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# Kinetics of High Temperature Reaction in Ni–Al System: Influence of Mechanical Activation

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High temperature ( $>1000$  K) reaction kinetics in the stoichiometric (1:1 by molar ratio) Al–Ni system was investigated by using the, so-called, electrothermal analysis (ETA) method. ETA is the only technique that allows studying kinetics of a heterogeneous gasless reaction at temperatures above the melting points of the precursors. Special attention was focused on methodological aspects of the ETA method. Two different reaction systems were studied: (i) initial Al/Ni clad particles; (ii) the same powders but after 15 min of high energy ball milling. Analysis of the obtained results leads to the conclusion that such mechanical treatment decreases the apparent activation energies of the reaction in the Ni–Al system, from  $47 \pm 7$  kcal/mol for the initial powder to  $25 \pm 3$  kcal/mol after ball milling. Comparison of these data with those reported previously was also made.

## 1. Introduction

High energy ball milling of powders is widely used for preparation of amorphous and other advanced alloys.<sup>1</sup> This approach can be applied for both the equilibrium phases as well as to mixtures with large negative heats of mixing, including highly reactive systems. Typically, several hours of high energy ball milling (HEBM) is required to produce such materials. A similar approach but with shorter processing time is used to enhance the powder mixture reactivity. This so-called arresting ball milling or mechanical activation process, leads to a decrease of self-ignition temperature in the reactive mixture or to an increase of the reaction front propagation velocity, as well as allows a self-propagating mode for the compositions that do not burn without the mechanical treatment.<sup>2–4</sup>

An exothermic mixture of nickel and aluminum powders (with 1:1 molar ratio, adiabatic combustion temperature 1910 K) was investigated by several research groups, and it was proved that relatively short-term (minutes) of HEBM results in the enhancement of systems reactivity.<sup>4–6</sup> The question is if indeed high energy ball milling leads to the reaction activation? Or in other words, does HEBM decrease the energy barrier for chemical reactions? To address this issue, it is required to compare values of the activation energy for conventional and mechanically activated Ni + Al mixtures. However, it is well-known that for the conventional Ni + Al mixture reaction self-initiated at temperatures around the melting point of Al and proceeds extremely rapidly in the temperature interval 912–1910 K. Thus it is impossible to measure kinetics of such rapid high-temperature reaction by using conventional isothermal methods, as well as TGA/DTA/DSC based approaches.

The only known up-to-date method that allows measuring rapid high-temperature kinetics for gasless heterogeneous reactions is termed the electrothermal explosion approach (ETA), the fundamental basis of which was developed many years

ago.<sup>7,8</sup> In this work, the ETA method was used to define the differences in kinetics of interaction for stoichiometric Ni:Al = 1:1 mixtures that were subjected to the different levels of high energy ball milling. Special attention was paid to the establishing of accurate physical basis for the application of ETA methodology for kinetics study in such systems.

## 2. Experimental Methods

Experiments were conducted with Al–Ni clad particles (Federal Technology Group, Bozeman, MT), i.e., 30–40  $\mu\text{m}$  diameter Al particles coated by a 3–3.5  $\mu\text{m}$  layer of Ni, which corresponds to an approximately 1:1 Al:Ni atomic ratio. A PM100 single-station Planetary Ball Mill (Retsch GmbH, Germany) was used for high-energy milling of the powder. The rotational speed of the mill is adjustable from 100 to 650 rpm. A special locking device and jar lid allows one to evacuate the vial and perform grinding operations in an inert atmosphere (e.g., argon). The mass to charge ratio between milling media (3 mm steel balls) and powder was 2:1. The durations of treatment investigated were 2, 5, 10, and 15 min. From these powder mixtures the cylindrical samples (diameter 5 mm and height 17 mm) were cold pressed to relative densities,  $\rho$ , in the range of 75–85% of the theoretical value. These samples were used to measure kinetics of reaction in Ni–Al system by using ETE approach.

An SDT-2960 (TA Instruments, New Castle, DE) was used to conduct simultaneous DTA analysis on the reactive mixtures under the following conditions: argon gas flow at 80 cc/min, heating rates 5, 10, and 50 K/min in the temperature range 300–1000 K. The microstructure and phase composition of the powders before and after milling were examined by different material science techniques, including field emission scanning electron microscopy (EVO 50 Series; Zeiss, Peabody, MA), energy-dispersive spectroscopy (INCAx-sight Model 7636; Oxford Instruments, Concord, MA), and X-ray diffraction (Scintag X1 Advanced Diffraction System; Scintag Inc. USA, Cupertino, CA).

