

## The effect of mechanical activation on the kinetic and formation mechanism of a niobium aluminide based nanocomposite

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### ABSTRACT

In this paper the feasibility of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> nanocomposite formation through mechanochemical reaction between Al and Nb<sub>2</sub>O<sub>5</sub> and the effect of mechanical activation on the kinetic and reaction mechanism were investigated. Structural and phase evolution during mechanical alloying were studied by employing transmission electron microscopy (TEM), X-ray diffraction (XRD) and differential thermal analysis (DTA) techniques. It was found from the results that the reduction of Nb<sub>2</sub>O<sub>5</sub> by Al takes place in an explosive mode and after 450 min of mechanical alloying leading to the formation a microstructure consisting of NbAl<sub>3</sub> matrix and nanocrystalline Al<sub>2</sub>O<sub>3</sub> particles. The results also showed that heating the as-blended powder mixture of Nb<sub>2</sub>O<sub>5</sub> and Al, leads to the formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite through two exothermic reaction; A thermite reaction at 880 °C followed by the synthesis of NbAl<sub>3</sub> intermetallic at 944 °C. The mechanical activation of Nb<sub>2</sub>O<sub>5</sub> and Al powder for 5h decreased the formation temperature of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> from 944 °C to 625 °C. Five isoconversional methods, of Kissinger-Akahira-Sunose, Tang, Flynn-Wall-Ozawa, Starink and Friedman were used to determine the activation energy,  $E_a$ , for NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> formation reaction and its variation with conversion degree  $\alpha$ . The results showed that these methods lead to similar or comparable values of  $E_a$ .

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### 1. Introduction

Developments of high temperature and high strength structural materials have always been of considerable interest to the aviation industry. Among various candidate materials, intermetallics can have higher melting temperatures than the superalloys and, with metallic bonding, at least the possibility of better toughness than ceramics [1]. Some intermetallics, e.g. NbAl<sub>3</sub> have been considered as a potential high temperature structural

material [2–4]. Titanium and nickel aluminides are generally limited to structural applications below 1000 °C. However, NbAl<sub>3</sub> can has extremely high strength even above 1200 °C [5]. In addition, NbAl<sub>3</sub> has a potential application as turbine blade material in aircraft engines and stationary gas turbine [6], thanks to its relatively low density (4.54 g/cm<sup>3</sup>) and very good resistance to oxidation processes. However, despite its attractive features, its use is limited by some shortcomings such as inadequate

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ductility at room temperature [7]. Several possible approaches seem to improve its major drawbacks such as grain boundary strengthening [8], oxide dispersion strengthening [9] and grain refinement [7,10,11]. All these factors have led investigators to synthesize intermetallic matrix nanocomposites. Various intermetallic-based composites such as NiAl/Al<sub>2</sub>O<sub>3</sub> [12], (Fe,Ti)<sub>3</sub>Al/Al<sub>2</sub>O<sub>3</sub> [13], NiTi/Al<sub>2</sub>O<sub>3</sub> [14] and TiAl/Al<sub>2</sub>O<sub>3</sub> [15] have been synthesized using mechanochemical reaction between Al and appropriate oxides. To the authors' knowledge, no comprehensive study has been reported in the literature on kinetic of reaction between Nb<sub>2</sub>O<sub>5</sub> and Al. So, the aim of this work is to improve our understanding of the fundamental aspects of the NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> formation mechanism and determine the effect of mechanical activation on the reaction mechanism between Nb<sub>2</sub>O<sub>5</sub> and Al.

## 2. Materials and methods

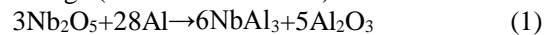
The powders of Al (99.5%) and Nb<sub>2</sub>O<sub>5</sub> (99.5%) were used as raw materials. The elemental powders with stoichiometric composition were mechanically alloyed in a planetary high energy ball mill. High chromium-carbon hardened steel vial (57 mm diameter and 76 mm height) containing the powders and the balls (15 mm diameter) was fixed onto a rotated disc and rotated in the opposite direction to that of the larger platform. The rotation speeds of the vial and the platform were fixed at 350 and 175 rpm respectively.

