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Adjustment of induction high-temperature synthesis to in situ synchrotron study of SHS-mixtures on the example of Ti-Al system

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Abstract. The paper discusses experimental complex, which allowing to carry out high-temperature synthesis of mechanically activated powder reagents by a method of induction heating in vacuum and inert environment. On the basis of this complex conducted in situ synchrotron research of the specific features of phase formation dynamics in mechanically activated 3Ti + Al powder composition. High temperature synthesis carried out under conditions of high volume inflammation by means of inductive heating. The developed complex is adapted to the method of dynamic diffractometry in synchrotron radiation beams to register the kinetics of phase transformations in the solid-phase reaction of the components of the powder mixture.

1. Introduction

These days big attention is paid to methods of nanostructured composition materials with targeted properties obtaining. An effective method of obtaining such materials is self-propagating high-temperature synthesis [1-3]. An effective way to influence the structure of initial reactionary furnace charge, thus regulating the kinetics of structural phase transition during the synthesis, is a method of mechanical pre-activation (MA) [4-7]. Background mechanical activation helps change the structure and phase composition of the final product, gives more way to chemical reactions, namely, extending concentration combustion limits, changing the temperature and speed of combustion, inflammation temperature, etc. [8-13].

To initiate the synthesis in a mechanically pre-activated system a heat impulse is required. It will fluctuate considerably depending on the type of powder composition. Another important factor for gaining a homogeneous structure and phase composition is speed of powdered composition heating. One mode to create an initiating temperature with high rate of heating is to apply extreme exposure to the powder system (blast wave, electric impulse), it can influence both macro kinetic parameters of self-propagating high-temperature synthesis and phase structure transfiguration [14-17]. However the most universal extreme treatment is inductive heating with the heat input of the 100 K/s order. The inductive heating method is noted for low energy consumption, high rate heating reaction, simple and reliable equipment, and pure final product [18-20]. But application of the inductive heating method, used for controlled synthesis of new materials with targeted properties, has not been thoroughly studied and it requires more research.

For better understanding of phase transformation during the mechanically activated systems synthesis by inductive heating one should know characteristics of synthesis under dynamic conditions. High speed of the self-propagating high-temperature synthesis implies application of the corresponding experimental research methods of the dynamics of initial composition transformation. Recently in situ research of structural change and chemical dynamics in the combustion zone in real time mode has become possible due to the application of synchrotron radiation. Today the synchrotron radiation method has no alternative in the research of high-rate processes which are made possible by means of:

- high speed of diffraction registration;
- high intensity facilitating operations with reverse beam;
- high special and time resolution of the detector, that altogether provide high accuracy [21, 22].

Many researchers apply synchrotron radiation of «diffraction movie», i.e. a consequential filming of diffractogram series enabling them to observe structural change of matter during its deformation, melting, crystallization, synthesis, etc. [23-25]. High density of synchrotron radiation of monochromatic beam and progress in detector rate resulted in getting an X-ray pattern per microsecond. In a classical registration scheme it is close to the limit, which is determined by the time of charge assembly and relaxation in the detector. Owing to the properties of synchrotron radiation it is possible to apply it in the process of phase and structural material analysis. Short wave length of synchrotron radiation is considered important. Its tolerance is commeasurable to interatomic distance in the points of the lattice.

In this paper a compound of Ti_3Al was chosen for the research. These α_2-phase alloys are widely used in aerospace engineering, automobile industry, shipbuilding and electric power engineering due to their high heat resistance, durability, resistance to aggressive environments, corrosion stability, low density, high resistance to fatigue failure and creep [26-30]. Reasoning from this fact there is a necessity to extend the research of structure and phase formation in Ti-Al system, and also to control the synthesis in order to obtain intermetallic compounds with targeted properties. The paper describes in situ experimental tests of the dynamics of phase formation of mechanically activated 3Ti + Al powder composition with different timing during high temperature synthesis in the mode of dynamic thermal explosion by means of heating the composition in quickly changing electromagnetic fields.