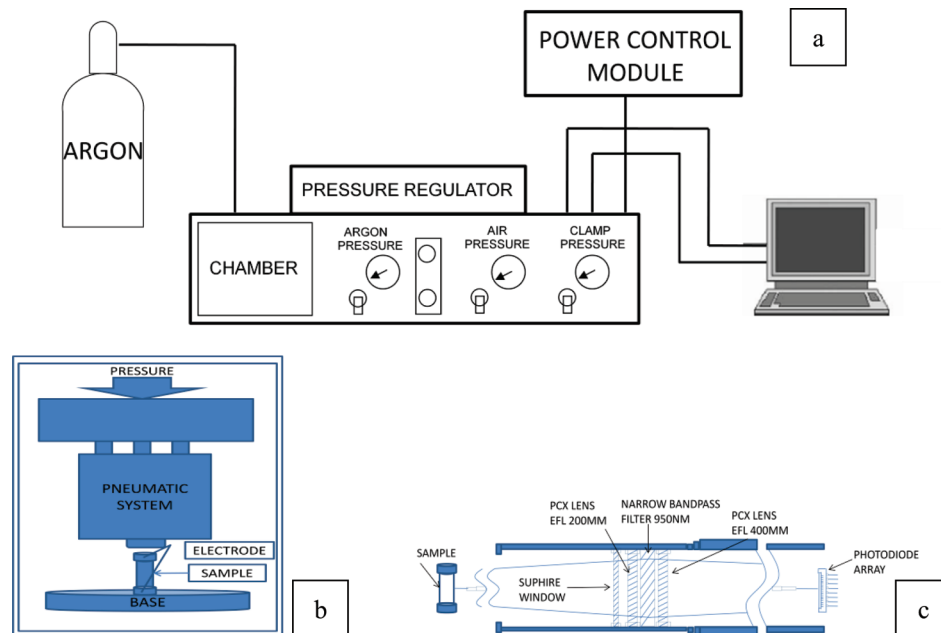
**2.1. ETA-100 Setup.** Electrothermal analysis (ETA) is an advanced method for studying the kinetics of rapid high-

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**Figure 1.** Schematic representation of the ETA-100 setup (a) with reaction chamber (b) and optical system (c).

temperature reactions in condensed heterogeneous systems. This method is based on the phenomenon of electrothermal explosion (ETE), i.e., the ability of reaction for self-initiation along the bulk of the sample under the conditions of its rapid preheating by an electrical current passing through the media. Note that external Joule heating is used only on the initial stage of the process and then the current is shut off while further temperature change occurs solely due to the heat of the exothermal reaction. The output of the ETA-100 apparatus (ALOFT, Inc., Berkley, CA) is a set of temperature–time profiles that are measured in different spots along the sample height and that can be processed to determine the kinetic parameters of the reaction. Let us consider in detail the experimental procedures used in this work for kinetic data extraction.

The schematic representation of the ETA apparatus is shown in Figure 1a. The sample pressed from the desired reactive mixture is clamped between two electrodes (Figure 1b) that are installed in the reaction chamber and connected to a dc power supply. The latter is used for rapid preheating of the sample up to the ignition temperature. The lower electrode is static, while the upper one can move in the vertical direction with the help of a pneumatic piston system. The gas, inert (e.g., argon) or reactive (e.g., nitrogen), can be purged through the chamber to reach the desired ambient environment. The optical system (Figure 1c) allows one to split and focus the light flux from the set of spots on the sample surface on a line of detectors (16 channels) with optical filters, which have a central wavelength of 950 nm with a band half-width of 25 nm. The output signals from detectors are used to measure the time–temperature profiles along the sample height. The optical system also enhances the image size of the sample twice in the plane of linear detectors, which register flux at 16 different points with a span of 1 mm. Thus, the space resolution of the temperature–time profile registration along the sample height is 0.5 mm, while time resolution of detectors is  $10^{-5}$  s and after calibration the measured temperature range is 900–3000 K. The photodiode outputs are converted into digital forms providing the following data: thermograms, i.e., space–temperature distribution every  $\Delta t$  ms; temperature profiles, time–temperature history of each of 16 channels; 3D-thermograms, space–time–temperature map.