X-ray diffraction technique was used to follow the structural changes of powders after milling and annealing. A Philips diffractometer (40 kV) with Cu K<sub>α</sub> radiation ( $\lambda = 0.15406$  nm) was used for XRD measurements. The XRD patterns were recorded in the  $2\theta$  range of 30–90° (step size 0.05 degree and time per step 1 s). Differential thermal analysis (DTA) was performed using a Reometric STA 503 differential thermal analyzer, with a temperature accuracy of  $\pm 0.1$  °C under argon supplied at a rate of 60 ml.min<sup>-1</sup>. Powder samples weighting 30 mg were encapsulated into aluminum pans and heated at continuous heating rates of 10 °C.min<sup>-1</sup> up to 1200 °C. The heat treatment of the samples was conducted in Ar atmosphere. The Al<sub>2</sub>O<sub>3</sub> and NbAl<sub>3</sub> crystallite sizes were evaluated using the Williamson–Hall formula from the line broadening of the diffraction peaks [16].

## 3. Results and Discussion

### 3.1. Mechanochemical reaction between Nb<sub>2</sub>O<sub>5</sub> and Al

The occurrence of reaction between Nb<sub>2</sub>O<sub>5</sub> and Al according to the following reaction is possible regarding its negative free energy change ( $\Delta G^\circ = -2700$  kJ/mol):



In order to synthesize NbAl<sub>3</sub>/40vol% Al<sub>2</sub>O<sub>3</sub>, a mixture of Al/51% wt Nb<sub>2</sub>O<sub>5</sub> were mechanically alloyed for different periods of time. Figure 1 shows the XRD patterns of powder mixture after different milling times.

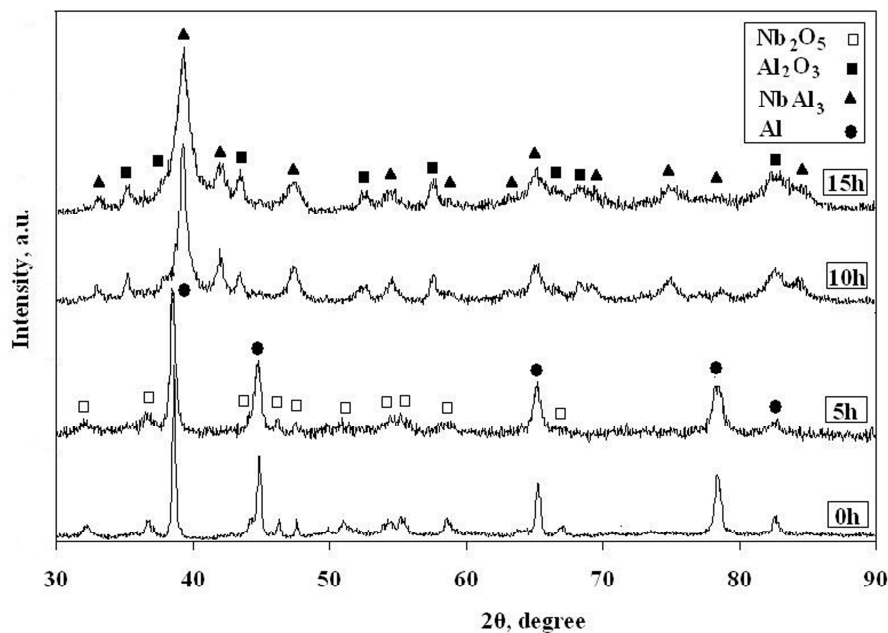


Fig. 1. XRD patterns of Al– Nb<sub>2</sub>O<sub>5</sub> powder mixture after 0, 5, 10 and 15h of milling.

The diffraction pattern of initial powder mixture shows all expected peaks of Al and Nb<sub>2</sub>O<sub>5</sub>. The early stages of milling are marked with the broadening and shortening of the peaks corresponding to the initial mixture as a result of refinement of crystallite size and enhancement of lattice strain. Refinement of the crystallite size to nanometer range and increasing the defect densities can promote the reaction kinetics by providing short circuit diffusion paths [17]. At longer milling times the activation energy required for reaction between Nb<sub>2</sub>O<sub>5</sub> and Al is provided through mechanical activation and reaction (1) occurs. So XRD patterns after 10h, showed no Nb<sub>2</sub>O<sub>5</sub> and Al peaks, while several peaks corresponding to Al<sub>2</sub>O<sub>3</sub> and NbAl<sub>3</sub> compounds developed on XRD patterns. As a result, the released heat from exothermic reaction (1) provides the activation energy for NbAl<sub>3</sub> formation at the same time.

The adiabatic temperature, T<sub>ad</sub>, for this reaction is 2525°C, which is higher than the critical value of 1027°C [18].

It is therefore expected that a combustion reaction takes place during milling of Al and Nb<sub>2</sub>O<sub>5</sub> powder mixture. In our previous work [19], the change of vial temperature during milling of Al and Nb<sub>2</sub>O<sub>5</sub> mixture was measured and it was found that after 450min of milling, the temperature of vial increases suddenly, suggesting that the exothermic combustion reaction occurs between Al and Nb<sub>2</sub>O<sub>5</sub>.

