Acoustic emission during combustion of Ni-Al composites

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Abstract. The paper studies the effect of acoustic emission (AE) during combustion of Ni and Al powder mixtures as well as Ni and Al twisted wires. The acoustic measurements show that AE occurs at the moment of ignition of the samples and moderates after the combustion is completed. The features of AE signals are low-ordered discrete pulses with duration of (0.5÷4.5) ms and regular vibrations with a frequency of (860÷900) kHz. The possible mechanisms of generation of acoustic vibrations in propagating combustion waves are analyzed.

1. Introduction
Powder and thread-like metal composites such as Al-Ni, Al-Pd etc. are of interest as pyrotechnic additives which increase the combustion rate of solid jet fuels [1-3] and also as reactive systems for the production of functional materials by self-propagating high-temperature synthesis [4]. The success of both applications is mainly determined by understanding the combustion kinetics of composites and the effective means of monitoring the reactions.

The measurement of acoustic emission (AE) refers to the known methods for direct diagnostics of destruction of solids [5, 6], phase transitions, chemical reactions [7, 8], and combustion of gases [9, 10]. Analysis of AE characteristics is a source of important information on the dynamics of rapid conversions in gas and condensed systems, and the mechanism for rearranging the internal structure of substances.

The effect of generation of acoustic vibrations was previously observed in wave reactions of condensed systems [11, 12], but it has not been fully investigated so far. The purpose of our work is to study the features of AE during the combustion of Ni-Al composites.

2. Materials and procedures
Powder mixtures with a composition of Ni+31.5 wt.% Al and thread-like composites in the form of Ni and Al twisted wires with a metal ratio of Ni+(28÷30) wt.% Al were used for the study. The components of the mixture were nickel (PNK-L5, manufactured by the NORILSK NICKEL Company, Russia) and aluminium (ASD-4, manufactured by the RUSAL Company, Russia). For nickel the content of the basic substance is 99.85 wt.%, and the particle size is not more than 20 μm. For aluminium the content of the basic substance is 99.7 wt.% and the particle size is not more than 10 μm.

The samples of the mixture were pressed into cylinders with a diameter of 20 mm, a length of 30 mm, and porosity of 40-50%. The length of the twisted wires was 30 mm, the diameter of the wires...
was as follows: Ni-0.4 mm, Al-0.4 mm. The content of the basic substances of the wires was more than 99.3 wt.%. A schematic of the AE measurement during the combustion of composites is presented in figure 1. The samples were ignited by a tungsten electric spiral. The moments of the onset and completion of combustion were recorded by the signals of thermocouples embedded into the upper and lower parts of the sample. The process was conducted under argon at a pressure of $10^5$ Pa and controlled by video recording.

Acoustic vibrations, generated by the combustion of the powder mixture, through a waveguide were transferred to an acoustic sensor (piezoelectric ceramics CTS-19: the sensitivity threshold of acoustic flux $-10^{-16}$ W/cm²). A tungsten wire 0.5 mm in diameter and 120-150 in length, one end of which was connected to the sample and the other end was connected to the acoustic sensor, was used as a waveguide.

The electrical signal of the sensor, proportional to the vibration intensity of the powder sample, was transmitted through an amplifying system (bandwidth is in the range of 100 Hz÷10 MHz, the amplification factor is up to 60 dB) and an analog-to-digital converter (LA-n150-14PCI, the sampling frequency is 10 MHz) and was recorded on a personal computer. When a twisted wires was studied, one of its ends was connected to the acoustic sensor, and another end was used to initiate the combustion process. In this case, the reactive system was used as a waveguide.

**Figure 1.** Schematic of the measurement of acoustic emission during the combustion of a powder mixture (a) and a twisted wires (b). (1) tungsten electrical spiral; (2) acoustic waveguide; (3) acoustic sensor; (4) amplifier of the acoustic emission signal; (5) analog-to-digital converter; (6) personal computer; (7) thermocouple; (8) powder mixture sample; (9) twisted wires sample; (10) combustion wave.

**Figure 2.** Structure (a) and frequency spectrum (b) of acoustic signal for the impact of the ceramic ball on the surface of the sample. The energy of impact is 15 μJ.

To test the measuring system, ceramic balls with a diameter of 1.5 mm and weight of $m=7$ mg were dropped onto the surface of the samples from a height $h=(20÷50)$ mm. The impact of the ball simulated a single pulse of acoustic combustion emission. The impact formed a signal in the form of a
packet of increasing and damped vibrations (figure 2a). The frequency spectrum of the signal characterizes a set of mechanical vibrations of an acoustic circuit (sample-waveguide-sensor). The spectrum is in the range of $f=(5\div500)$ kHz (figure 2b). The signal increasing time characterizes the active pulse interval that corresponds to the transfer of mechanical energy from ball to the sample. The signal damping time is a passive interval, when the vibrational dissipation of energy takes place. According to the measurements, the maximum amplitude of the signal is proportional to the impact energy $E=mgh$. The measured coefficient of proportionality makes it possible to estimate the energy AE of combustion.

