanium and Graphite at 1100 °C sintering temperature.

2- Nano additives of Al$_2$O$_3$ and CuO to Ti-C base mixture helps to stabilize the TiO$_2$ phase after sintering at the same temperature range as a result of reaction with oxygen.

3- Nano Al$_2$O$_3$ causes to add elemental aluminum in the microstructure as a result of reaction with preliminary formed TiC.

4- Nano CuO causes to add elemental cooper in the microstructure as a result of reaction with preliminary formed TiC.
Figure 3. The EDX result of Ti-C/2% Al₂O₃ mixture sintered at 1100 °C
a) Chemical composition analysis, b) Mapping analysis

Figure 4. The XRD result of Ti-C/2% CuO mixture sintered at 1100 °C and pressed at a) 5 Ton, b) 10 Ton, and C) 15 Ton

Figure 5. The EDX result of Ti-C/2% CuO mixture sintered at 1100 °C
a) Chemical composition analysis, b) Mapping analysis

Figure 6. Lattice constant determination of resultant TiC

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