In the final stage of milling, NbAl<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> had a crystallite size of about 8 and 13 nm, respectively.

The microstructure and Corresponding selected area diffraction pattern (SADP) of the 15 h milled sample observed in TEM are shown in Figure 2. As can be seen in Figure 2(a), several rings in SAD pattern and the lack of preferred orientation confirmed the formation of nanostructure intermetallic. In addition, it can be seen in Fig, 2(b) that reinforcement particles i. e. Al<sub>2</sub>O<sub>3</sub> particles have been formed in nanosize scales which are distributed uniformly in the niobium aluminide matrix.

### 3.2. Thermal analysis

In order to determine the reaction mechanism between Nb<sub>2</sub>O<sub>5</sub> and Al, the thermal analysis of the as-blended powder was carried out. As observed in Figure 3, there are one endothermic peak at 660 °C which is responsible for the melting of Aluminum and two exothermic peaks at 880 °C and 944 °C.

The first exothermic peak at 880 °C corresponds to the reduction of Nb<sub>2</sub>O<sub>5</sub> producing Al<sub>2</sub>O<sub>3</sub> and Nb. After this thermite reaction, the remaining Al in the system reacts with Nb at 944 °C to produce NbAl<sub>3</sub> intermetallic compound. In other words, the second exothermic peak at 944 °C is responsible for the production of NbAl<sub>3</sub> intermetallic. In fact for the as-blended powder, the NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite is produced through a thermite and a synthesis reaction according to the following:

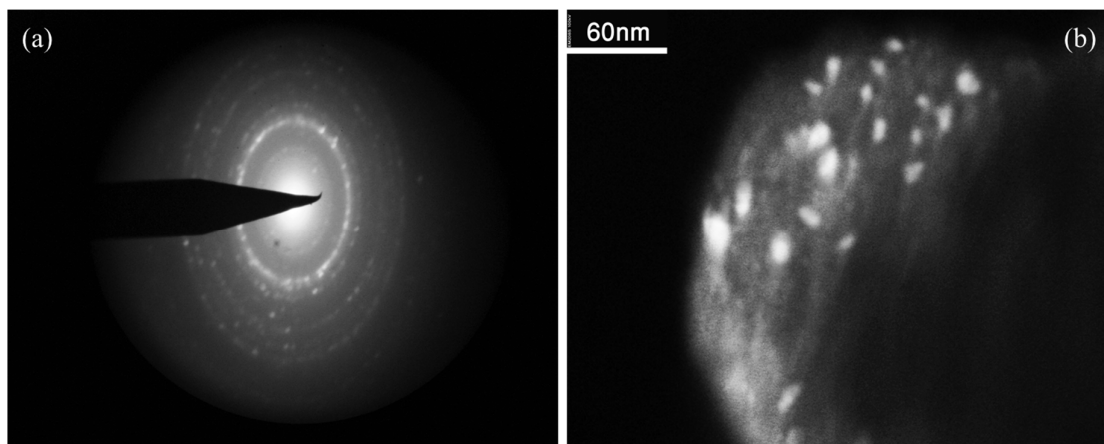
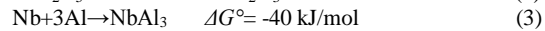
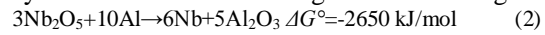


Fig. 2. TEM dark-field image and corresponding SADP taken after 15 h of MA.

These two reactions have negative standard free energy changes and can thermodynamically occur. It seems that the released heat from reaction (2) provides the required activation energy for reaction (3) to occur. Hence, in order to elucidate the effect of mechanical activation

on the formation mechanism of  $\text{NbAl}_3/\text{Al}_2\text{O}_3$  composite, thermal analysis was also carried out on the  $\text{Nb}_2\text{O}_5/\text{Al}$  powder mixture milled for 5h. Figure 4 shows the DTA results for this mixture and as can be seen there is only one exothermic peak at 625 °C.