**2.2. Methodology for Kinetics Measurement.** Typically the following model for the thermo explosion is considered.<sup>8</sup> A cylindrical sample (radius  $r_0$  and height  $l$ ) with initial temperature  $T_{in}$  immediately is exposed to temperature  $T_0$ . Heat exchange with the ambient environment follows the Newton law  $q = \alpha(T - T_0)$ . The heat source in the system is a one-step irreversible reaction (e.g.,  $Ni + Al \rightarrow NiAl$ ), which is characterized by the degree of conversion:

$$\eta = \frac{(m_0 - m)}{m_0}$$

where  $m_0$  and  $m$  are the initial and instantaneous amounts of the reactive substance, respectively. The rate of chemical reaction can be expressed as follows:

$$\frac{d\eta}{dt} = k_0 \exp\left(-\frac{E}{RT}\right) \cdot \phi(\eta) \quad (1)$$

where  $\phi(\eta)$  is a kinetic function, which for the  $n$ -order reaction can be written as  $\phi(\eta) = (1 - \eta)^n$ . Under the above conditions, kinetic eq 1 and the following heat transfer eq 2 are described by the thermal explosion process:

$$c\rho \frac{\partial T}{\partial t} = Qk_0 e^{-E/RT} \cdot \phi(\eta) + \lambda \cdot \left[ \frac{1}{r} \frac{\partial}{\partial r} \left( r \cdot \frac{\partial T}{\partial r} \right) + \frac{1}{r^2} \cdot \frac{\partial^2 T}{\partial \psi^2} + \frac{\partial^2 T}{\partial z^2} \right] \quad (2)$$

with initial conditions

$$t = 0 \quad T = T_0 \quad \eta = 0$$

and boundary conditions

$$r = r_0 \quad -\lambda \frac{\partial T}{\partial r} = \alpha(T - T_{\text{in}})$$

$$r = 0 \quad \frac{\partial T}{\partial r} = 0$$

$$z = \pm \frac{1}{2l} \quad T = T_{\text{in}}$$

As compared to the above classical scheme in the case of ETE, Joule preheating ( $q_{\text{el}}$ ) of the sample rapidly ( $>10^4$  K/s) brings the system to the temperature  $T_0$ , after which the current is turned off. It is experimentally proved that under such a high heating rate the reaction cannot proceed to a significant degree of conversion during this stage, thus it can be considered as a *preheating of the inert body*.<sup>9</sup> A second advantage of Joule preheating is that it leads to the *uniform distribution of temperature* along the sample volume; thus one may neglect the last two terms in the eq 2. Finally, it is also experimentally proved (see below) that for under ETE (the process duration is of the order of milliseconds) the rate of heat accumulation ( $\rho c(dT/dt)$ ) in the middle part of the sample during thermal explosion (TE) is much faster than the rate of heat conduction ( $\lambda(\partial^2 T/\partial z^2)$ ) and thus, after reaching  $T = T_0 = T_{\text{cur,off}}$ , this process can be considered an *adiabatic thermal explosion*. It is logical to assume that in the initial stage of reaction the contribution of the degree of conversion  $\phi(\eta)$  to the kinetics is negligible compared to the strong temperature dependent part. Under the above assumptions by *accurate* measurements of the temperature–time profiles in the middle of thermal explosion spot and plotting obtained data in Arrhenius form, i.e.,  $\ln(dT/dt) = F(1/T)$ , from the slope of the plot one can obtain the apparent activation energy of the reaction. Thus rapid uniform sample preheating to high temperatures and the ability of accurate measurements of temperature–time profiles along the sample height are the key factors that define the uniqueness of the ETE approach for high temperature kinetics in heterogeneous gasless reactions. It is also important that the above analysis does not require knowledge for heterogeneous media thermal conductivity, which is difficult to measure.

**2.3. Procedure for Experimental Data Analysis.** Figure 2a shows an example of the temperature–space distributions along

the sample height measured every 0.1 ms. It can be seen that the process involves two major stages: (i) initial “inert” Joule preheating (lower part of the profiles), where the temperature is practically uniformly distributed along the sample; (ii) explosion—at some spot (in the considered case it is channel 11) temperature rapidly ( $2.5 \times 10^5$  K/s) increases, reaching its maximum (2000 K) in 3 ms. Also note that the electrical current has been turned off at  $T = 1270$  K, which is below the TE initiation temperature ( $\sim 1350$  K).