2. Materials and methods

The object of research in this paper is the installation of induction heating. In general, the induction plant consists of a generator and an induction coil – inductor. The most interesting of induction systems for purposes of present study are microwave induction units. The frequency of these induction units exceeds 100 kHz.

Thus, the microwave induction heater 6AV was used as a base for the creation of the experimental complex in this work. Frequency of inductor varies between 400-1100 kHz and the maximum output power is 6 kW. Induction heater is connected to the mains voltage of 220 V.

High-frequency setting automatically adjusts the inverting frequency from the specified range to any inductor.

Thus, in order to register the dynamics of phase formation in process of self-propagating high-temperature synthesis, experimental complex was constructed. It was based on high frequency electromagnetic heating unit 6AV that generates electromagnetic energy in a wide range of power (figure 1). It should be noted that advantage of induction method over classical heating methods (such as muffle furnace or an electric filament) is as follows: induction method allows flash heat of powder composition, and that can be crucial for mechanically activated systems with high content of non-equilibrium defects of structure [12].

Testing operation of the induction heater 6AV was performed on previously mechanically activated powders of titanium and aluminum.

The ratio of the powder used to make the composition was 16 wt.% Al + Ti. Afterwards, mechanically activated treatment of initial powder compound was carried out with planetary ball mill AGO-2.

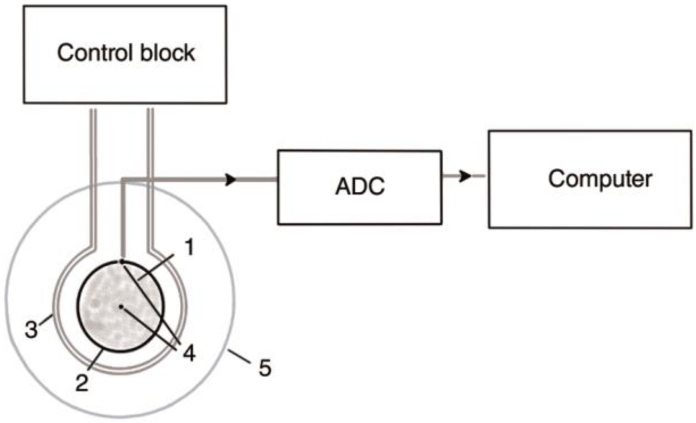


Figure 1. Experimental complex of high speed induction heating: 1 – powder composition; 2 – graphite melting pot; 3 – induction filament; 4 – wolframite thermocouple units; 5 – vacuum lid.

Next stage represented in situ research of dynamics of mechanically activated composition phase formation during high temperature synthesis with the method of dynamic diffraction analysis in synchrotron radiation beams. We used radiation of electron storage ring VEPP-3 of the 5b station («Diffraction movie») in Budker Institute of Nuclear Physics of Siberian Dept. of Russian Academy of Science (Russia, Novosibirsk). Nonstop filming of diffractograms was carried out with monochrome radiation, wave length being λ = 0.1505 nm, range of scanning angles being 30° – 70°, frame interval being 10 sec. System of electron storage ring is presented in figure 2. Method of diffraction analysis is based on radiation of electromagnetic waves with charged particles, particles moving from relativistic velocity within one magnetic field [31, 32].