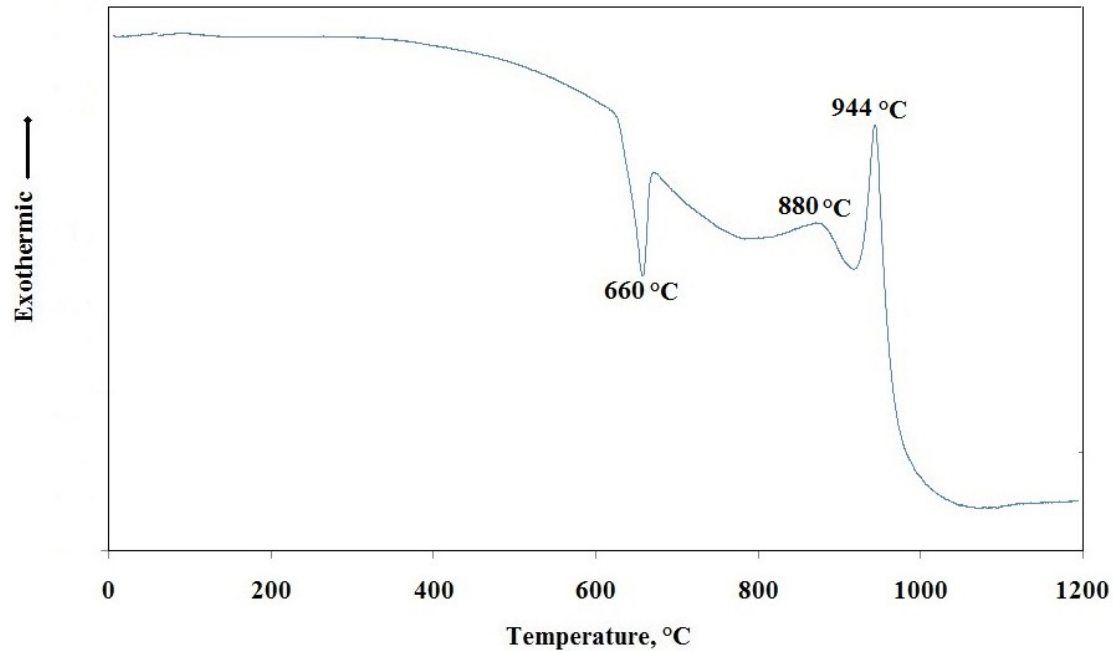


Fig. 3. DTA curve of the as-blended 49wt%Al/ $\text{Nb}_2\text{O}_5$  powder mixture, at a heating rate of  $20^\circ\text{C}\cdot\text{min}^{-1}$ .

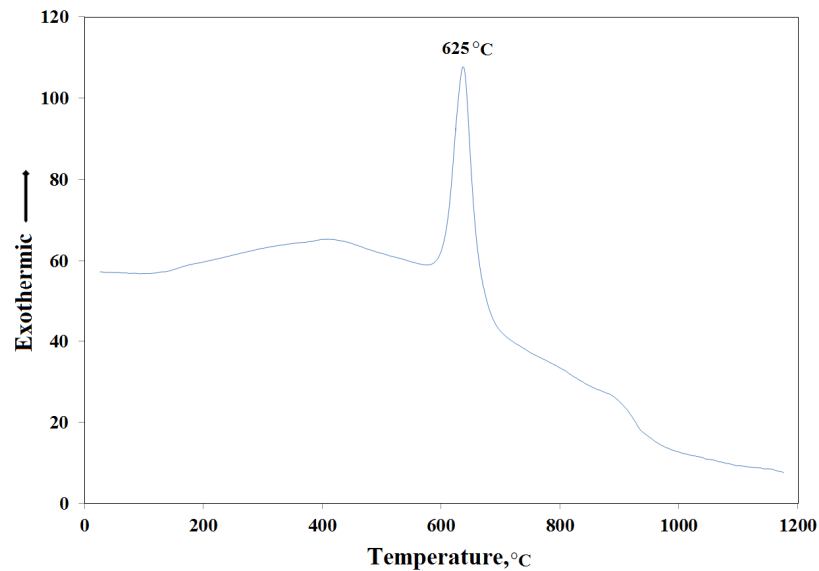


Fig. 4. DTA curve of the 49wt%Al/ $\text{Nb}_2\text{O}_5$  powder mixture milled for 5 h, at a heating rate of  $20^\circ\text{C}\cdot\text{min}^{-1}$ .

To analyze the transformation process responsible for this exothermic peak, the as-blended powder was heated up to 700 °C in Ar atmosphere at a heating rate of 20 °C/min similar to the DTA condition. The XRD patterns of the powder mixture milled for 5h, before and after annealing are presented in Figure 5.

As can be seen, the annealed sample mainly consists of NbAl<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> phases. So, this exothermic peak corresponds to the formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite. So, it can be concluded that mechanical activation for 5h has two major effects on the formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite. First of all, the formation temperature of this compound is decreased from 944 °C to 625 °C. This is due to the promotion of diffusion process because of increase in grain boundaries and defects which need a lower energy and provide more rapid pathways for solid-state diffusion. Also, increasing contact areas between the particles which improve the reaction kinetics [20]. The second effect is that the two reactions (2) and (3) take place simultaneously. In the other word, mechanical activation accelerates the occurrence of reaction (3) more than reaction (2).

