The Study of Combustion synthesis, Microstructure and Mechanical Properties of In Situ Composite Produced In Al-V2O5 System

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1- Introduction

In the advanced industries, the need to produce parts with high strength and abrasion resistance, yet with light weight and flexible, is an increasing tendency for making advanced materials, particularly composites. This is because a single material alone generally cannot provide all engineering needs. Composites produced by in situ are new categories of composite materials that are suitable for advanced applications and wear. In situ composites compared with non-in situ composites have attractive advantages that include:

1. The thermodynamic stability of reinforcements, which resulted in the less loss of mechanical properties at elevated temperatures.
2. A Clean interface of matrix and reinforcement that improved interface strength.
3. The uniform dispersion of fine reinforcement particles leading to top mechanical properties of the composite.

Newly the aluminothermic combustion synthesis for production of composites and aluminide-alumina alloys developed. The basis of this method is an exothermic reaction that leads to in-situ solid metal-ceramic particles (less than one micron) in the aluminide matrix.

This method is used and studied by other researchers for manufacturing in-situ composites, such as Al-TiO2, Al-TiO2-C, Al-ZrO2, Al-ZrO2-B and Al-ZrO2-C.

In the present study, due to advantages such as the clean interface and free of contaminants of in situ composites and also low density and resistance to creep and wear at elevated temperatures, to produce intermetallic precipitates aluminum and vanadium and phase ceramic Al2O3 production of in situ composites Al3V / Al2O3 in Al-V2O5 system was studied by using the aluminothermic combustion synthesis.

2- Experimental

For the synthesis of Al3V / Al2O3 composite, powders of Al (with 99.5 percent purity and average particle size 45μm) and V2O5 (with 99.2 percent purity and average particle size 120μm) were used as raw materials. In order to create an activated and homogeneous mixture, Al and V2O5 powders with molar ratio of 28:3 (in accordance with Equation 1) in the planetary ball mill (Model Pulverisette Fritsch 6) was milling with the container and balls of steel, under argon atmosphere with duration of one hour.

Weight ratio of Ball to powder was considered 4:1 having a rotational speed of 250 rpm. Due to former studies, milling parameters were selected in such a way that prevented the combustion reaction in container, in order to investigate the effect of temperature at different stages of the composite formation. Mixed powder was compacted under the pressure of 500 MPa to make cylindrical samples with a diameter of 10 and a thickness of 5 mm.

28Al + 3V2O5 = 6Al3V + 5Al2O3 \( (1) \)

To study the transformation temperatures and predict the reactions temperature, thermal analysis was done by using BAHR Thermoanalyse apparatus (STA 504) from ambient temperature to a temperature of 1100 °C, under argon atmosphere and heating rate of 10 °C/min on a sample with 10 mg weight. Cylindrical samples after cold press were heated in a tube furnace under an argon atmosphere and heating rate 10 °C/min to a temperature between the temperature range of 650 °C to 1000 °C and were held at that temperature for 10 minutes.

Phase transformation after heating the samples at different temperatures were studied by using X-ray diffraction (Philips PW-3040) equipped with light-producing Cu-ka and the accelerating voltage was 40 kV. Microstructure was studied by using a scanning electron microscopy (VEGA \ TESCAN ) with energy resolution analyzer (EDS).

3. Results and Discussion

Differential thermal analysis (DTA) curve of raw sample that was heated to 1100 °C under argon atmosphere is shown in Figure 1. In this curve two endothermic peaks and three exothermic peaks can be seen. Due to the fact that the combustion reaction did not occur at milling time it can be expected that the powder mixture is at activated state. There are two endothermic peaks in 656 °C and 728 °C temperatures and three exothermic peaks in 569 °C, 770 °C and 863 °C temperatures in this curve. Three exothermic peaks indicated the various reactions and the formation of intermediate and transitional phases and compounds during the composite structural changes.

![Fig. 1 Thermal analysis curves](image)
Figure 2 shows X-ray diffraction pattern of the sample that was heated at 900 °C. In this figure, there are not peaks of aluminum and vanadium oxides with lower capacity, so this subject indicates completion of the reduction reaction of vanadium oxides by aluminum and consumption of these phases. In this pattern, also there are not peaks of \( \gamma \)-Al\(_2\)O\(_3\), so phase transition \( \gamma \)-Al\(_2\)O\(_3\) is fully taken into \( \alpha \)-Al\(_2\)O\(_3\). At Figure 2, also the relative intensity of Al\(_3\)V phase has increased. At this stage, Al\(_3\)V phase, by reduction of vanadium atom with the remaining aluminum is formed. Thus, the third exothermic peak of thermal analysis curve formed from two overlap peaks is related to the following reactions:

\[
2\text{Al} + 3\text{V}_2\text{O}_3 = \alpha \text{-Al}_2\text{O}_3 + 6\text{V} \tag{2}
\]

\[
3\text{Al} + \text{V} = \text{Al}_3\text{V} \tag{3}
\]

With accordance to whatever has been observed in the pattern of diffraction, there are peaks of V-phase in products. Existence of this phase also confirmed happening of reaction 2.

**Microstructural study:**

Figure 3 shows a microscopic image taken with SEM from the composite structure of a sample that was heated at 1000 °C. As can see in this figure, composite microstructure is composed of three regions of black, gray and white, which confirmed existing of three phases in the structure. According to the results of EDS analysis, dark areas for \( \alpha \)-Al\(_2\)O\(_3\) particles is put together. The EDS analysis of gray areas with percent of vanadium are less than white area in Al\(_3\)V and white areas will be Al\(_8\)V\(_5\). Thin areas of white can be seen in the image of this temperature. These areas are performed due to less distances penetration of V, so Al\(_3\)V is completely transformed to Al\(_8\)V\(_5\).  

**4-Conclusion**

1- Study of Thermal analysis and X-ray diffraction shows that production of in situ composites Al\(_3\)V / Al\(_2\)O\(_3\) by Aluminothermic Combustion synthesis which is a step by step process. Vanadium oxides \( \text{V}_2\text{O}_4\) and \( \text{V}_2\text{O}_3\) are formed as the main intermediate phase and two phases \( \gamma \)- Al\(_2\)O\(_3\) and Al\(_{23}\)V\(_4\) as transitional phases.

2- With increasing of temperature transition phase of \( \gamma \)-Al\(_2\)O\(_3\) fully converted to \( \alpha \)-Al\(_2\)O\(_3\) and due to the reaction of vanadium remaining in the interface of Al\(_3\)V and \( \alpha \)-Al\(_2\)O\(_3\), Al\(_8\)V\(_5\) phase occurs in a layer around Al\(_3\)V phase.

3- Hardness and strength of samples are increased with increasing temperature due to the more volume fraction of intermetallic compounds and ceramic, but seeking to increase the amount of brittle phase Al\(_8\)V\(_5\), samples shows less deformation.