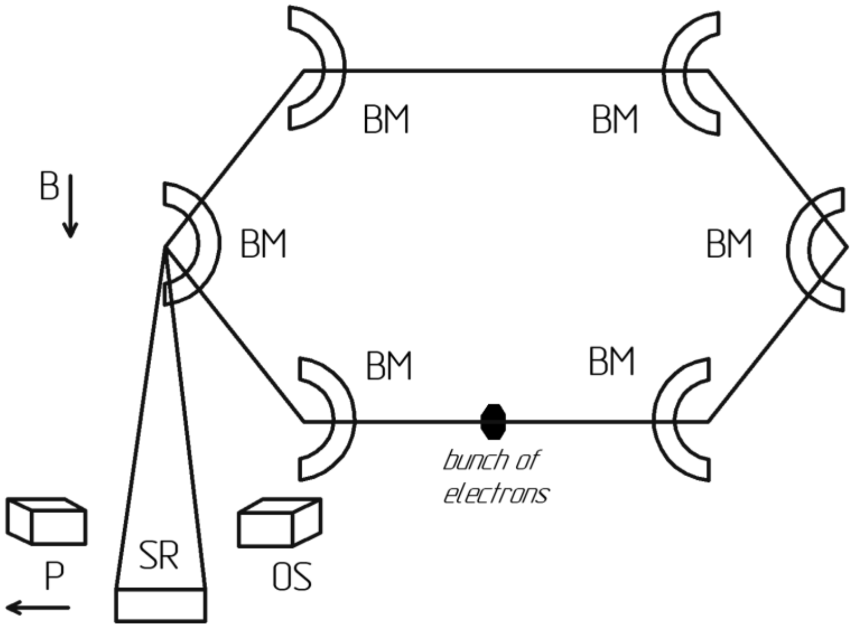


Figure 2. Electron storage ring structure: BM – bend magnets; B – magnetic field; P – photon polarization vector, radiated in electron orbit; OS – output slot, horizontally restricting synchrotron radiation beams.

From injector (linear accelerator) pre-accelerated electrons with relativistic velocity (energy limit equals approximately 300 MeV) get to circular orbit of accelerator and are held there by the magnetic field of bend magnets. Radiation is concentrated in a cone with an 1/Y angle and is tangential to trajectory of radiation point.

$$Y \approx \frac{m\,c^2}{E},$$

where Y – relativistic factor, m and E – mass and energy of particles, c – velocity of light in vacuum. There is a vacuum channel going out of cyclic orbit accelerator which is 10 – 15 m long and carries radiation to the experimental stations.

To conduct a phase analysis of a final synthesis product we used X-ray diffractometer DRON-6 with CuKα-radiation (λ = 0.15418 nm). Diffractograms were registered under similar conditions with scanning interval of 0.05 degrees and time-exposure of 3 seconds at each point. Processing and analysis of data were performed by means of PDWin program pack, designed for automatization of X-ray pattern processing [34].

3. Results and discussion

Preliminary tests have shown possibility of using an induction heater 6AV for study of dynamic synthesis diffractometry in Ti-Al system under extreme heating.

However, this heater must be adapted to conditions of the experiment. For this purpose, the installation was retrofitted with a mobile and productive water cooling system of inductor, as well as a specialized chamber that allows induction heating both in vacuum and in inert environment. Specialized chamber was also equipped with a cooling system. The scheme of the adapted complex is shown in figure 3.

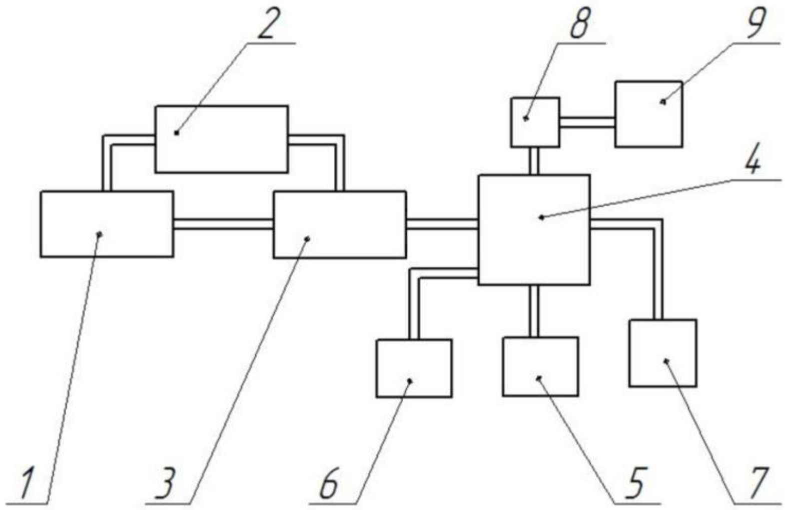


Figure 3. scheme of installation of the experimental complex of high-frequency induction heating:
1 – control unit, 2 – induction heater cooling system, 3 – induction heater, 4 – vacuum chamber, 5 – vacuum pump, 6 – inert gas supply system, 7 – vacuum chamber cooling system, 8 – multichannel LA-2usb board, 9 – personal computer.