### 3.3 Kinetics of mechanically alloyed 49wt%Al/Nb<sub>2</sub>O<sub>5</sub> powder mixture conversion to NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> compound

In this section formation kinetics of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> compound by means of nonisothermal techniques will be discussed. General aim of any kinetic analysis is the description of the progress of a transformation. In the case of the solid-state reactions (or transformations) due to the fact that not only one mechanism (as diffusion or interface reaction) might participate and several intermediate states might bypass, all of which can have different temperature dependences, the analytical studies need to be simplified. For the kinetics of thermally stimulated solid-state reactions, the following formula has been widely accepted:

$$d\alpha/dt = A \exp\left(\frac{-E_a}{RT}\right) f(\alpha) \quad (4)$$

where  $\alpha$  is the extent of reaction,  $t$ , the time,  $R$ , the universal gas constant,  $T$ , the temperature,  $f(\alpha)$ , the kinetic model function, and  $A$  and  $E_a$  are apparent pre-exponential factor and apparent activation energy, respectively. This equation can be integrated by separation of variables:

$$\int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \int_{T_0}^T \exp\left(\frac{-E}{RT}\right) dT \approx \frac{AE}{\beta R} \int_0^y \frac{\exp(-y)}{y^2} dy \quad (5)$$

where  $T_0$  is the initial temperature,  $y = E/RT$  and  $T$  is the temperature at an equivalent state of transformation.

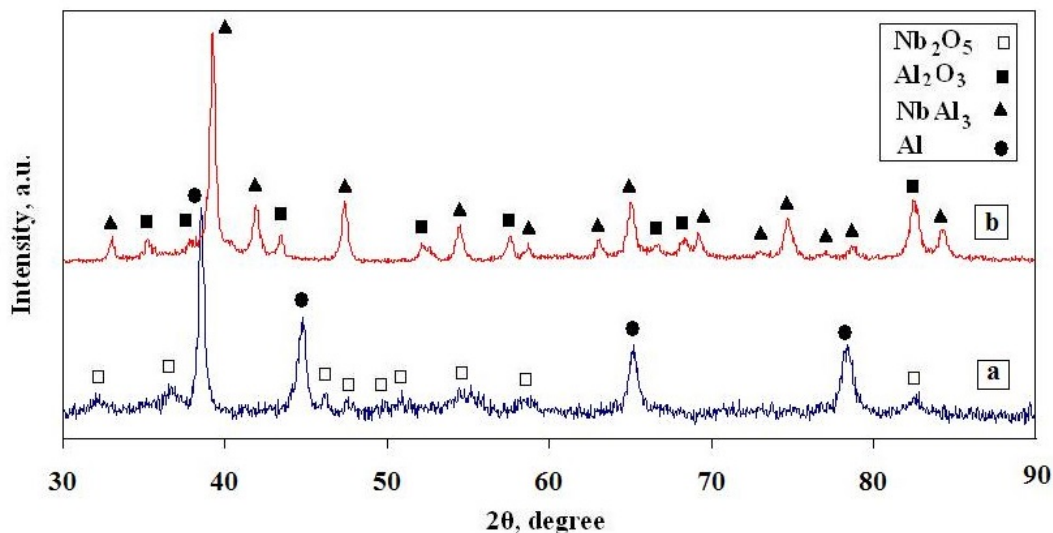


Fig. 5. XRD patterns of the 49wt%Al/Nb<sub>2</sub>O<sub>5</sub> powder mixture milled for 5h, before and after annealing.