Data in Figure 2a are used to obtain important information: (i) to verify if indeed the thermal explosion (TE) mode has been accomplished (contrarily, local ignition may occur followed by reaction wave propagation); (ii) to select experiments where TE occurred in the middle part of the sample and thus to exclude the influence of heat loss ends effect; (iii) to define the spot (channel no.) where TE initiates.

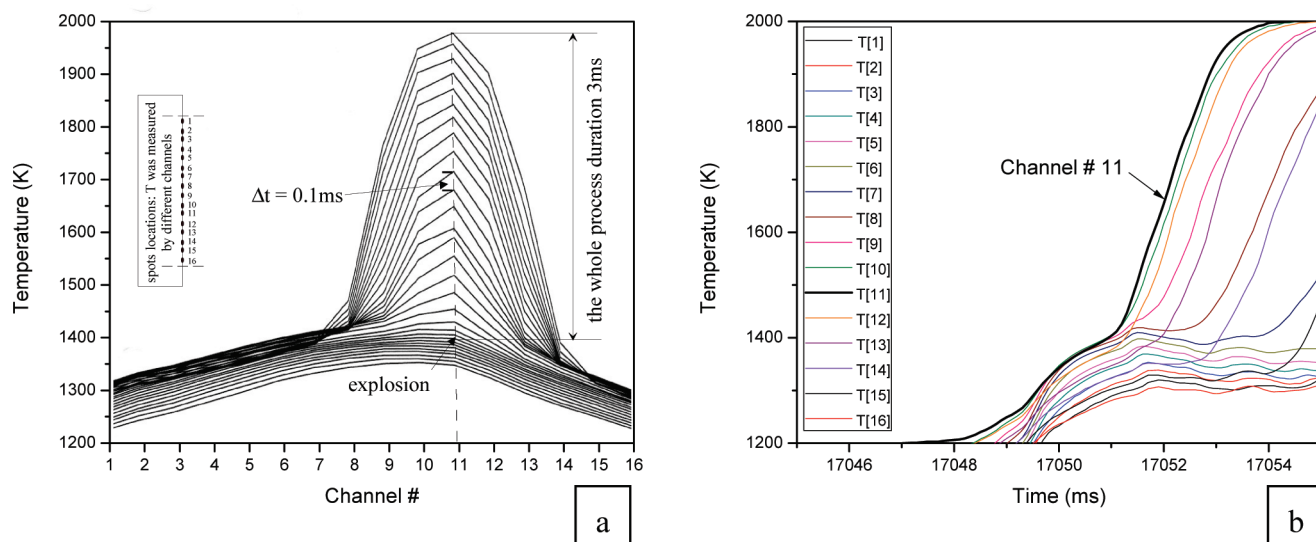
The statistical treatment of such  $T = F(z)$  profiles showed that the dependence of the temperature distribution along the sample height can be presented as follows:

$$T(z, t_i) = T(0, t_i) - \Delta T(z, t_i) = T(0, t_i) - \left[ \frac{\Delta T_0(z, t_i)}{l^2} \right] z^2 \quad (3)$$

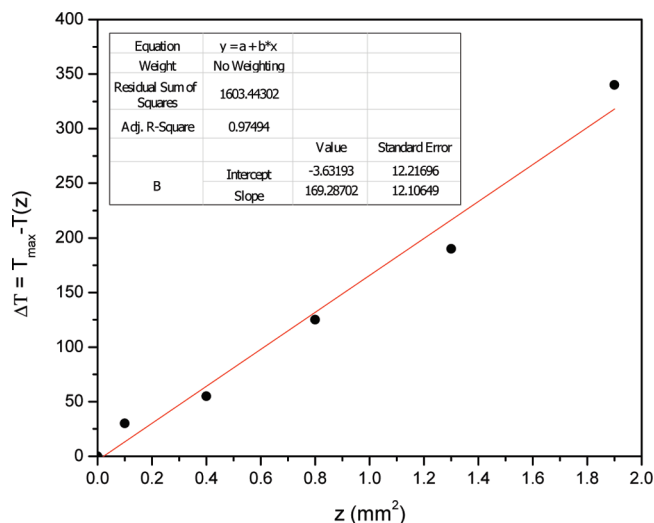
where  $T(0, t_i)$  is the maximum sample temperature at  $t = t_i$ ;  $\Delta T_0(z, t_i)/l^2$  is a scaled up parameter that can be defined by treatment of the experimental data. The typical dependence of  $\Delta T(z, t_i) = T(0, t_i) - T(z, t_i) = F(z^2)$  is shown in Figure 3. First it can be seen that indeed it is a linear dependence and the value of the slope,  $\Delta T_0(z, t_i)/l^2 = 170$  K/mm<sup>2</sup>. In this case,  $d^2T/dz^2$  is about 340 K/mm<sup>2</sup>. The typical value for thermal diffusivity ( $\alpha = \lambda/\rho c$ ) of investigated heterogeneous media is on the order of 0.1 cm<sup>2</sup>/s.<sup>10,11</sup> Thus the heat conduction term in eq 1 can be estimated as follows:  $(\lambda/\rho c)(\partial^2 T/\partial x^2) = 3.4 \times 10^3$  K/s.