One of important components of complex is a specialized vacuum chamber. Inside this chamber is placed a metal crucible with a mechanically-activated mixture of 3Ti + Al and a thermocouple necessary to control synthesis of substance. These temperature measurements were processed by multichannel board LA-2USB, connected to personal computer. Under the influence of magnetic field, powder mixture is heated to a certain temperature, as a result of which synthesis reaction of Ti_3Al compound is initiated.

Thus, mechanically activated composition was placed into graphite melting pot, which is open top cylinder, and then packed down. Then melting pot was isolated with asbestos layer. Signal from thermocouple unit was transmitted to analogue-digital converter (ADC).

System was covered with the vacuum lid, air being extracted and argon pumped down into it. Graphite melting pot was heated to high temperatures by high frequency electromagnetic field, and the system heated powder composition up to 1500 °C and more.

One peculiarity of method of dynamic diffraction analysis is the fact that in order to use it one needs composition materials with open surface to which synchrotron radiation beam fall. Width of synchrotron radiation beam is 2 mm. To carry out experiment a container was constructed, designed as a big hollow cylinder (figure 4).

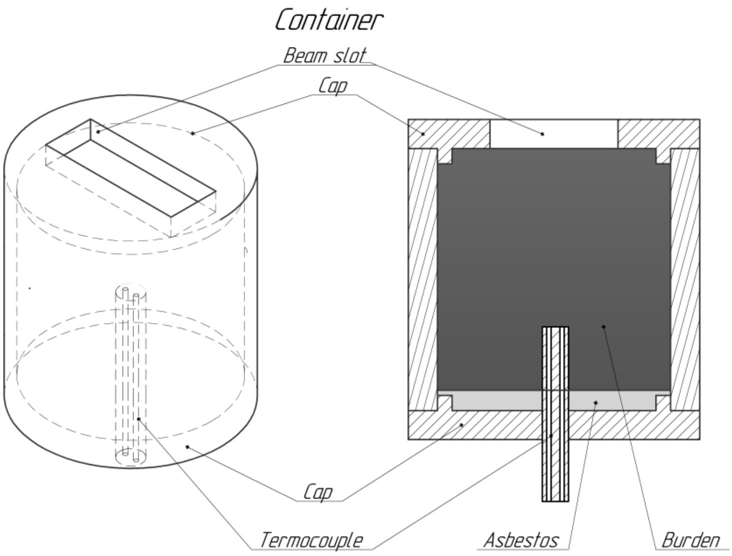
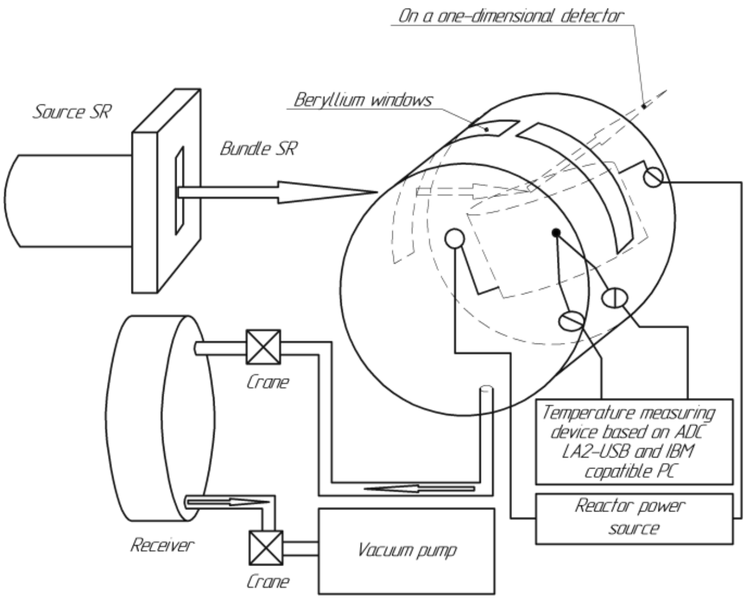


