The Effect of Milling of TiO_2-Al Powder Mixture on Microwave Synthesis of $TiAl/Al_2O_3$ Composite

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1- Introduction

Titanium aluminide intermetallic compounds are good candidates for application in aerospace industries due to their high strength to weight ratio, high melting point and high oxidation resistance. Their creep resistance could also be improved by addition of Al_2O_3 particles. In-situ formation of these particles provides smaller particles with a more uniform distribution which results in higher strength and higher fatigue and creep resistance. One of the methods of in situ formation of composites is through chemical reactions for reinforcement formation. The reduction reaction of TiO_2 with Al could be used for in situ synthesis of $TiAl/Al_2O_3$ composite. Mechanical activation of the powder mixture would also provide a composite with a more uniform microstructure and smaller reinforcement particles. It was reported that without mechanical activation the reaction temperature would be higher than 1100 °C while after activation of the powder mixture, the reaction temperature would be reduced to 550 °C. The required activation energy would also be reduced from 208 to 33 kJ/mol after mechanical activation. The research results show that for formation of the composite, the powder mixture needs to be heat treated at 550 °C under vacuum atmosphere. By microwave heating the products would be obtained at smaller time durations than conventional heating routes. It was observed that silicon carbide and graphite are good microwave absorbers. These materials can provide temperatures of 500-1100 °C in a few seconds. Therefore the reaction would occur easily. In this research, $TiAl/Al_2O_3$ composite was prepared from Al and TiO_2 powder mixture by mechanical activation and microwave heating. The effect of activation intensity on combustion time and phase constituents was studied.

2- Experimental

Al and TiO_2 powders were mixed according to the following reaction with 10 wt% of additional Al.

$$3TiO_2 + 7Al = 3TiAl + 2Al_2O_3 \quad (1)$$

The powder mixture was mechanically activated in a planetary ball mill with stainless steel cup and balls (15 mm diameter). The milled powders (1 g) were pressed by 30 kN force to tablets with 12.4 mm diameter and 3.4 mm height. The pressed samples were placed on an alumina block and covered by graphite powder in a household microwave oven (GE 2370G, Samsung) and were heated for 5 minutes using 850 W of power.

The milling intensity was assumed to be proportional to a dimensionless number defined by the following equation:

$$I = \frac{m_b(g)}{m_p(g)} \times t\ (\text{min}) \times \omega\ (\text{min}^{-1}) \quad (2)$$

where I is the activation intensity number, m_b/m_p is the ball to powder weight ratio, t is the milling time and ω is the angular velocity of the sun disc of the mill. Table 1 shows the mechanical activation conditions for all the experimental runs.

Table 1 Milling conditions employed to induce different levels of mechanical activation to the samples

Sample	$(m_b/m_p) \times t \times \omega$	$I \times 10^{-4}$	I/I_0*
A1	15×200×240	72	1
A2	20×250×240	120	1.66
A3	20×200×360	144	2
A4	15×300×360	162	2.25
A5	15×250×480	180	2.5
A6	25×300×240	180	2.5
A7	25×250×360	225	3.125
A8	25×200×480	240	3.33
A9	25×300×480	288	4

* I_0 corresponds to the smallest intensity number related to sample A1

Phase identification of the synthesized powders was performed by Philips X'pert Pro X-ray diffractometer (XRD) using Cu-K_α radiation. Scanning electron microscopy (SEM) TESCAN VEGAII was used to study the microstructure of the synthesized samples.

3- Results and Discussion

The results showed that even the highest intensity of mechanical activation (Sample A9) would not result in the formation of $TiAl/Al_2O_3$ composite and the samples needed thermal activation. The results also revealed that the sample without mechanical activation ignited after 140 seconds but the aimed phases ($TiAl/Al_2O_3$ composite) were not detected. Fig. 1 shows changes in ignition time of the samples with activation intensity number. The results show that activation would decrease the energy barrier of the reaction occurrence which accelerates reaction progress.