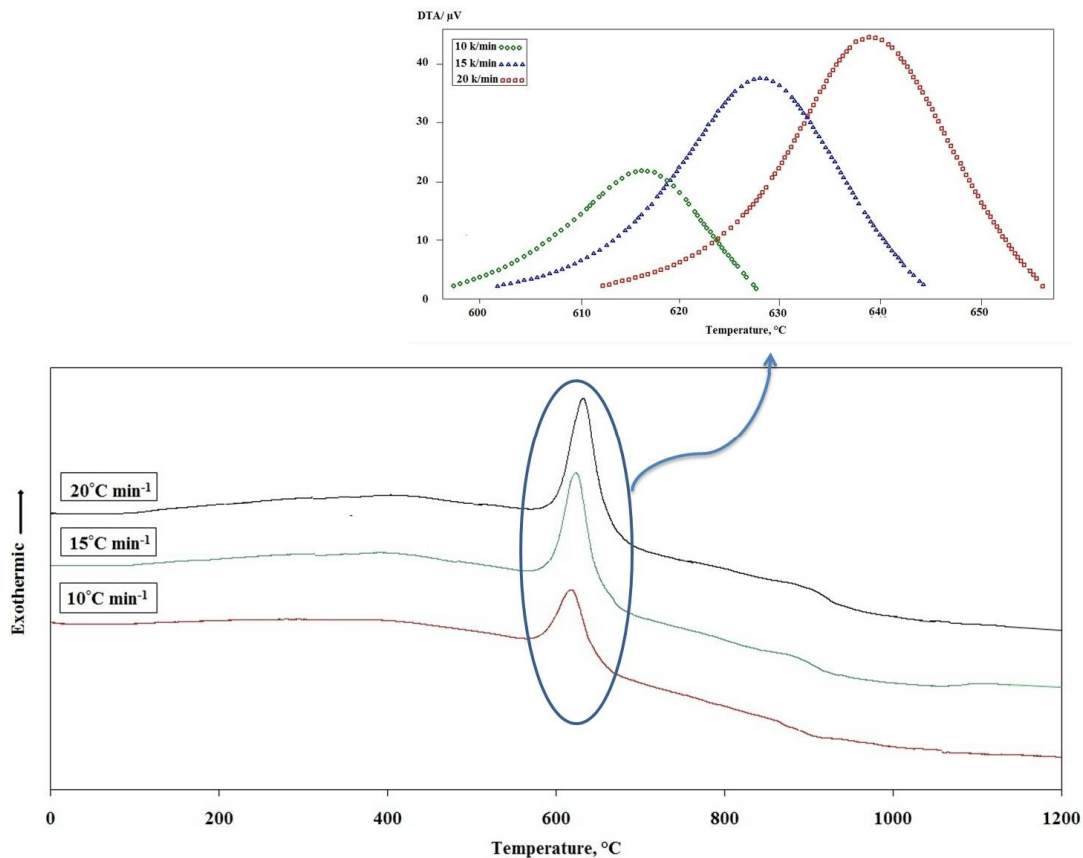


Fig. 6. DTA curves of the mechanically activated 49wt% Al/Nb<sub>2</sub>O<sub>5</sub> powder mixture at different heating rates.

The integral on the right-hand side is called the temperature integral,  $P(y)$ , and does not have analytical solution.

$$P(y) = \int_{y_f}^{\infty} \frac{\exp(-y)}{y^2} dy \quad (6)$$

To solve the temperature integral, several approximations were introduced. In general, all of these approximations lead to direct isoconversion methods. In this paper, model-free direct isoconversion approaches were used in kinetic evaluation. Model-free methods allow evaluation of the Arrhenius parameters without choosing the reaction model  $f(\alpha)$ . The best-known approaches, if several measurements with different heating rates and/or with different temperatures have been made, are the isoconversional methods according to Friedman (FR) and the integral isoconversional method according to Kissinger-Akahira-Sonose (KAS), Flynn-Wall-Ozawa (FWO) and finally the Tang method [21]. On the basis of KAS method, the activation energy  $E_{\alpha}$  can be determined for each

$\alpha$  using the following expression:

$$\ln \left( \frac{\varphi_i}{T_{\alpha i}^2} \right) = Const. - \frac{E_{\alpha}}{RT_{\alpha i}} \quad (7)$$

The Tang method can be put in the form:

$$\ln \left( \frac{\varphi_i}{T_{\alpha i}^{1.894661}} \right) = Const. - 1.00145033 \frac{E_{\alpha}}{RT_{\alpha i}} \quad (8)$$

The FWO method is given by:

$$\ln \varphi_i = Const. - 1.0518 \frac{E_{\alpha}}{RT_{\alpha i}} \quad (9)$$

In all of the Eqs. (7)-(9) the subscript  $i$  denotes different heating rates. The differential isoconversional method suggested by FR is based on the general form of rate equation, written in its logarithmic form:

$$\ln \frac{d\alpha}{dt} = \ln \varphi \frac{d\alpha}{dT} = \ln A f(\alpha) = \frac{E_{\alpha}}{RT} \quad (10)$$

However, these methods are subject to approximations which can introduce significant inaccuracies in the determination of  $E_{\alpha}$ . A new method for the derivation of activation energies is proposed by Starink [22]. It was shown that this method is an order of magnitude more accurate than the other mentioned methods.