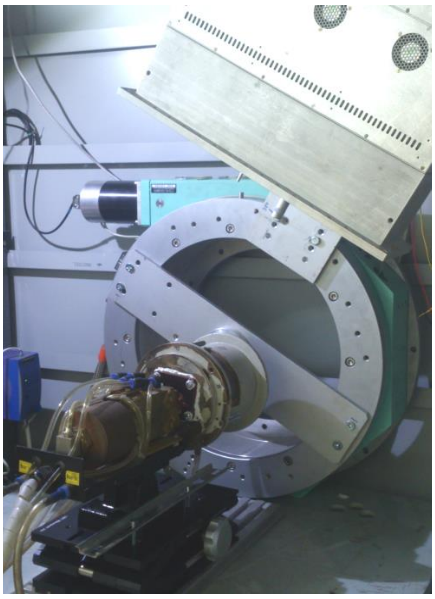
Figure 4. Container, adjusted for the research by means of synchrotron radiation

Specific feature of reactor is sub open surface of composition to provide penetration and reflection of synchrotron radiation beams. It was made possible due to thick plate made of metal with a slot 3 mm wide and 30 mm long. That is enough for a beam to fall to furnace charge surface and to be reflected at angle of 35-40 degrees. The use of metal plate provides additional fixation of composition surface at one level during heating and active chemical reactions.

Finally experimental complex was mounted to unit of 5b synchrotron radiation station of the VEPP-3 electron storage ring (figure 5). Filming of in situ dynamics of phase formation («diffraction movie») was started from the begging of system heating. The heating was switched off when reacting furnace charge reached its maximum temperature with consequent cooling to room temperature.



(a)



(b)

Figure 5. «Diffraction movie» experimental station 5b of the electron storage ring VEPP-3: (a) Experimental complex for the registration of phase formation of titanium aluminide in the process of inductive heating in the mode of thermal explosion; (b) exterior view of the station with a mounting experimental device based on the inductive heating unit.

Figure 6 represents in situ dynamics of phase formation in conditions of inductive heating of mechanically activated 3Ti + Al composition with different periods of activation.

Analysis of dynamics of phase formation during high temperature synthesis for 1 min. activated 3Ti +Al powder composition proves that reflection on the first diffractogram (first frame of «diffraction movie») corresponds to initial titanium and aluminum components (figure 6).

After that comes the stage of initial structure formation, starting from 564 °C and on change of initial composition reflexes takes place as well as their gradual broadening with a shift to lower angles. That indicates average parameter increment of crystalline grids and phase changes. At that stage along with stable compounds Ti_3Al, $TiAl_3$, $TiAl_2$, some metastable virtual phases Ti_9Al_{23}, Ti_5Al_{11}, Ti_2Al_5 that had formed before the synthesis, were identified. After turning off heater compounds Ti_3Al, $TiAl_3$, Ti_5Al_{11}, Ti_9Al_{23} emerge as reaction products due to quick temperature decrease of furnace charge.