This value should be compared with a typical rate for temperature change during the TE process that is for all investigated mixtures on the order of  $dT/dt = 1.2 \times 10^5$  K/s, thus being 2 orders higher than the heat transfer rate. These estimations allow us to consider conditions for the TE process as adiabatic.

On the next stage one has to use temperature–time profiles shown in Figure 2b. The data in the vicinity of the TE initiating



**Figure 2.** (a) Space–temperature distribution and (b) temperature–time profiles of ETE process.

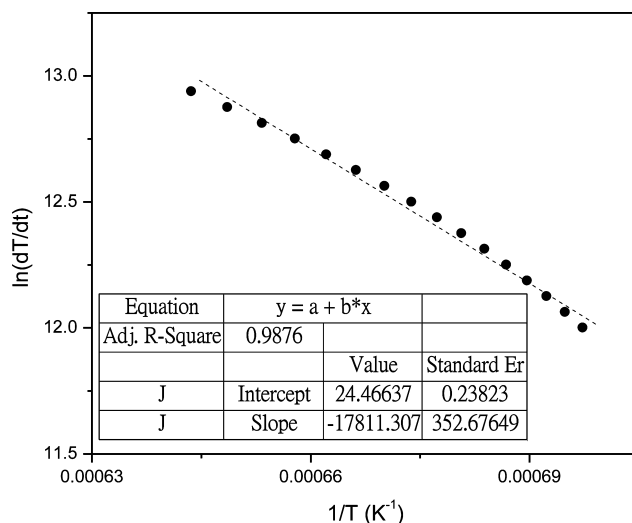


**Figure 3.** For estimation of the heat conduction rate along the sample.

*spot* is of most importance (i.e., channels 9, 11, 12) to obtain the average temperature–time history for the TE process (see Figure 4a). The derivatives function,  $dT/dt$ , indicates the part of the temperature profile (region I) where the degree of conversion ( $\eta$ ) is not high enough and kinetics can be approximated by an Arrhenius zero-order reaction function, as discussed above. In region II, while the temperature continues to increase, its grow rate decreases due to the burned-out part of the kinetic function, i.e.,  $\exp(-E/RT)(1 - \eta)^n$ . The part of the temperature profile corresponding to region I is approximated by the exponential function (Figure 4b), typically with an adjustable  $R^2$  factor  $>0.96$ . Finally, obtained the fitting curve is used to extract kinetics, by creating an Arrhenius type of plot  $\ln(dT/dt) = F(1/T)$ , as shown in Figure 5. The slope of this plot allows calculating the reaction activation energy ( $E_{ac}$ ) of the reaction. Thus Figures 2–5 describe the procedure used in this work to extract kinetics data (primarily apparent  $E_{ac}$ ) for the chemical reaction in conventional and mechanically activated Ni–Al systems.

### 3. Results and Discussion

Figure 6a shows two typical temperature–time profiles obtained in the thermal explosion spot in the middle of the sample for a conventional clad-particle mixture (curve 1) and