3. Results and discussion

According to the data of video recording, the combustion of the powder mixture is observed in the form of a narrow thermal wave propagating at a velocity: $U_{c}=46$ mm/s. The combustion wave in the twisted wires is a liquid high-temperature drop with a diameter of up to 2 mm, where the melts of Al and Ni are spontaneously mixed and react with each other. The velocity of the drop moving along the twisted wire (propagation of combustion): $U_{c}=300$ mm/s. Local reaction flashes with a lifetime: $t_{r}=(0.5\div6)$ ms are observed in the combustion waves of both systems (figure 3). The presence of flashes is explained by the thermal instabilities of combustion [13-14].

![Figure 3](image)

**Figure 3.** Video recording frames of the combustion of the powder mixture (a) and twisted wires (b). (1) initial sample; (2) front of combustion; (3) reaction flashes in the combustion wave; (*) direction of combustion.

As acoustic measurements have shown, the AE signal appears at the moment of ignition of the sample and damps after the combustion is completed (figure 4a). The signal structure consists of a set of discrete repeating pulses with duration of $(t_{i})$ and a repetition frequency $(\nu_{i})$ in the intervals as follows: $t_{i}=(0.6\div4.5)$ ms, $\nu_{i}=(200\div1600)$ Hz (powder mixture); $t_{i}=(0.5\div2.5)$ ms, $\nu_{i}=(280\div1700)$ Hz (twisted wire). Taking into account the maximum amplitude of the signal and the calibration of the measuring system, the energy of AE pulses reaches $(1\div10)$ $\mu$J. The discrete AE pulse (figure 4c) is a packet of continuous relaxation vibrations with a frequency of $f>>\nu_{i}$. Qualitative similarity of the frequency spectra of AE combustion (figure 4b) and a modeling signal (occurring during the impact of a ceramic ball with a sample (figure 2b)) are observed in the range of $f=(5\div500)$ kHz. In the range of $f>500$ kHz, the AE combustion spectrum has narrow selective bands near $f=900$ kHz (powder mixture) and $f=860$ kHz (twisted wire), which reflect the generation of regular specific high-frequency acoustic vibrations during reactions. The regular vibrations appear occasionally in the form of individual packets (figure 4d).

Analysis of the obtained data shows that the values of $t_{r}$ and $t_{i}$ are nearly close. This fact suggests that the source of discrete AE pulses is the reaction flashes, where pulsed physical and chemical transformations of substances cause corresponding mechanical strains of the samples. Generation of regular high-frequency acoustic vibrations cannot be explained by the macroscopic reaction process.
Characteristic time of heterogeneous chemical conversions of the Ni-Al composites ($\geq 10^{-4}$s) is significantly less than the period of acoustic vibrations ($\sim 10^{-6}$s).

![Image]

**Figure 4.** Structure (a, c, d) and frequency spectrum (b) of the acoustic signal during the combustion of the powder mixture. (c) discrete pulses of AE; (d) regular vibrations of AE; (*) ignition of the sample; (**) completion of combustion; (o) envelope of the discrete AE pulse.

The source of high-frequency vibrations can be rapid phase transformations of reaction products. An example of such transformations is diffusion-free martensitic transitions which develop for the time $\sim(10^{-6}÷10^{-7})$ s [15]. A probable mechanism of rapid conversion of combustion products is as follows: a metastable supersaturated liquid solution of Ni-Al $\rightarrow$ clusters of a crystalline NiAl compound. In this case, high-frequency vibrations can be caused by sequential periodic accumulation and relaxation of metastable solutions. The latter are continuously formed in the combustion wave. The regular mode of vibrations reflects the effect of synergistic interaction between the parameters of phase transformations and the propagation of acoustic waves in a reactive condensed medium.

Further research is required for deeper understanding the nature of acoustic emission of condensed systems during combustion.

4. Conclusion
Combustion of Ni-Al metal composites is accompanied by acoustic emission which takes place during the propagation of the reaction wave along the sample. AE is observed in the form of discrete pulses with duration of $(0.5÷4.5) \text{ ms}$ and a repetition frequency of $(200÷1700)$ Hz, as well as in the form of regular vibrations with a frequency of $(860÷900)$ kHz. Probable sources of AE are the instabilities of reaction waves (discrete pulses) and rapid diffusion-free phase transformations of metastable combustion products (regular high-frequency vibrations).
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