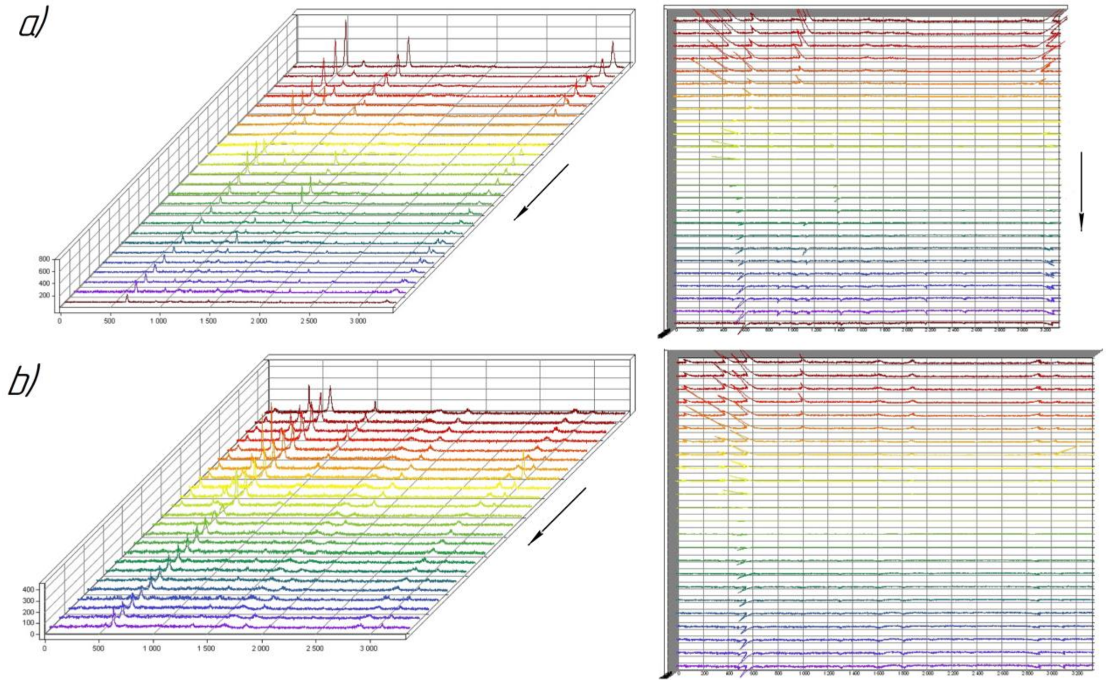


Figure 6. «Diffraction movie» synthesis of the mechanically activated 3Ti + Al composition. Horizontal axis – 2 Theta, degrees, axis of coordinates – intensity, a) MA – 1 min., b) MA – 3 min.

In order to figure out phase content of final product we made a diffractogram of the synthesis product upon cooling. As we can see dissipation of metastable phases Ti_9Al_{23}, Ti_5Al_{11} takes place in the process of furnace charge cooling. There are three phases present in final product – Ti_3Al, $TiAl_3$, $TiAl_2$, predominantly $TiAl_3$ phase of intermetallic compound.

4. Conclusion

The following conclusions were made:

1. Developed experimental system based on ultra-high frequency induction heater 6AV for research to study dynamics of phase formation processes in mechanically-activated systems, adapted to technique of dynamic diffraction of synchrotron radiation. Designed experimental complex is based on inductive heating and adjusted to self-propagating high-temperature synthesis by means of synchrotron radiation, it enabled us to carry out in situ synchrotron research of dynamics of phase formation during synthesis of mechanically activated 3Ti + Al composition.

2. The experimental complex developed and tested on the mechanically-activated Ti-Al system, adapted to the method of dynamic diffractometry in synchrotron radiation beams, which can be used to study dynamics of phase formation processes in induction heating mode, is of practical importance. Application of designed experimental complex is possible not only for registration of dynamics of phase formation of powder SHS Ti-Al systems in thermal explosion mode, but also for fuel air explosions in any SHS system due to fact that induction heated powder composition can be 1500 °C and more, what is enough for SHS formation in most systems.

3. As a result of pre-activation, reaction of high temperature synthesis of Ti-Al composition goes without liquid phase in mode of actual solid phase combustion what proves the emergence of new mechanisms of diffusion and mass-transfer in solid phase combustion of mechanically activated compositions [35, 36]. The studied dynamics of phase transformations of mechanically-activated system will allow to control high-temperature synthesis and to obtain ultrafine structures of synthesized materials-mechano-composites, providing increased properties of products, for example, in case of surfacing with similar composites [37].

5. Acknowledgments

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