The Starink method is given by:

$$\ln\left(\frac{\varphi_i}{T_{ai}^{1.92}}\right) = Const. - 1.0008 \frac{E_\alpha}{RT_{ai}} \quad (11)$$

By applying the value of  $T_{ai}$  at different transformation stages, the variation of activation energy with  $\alpha$  can be obtained by equations (7)-(11). Figure 6 shows the DTA curves of the 49wt% Al/Nb<sub>2</sub>O<sub>5</sub> powder mixture at different heating rates. In each of the curves, the observed exothermic peak at about 600°C is associated with the reaction between Al and Nb<sub>2</sub>O<sub>5</sub>. Interestingly, the peak position is influenced significantly by the heating rates. This increase in the height of peaks is usual for all exothermic reactions. It is also worthy to note that the reaction occurs before melting of Al and so it can be said the niobium aluminide based composite is formed via a solid state reaction.

The isoconversional methods in equations (7), (8), (9) and (11), which are linear integral methods, were used on the overall transformation data to evaluate the activation energy,  $E_\alpha$ , for reaction (1), and to compare various isoconversional methods. Figure 7 shows the dependence of activation energies of reaction (1) on  $\alpha$ .

As can be seen in this figure, the variation of activation energy by degree of conversion is negligible. Very small variation of activation energy by degree of conversion may be attributed to the unique conversion mechanism during reaction between mechanically activated Nb<sub>2</sub>O<sub>5</sub> and Al. The curves also show that all methods lead to similar values of  $E_\alpha$  and have a good agreement with each other.

FR method which is a linear differential method was also applied in order to determine the dependence of  $E_\alpha$  on  $\alpha$ . Figure 8 shows the changes of  $\ln \varphi_i \frac{d\alpha}{dT}$  versus  $1/T$  for the formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite using FR method. Activation energy can be obtained from the slope of each line.

The results obtained from this method are shown in Figure 9. As can be seen, the change of  $E_\alpha$  with  $\alpha$  is similar to the KAS, FWO, Tang and Starink methods.

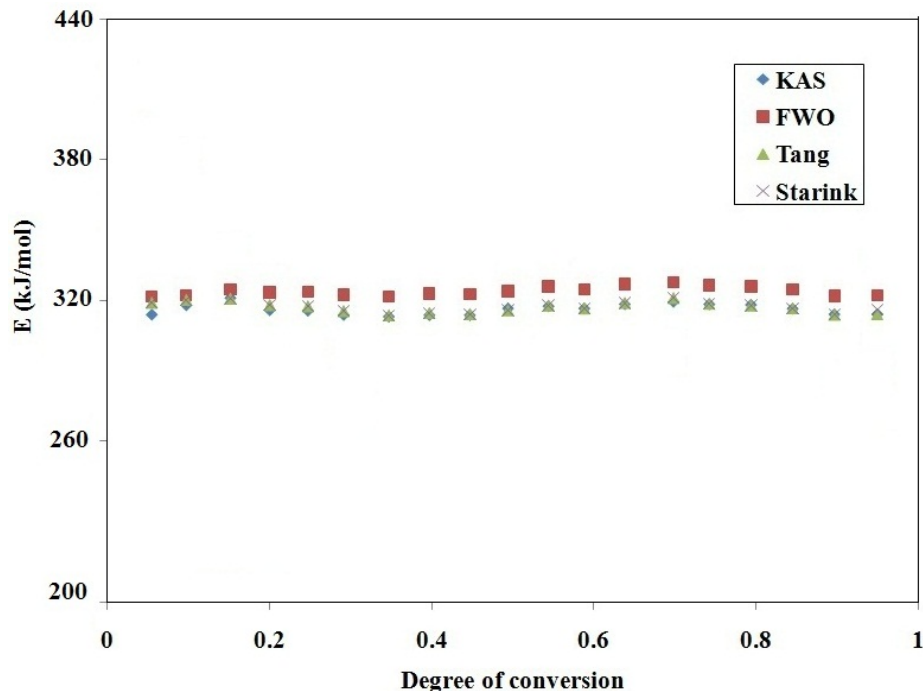
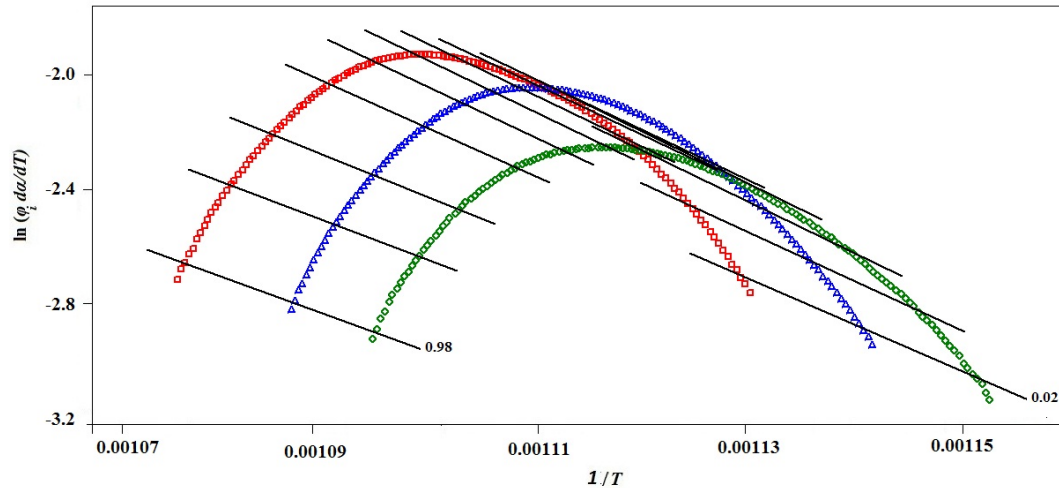
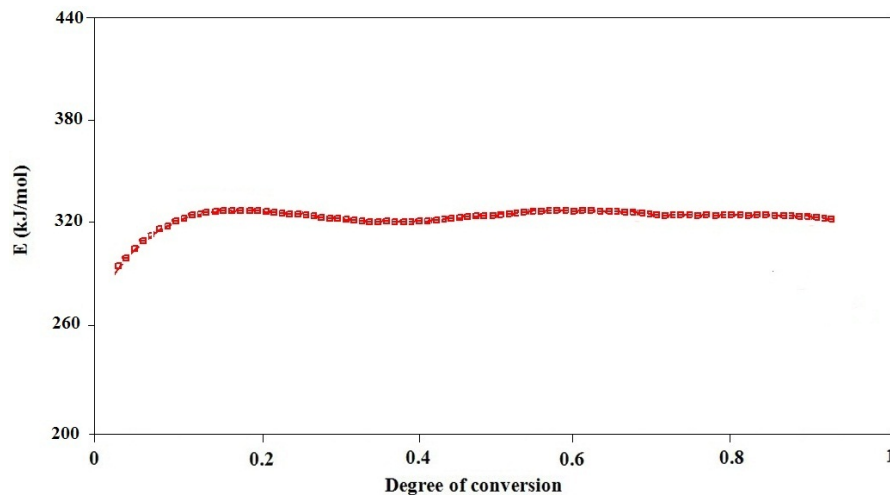