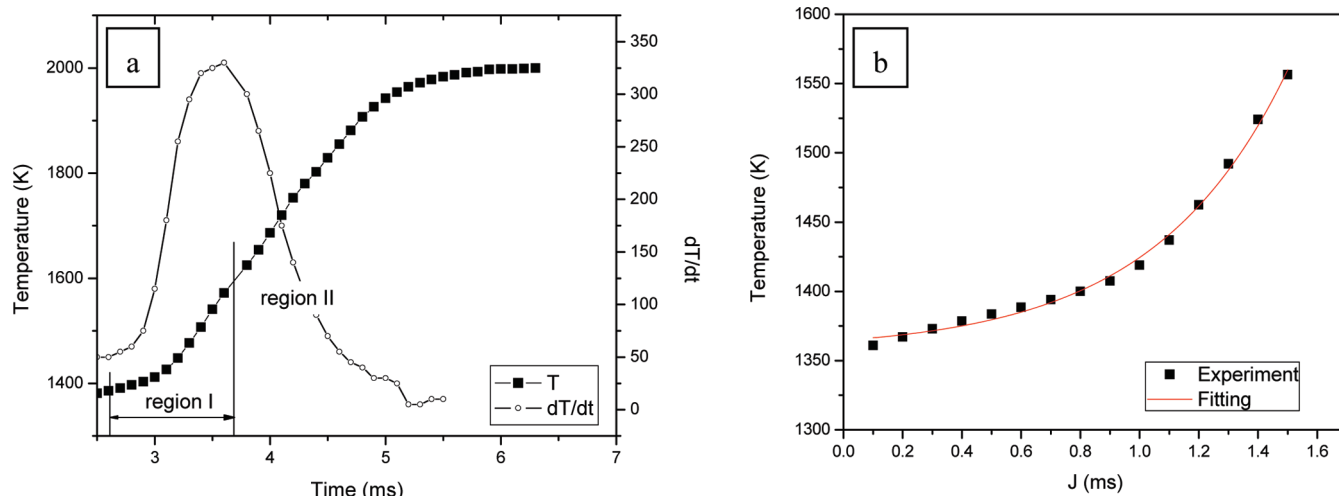


**Figure 5.** Typical Arrhenius plot to extract reaction apparent activation energy.

for a mixture after 15 min of high energy ball milling (curve 2). Figure 6b provides information about the typical rates of temperature change during TE in these systems. It can be seen that, for the initial clad particle (Figure 6a, curve 1) in the range 1000–1750 K, temperature changes non-monotonically and two  $dT/dt$  peaks with absolute values on the order of  $(1-2) \times 10^5$  K/s are observed (Figure 6b, curve 1). In turn, in this range temperature increases monotonically for a mechanically activated mixture and its  $dT/dt$  changes extremely rapidly,  $dT/dt \sim (5-6) \times 10^5$  K/s.

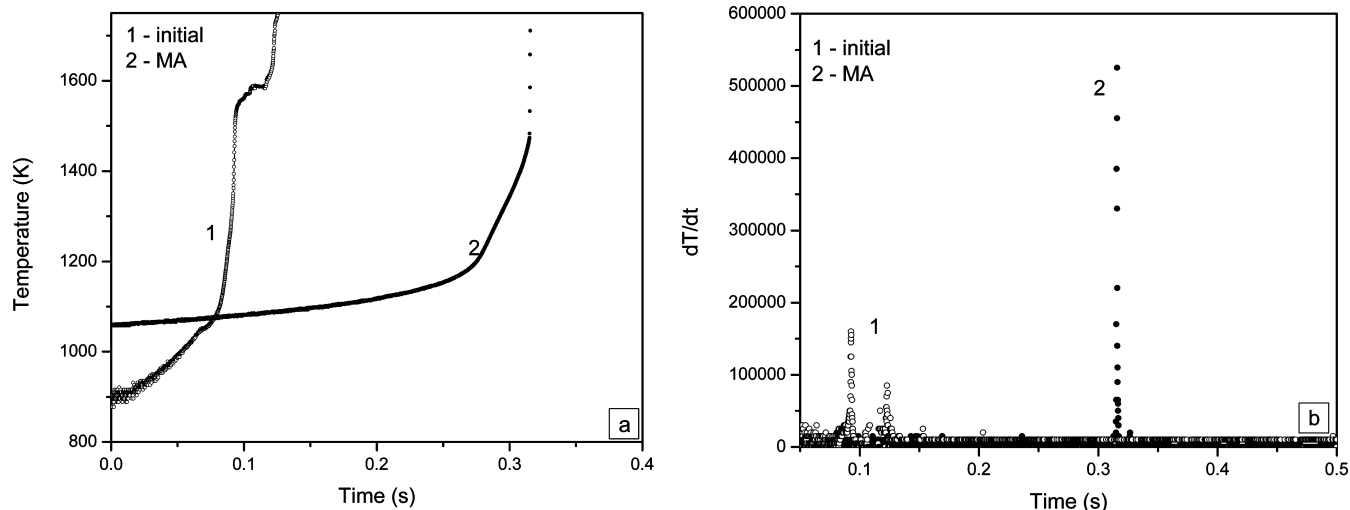
The Arrhenius function for the TE of the initial clad-particle mixture, plotted on the basis of the data shown in Figure 6a, is presented in Figure 7. The plot can be divided into four specific regions: (i) below  $T = 1250$  K, which corresponds to the thermodynamically calculated degree of conversion of  $\eta \sim 27\%$ , where a monotonic increase of function is observed; (ii) 1250–1400 K, which characterized by a temperature plateau; (iii) 1400–1580 K, with again monotonically function increases ( $\eta \sim 57\%$  at  $T = 1583$  K); (iv) above 1580 K, where the Arrhenius function decreases.