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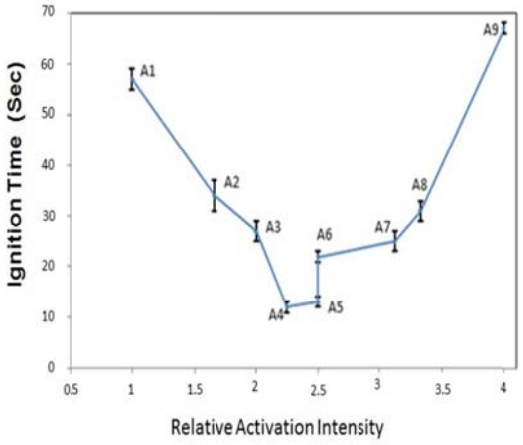

Figure 1 Variation of the ignition time of the samples with the relative activation intensity number

Mechanical activation would increase the interfaces between the starting materials. This would facilitate nucleation of the combustion products. However, there is an optimum amount of activation that would result in the minimum ignition time. Higher intensities of mechanical activation would cause partial reaction between starting materials during the mechanical activation itself. The presence of products at the interfaces would be a barrier for direct contact of starting materials. The consequence is higher ignition times for these samples.

Fig. 2 shows the results of XRD analysis of the samples. It is seen that the combustion products in all the samples consist of titanium aluminide and alumina phases. In addition to these phases, presence of TiC and $Al_2Ti_4C_2$ phases are observed that could be due to the presence of stearic acid as PCA in the milling process. Fig. 3 shows a representative SEM micrograph of the synthesized composites. The bright matrix is the intermetallic phase and the dark dispersed particles are Al_2O_3 particles.

4- Conclusions

In this research, $TiAl/Al_2O_3$ composites were synthesized using microwave heating from mechanically activated TiO_2-Al powder mixtures. The results confirmed that the obtained composites mainly consist of TiAl and dispersed Al_2O_3 particles. The results also revealed that with increasing the intensity of activation, the ignition time is first decreased and then increased. At the optimum ignition time of 27 s, the composite structure was more uniform and less porous which consisted of 23.2% TiAl and 69.6% Al_2O_3.

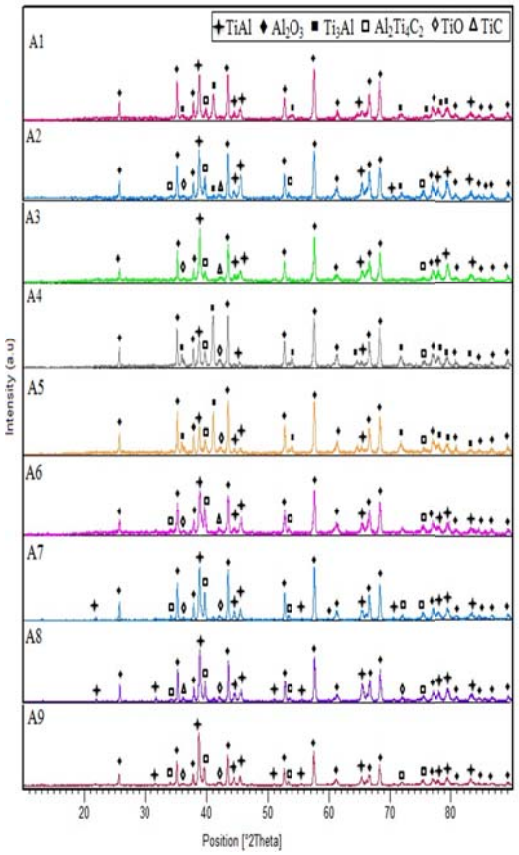

Figure 2 XRD patterns of the activated samples after microwave synthesis

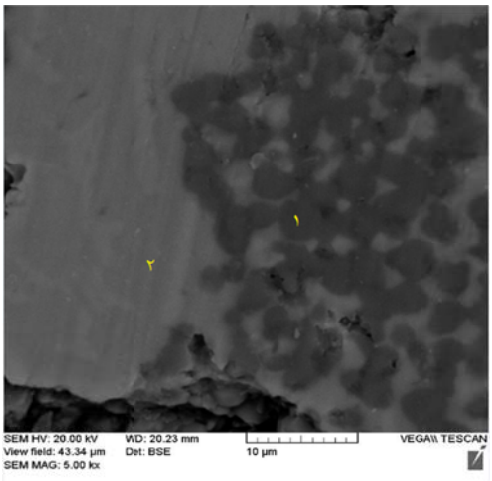

Figure 3 SEM micrograph of the synthesized composite