Fig. 7. Dependence of activation energies ( $E_\alpha$ ) for reaction between mechanically activated Nb<sub>2</sub>O<sub>5</sub> and Al powder mixture for formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite on the degree of conversion,  $\alpha$ .





**Fig. 8.** The changes of  $\ln \phi_i \frac{d\alpha}{dT}$  against  $1/T$  in the formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> Composite.



**Fig. 9.** Dependence of activation energies ( $E_\alpha$ ) for reaction between mechanically activated Nb<sub>2</sub>O<sub>5</sub> and Al powder mixture for formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite on the degree of conversion,  $\alpha$ , determined using FR method.

As can be seen, the results indicate that  $E_\alpha$  is not a constant, varying slightly with  $\alpha$ . It is obvious that,  $E_\alpha$  increases to some extent with the degree of conversion,  $\alpha$ , and then keep a constant trend. It has been shown that in some reactions, the FR method is incapable to exact prediction of activation energy. So, it can be interpreted that the increase of activation energy at the onset of reaction is not accurate and can be attributed to the nature of FR method.

#### 4. Conclusions

1- Formation of NbAl<sub>3</sub> based nanocomposite is possible through mechanochemical reaction between Nb<sub>2</sub>O<sub>5</sub> and Al. This reaction takes place in an explosive mode and after about 450min of mechanical alloying.

2- It was revealed that the formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite from the as-blended powder occur in two separate stages. One stage is thermite reaction at 880 °C and the subsequent



stage at 944 °C which is the synthesis of NbAl<sub>3</sub> intermetallic.

3- DTA results of milled powder for 5h showed that the mechanical activation leads to decrease the temperature of formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite from 944 °C to 625 °C due to the increase in grain boundary and defects which need a lower energy and provide more rapid pathways for solid-state diffusion and increase in contact areas between the particles.

4- DTA result of this mixture shows that the thermite reaction and synthesis reaction of NbAl<sub>3</sub> take place simultaneously.

5- Kinetic study showed that the activation energy of formation of NbAl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> composite does not vary with  $\alpha$ , suggesting unique reaction mechanism between mechanically activated Nb<sub>2</sub>O<sub>5</sub> and Al powder mixtures. The results obtained from different method, KAS, Tang, FWO and Starink are in a good agreement and comparable with the FR method.

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