Such complex function behavior can be explained by taking into account the following well-known specifics for reaction in the Al/Ni clad system. As shown in previous works,<sup>12–14</sup> the

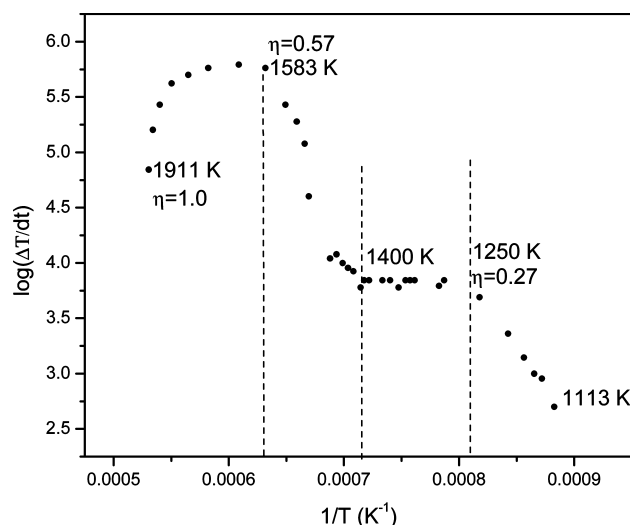


**Figure 4.** Typical average temperature and  $dT/dt$  profiles for ETE experiments (a); region I of the  $T$ -profile and its exponential fitting (b).

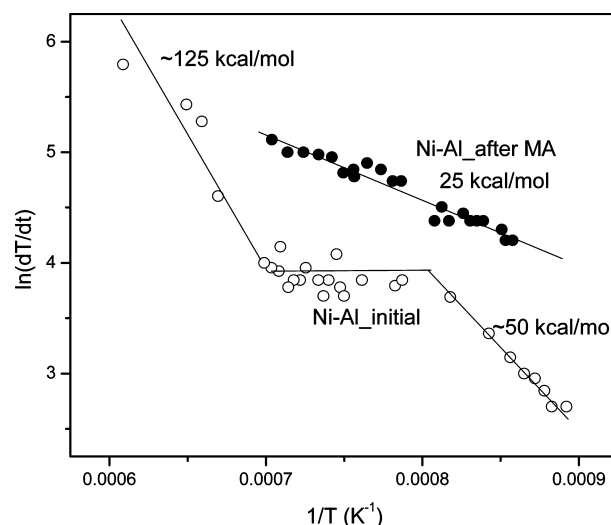




**Figure 6.** Typical temperature profiles for initial and mechanically activated samples (a) and rates of temperature change (b) during TE process in these systems.



**Figure 7.** Typical Arrhenius plot for reaction in the Al clad by Ni system.



**Figure 8.** Arrhenius plots for reactions in Al clad by Ni systems before and after high energy ball milling.

noticeable interaction in Al/Ni clad particles starts at about the melting point of Al (933 K) and proceeds in two steps. First step is dissolution of the Ni layer in the core of melted Al. However, at some temperature point, due to thermal mechanical stress, the solid Ni core cracked, followed by liquid aluminum spreading out of the shell and its reaction with the rest of solid Ni layer until it melts at 1728 K. Thus the existence of three first stages on the Arrhenius plot can be attributed to these steps of clad particle interaction, i.e., (i) 933–1250 K, reaction before Ni layer cracked; (ii) 1250–1400 K, cracking the layer; (iii) 1400–1580 K, reaction after the solid layer cracked. Last stage (>1580 K) is obviously related to the conditions with high degree of conversion where simplified model used in this study does not work. As shown above, the  $\phi(\eta)$  function should be taken into account to obtain kinetic data in this temperature range.

The typical Arrhenius plots for initial and mechanically activated (15 min of high-energy ball milling) samples with corresponding linear approximations are shown in Figure 8. Statistical treatment of such curves, obtained for at least five samples for each composition, shows that two different apparent activation energies are characterized combustion of initial Al/Ni clad particles:  $47 \pm 7$  kcal/mol for the low temperature first stage, and  $125 \pm 20$  kcal/mol for the relatively high temperature

(>1400 K) third stage. For the mechanically activated system one value of activation energy ( $25 \pm 3$  kcal/mol) describes the reaction behavior in the entire range of investigated temperatures. And this value is much lower than those measured for nontreated system.

Thus it is shown that the change of the apparent activation energy during interaction in the Al/Ni clad system, obtained by using ETA method, qualitatively fit the observation on the microstructure transformation during combustion of such particles, capturing such features as changing of the reaction mechanism owing to rupture of the nickel layer. It is also important that the actual value of the activation energy,  $E_1 = 46 \pm 7$  kcal/mol, obtained for the low temperature stage of reaction (950–1250 K) is in good agreement with those reported in the previous works for the reaction of the Al + Ni powder mixtures. Indeed, the activation energy  $38 \pm 1$  kcal/mol was obtained<sup>15</sup> for this system by using high-speed DTA. Also, the activation energy for Ni dissolution in molten aluminum appeared to be 40 kcal/mol.<sup>16</sup>

A significant increase of activation energy ( $E_{II} = 125 \pm 20$  kcal/mol) at higher temperature (1250–1600 K), observed in this work, may be related to the changes of the reaction mechanism. For example, reported data on the activation energy

of diffusion of Al to Ni in the temperature range 1375–1580 K is 135 kcal/mol,<sup>17</sup> which is close to the  $E_{II}$  value. Such a comparison allows one to conclude that the ETA approach is very sensitive and a reliable method for measuring high-temperature kinetics in heterogeneous systems.

It is no surprise that after high energy ball milling only one stage reaction was observed. Indeed, it was previously shown<sup>4,5</sup> that HEBM leads to the formation of composite metal particles that involves nickel layers immersed in an aluminum matrix. Thus reactants are nicely mixed with very high contact surface area, and no significant morphological transformation takes place during combustion of such particles. It is more important that for the first time ETA provides direct experimental evidence that HEBM results in a significant decrease of apparent reaction activation energy. The obtained value  $E_{HEBM} = 25$  kcal/mol is almost 2 times less than  $E_I$ , measured for the low temperature stage and 5 times less than  $E_{II}$  measured for the high temperature stage of interaction in the initial Al/Ni clad system.

Let us mention one more interesting aspect. The activation energy for HEBM Al/Ni clad particles at low temperatures (500–900 K) was recently determined by the conventional DTA method.<sup>5</sup> The value of  $E = 25 \pm 2$  kcal/mol was reported for solely the solid state reaction (913 K is the eutectic temperature for the Ni–Al system). However, this value fits well the value of  $E_{HEBM}$  obtained in this work by the ETA method for much higher temperatures. Typically, one may expect that after melting of the reagents the reaction activation energy in the system should decrease.<sup>18</sup> Obviously it is not the case for the reactions in the media prepared by high energy ball milling. The last conclusion is worth investigating in future works in more detail.

#### 4. Concluding Remarks

This work demonstrates that ETA is a powerful tool to investigate high temperature (>1000 K) kinetics in heterogeneous gasless systems. It is important that the above analysis does not require knowledge for heterogeneous media thermal conductivity, which is difficult to measure. Also, it was for the first time directly revealed that high energy ball milling indeed leads to the decrease of the apparent activation energy of the reaction. It means that the term “mechanical activation” in some sense is a suitable term to describe the observed phenomenon. On the other hand, it looks like such mechanical treatment changes the main route of chemical interaction. Without ball milling, thermal explosion (TE) occurs at temperatures above eutectics (912 K) in the investigated system; thus the chemical route primarily involves solid–liquid reactions. After mechanical treatment the TE initiates at much lower temperatures and solid state reactions are predominantly responsible for the process. Thus it is possible that the observed change in apparent activation energy is related not to the real activation of the

reaction but to the change of the route for chemical interaction in the system. Additional studies are required to verify the above hypothesis.

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