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In situ synchrotron research of phase formation in mechanically activated 3Ti + Al powder composition during high-temperature synthesis under the condition of heating with high-frequency electromagnetic fields

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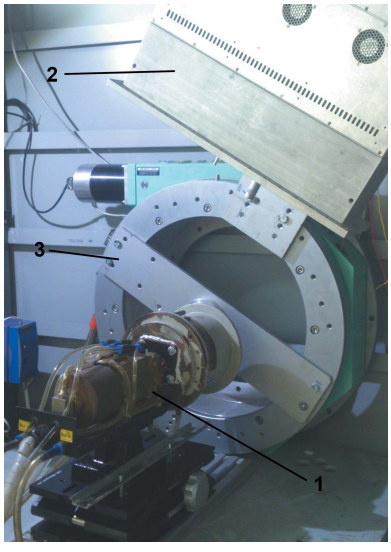
An *in situ* synchrotron study of the specific features of the phase formation dynamics in mechanically activated 16 wt% Al + Ti powder composition is described, the high-temperature synthesis being carried out under the condition of high volume inflammation by means of inductive heating. The kinetics of the phase formation were registered with an experimental complex, especially designed, constructed and adjusted for the method of dynamic diffraction analysis in synchrotron radiation beams. It has been experimentally *in situ* shown that increasing the time of mechanical activation of the initial powder mixture reduces the temperature at which components start to react and the time of realization of the high-temperature synthesis. With the latter set at 1 min of mechanical activation, the temperature of the reaction in the mixture is T = 603°C; at 3 min of mechanical activation, T = 442°C; and at 7 min, T = 359°C. The maximum burning temperatures are: for 1 min of mechanical activation, T_{max} = 1080°C; for 3 min, T_{max} = 1003°C; and for 7 min, T_{max} = 820°C. It was found that formation of both stable compounds Ti_3Al, $TiAl_3$, $TiAl_2$, TiAl and metastable phases Ti_9Al_{23}, Ti_5Al_{11}, Ti_2Al_5, Ti_3Al_5 occurs at the stage of primary structure formation, before the system goes to thermal explosion. High-temperature synthesis of a mixture of the studied composition takes place without formation of a liquid phase, in the solid-phase combustion mode. It was found that the increase in the time of mechanical activation of the initial powder mixture contributes to the formation of a product with a dominant content of intermetallic compound Ti_3Al. By synthesis of the powder mixture of composition 16 wt% Al + Ti, mechanically activated for 7 min, the content of Ti_3Al in the final product was found to be 68%.



1. Introduction

Nowadays, much attention is paid to methods of obtaining nanostructured composition materials with targeted properties. An effective method of obtaining such materials is self-propagating high-temperature synthesis (Yi *et al.*, 1992; Shon *et al.*, 2007; Merzhanov, 1998). An effective way to influence the structure of the initial reactionary furnace charge, thus regulating the kinetics of the structural phase transition during the synthesis, is the method of mechanical pre-activation (Bernard & Gaffet, 2001; Levashov *et al.*, 2007; Philpot *et al.*, 1987; Mukasyan *et al.*, 2010). Background mechanical activation helps to change the structure and phase composition of the final product, increasing the possibility of chemical reactions, namely extending concentration combustion limits,

changing the temperature and speed of combustion, inflammation temperature, *etc*. (White *et al.*, 2009; Loginova *et al.*, 2015, 2017; Park *et al.*, 2009; Zhu *et al.*, 2010; Mukasyan *et al.*, 2011).

To initiate the synthesis in a mechanically pre-activated system, a heat impulse is required. This will fluctuate considerably depending on the powder composition. Another important factor for gaining a homogeneous structure and phase composition is the speed of heating of the powdered composition. One way of creating an initiating temperature with high heating rate is applying extreme exposure to the powder system (*e.g.* blast wave, electric impulse), which can influence both macro kinetic parameters of the self-propagating high-temperature synthesis and phase structure transfiguration (Che & Fan, 2009; Loginova *et al.*, 2018*a*; Gedevanishvili *et al.*, 1999; Naplocha & Granat, 2009). However, the most universal extreme treatment is inductive heating with a heat input of the order of 100 K s^{-1}. The inductive heating method is noted for low energy consumption, high-rate heating reaction, simple and reliable equipment, and pure final product (Filimonov *et al.*, 2014; Shi *et al.*, 2015; Sobachkin *et al.*, 2018). However, the application of the inductive heating method, used for controlled synthesis of new materials with targeted properties, has not been thoroughly studied and requires further research.

For a better understanding of the phase transformation during the synthesis of mechanically activated systems by inductive heating, one needs to understand the characteristics of the 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* investigations of the structural changes and chemical dynamics in the combustion zone in real-time mode have become possible due to the application of synchrotron radiation. Today, there is really no alternative to synchrotron radiation methods in the research of high-rate processes, which are made possible by means of the high speed of diffraction registration, high intensity facilitating operations with reverse beam, and high spatial and time resolution of the detector, that together provide high accuracy (Gauthier *et al.*, 1999; Larson & Von Dreele, 2004). Many researchers use synchrotron radiation to form a 'diffraction movie', *i.e.* a consequential filming of a series of diffractograms enabling observation of the structural change of matter during its deformation, melting, crystallization, synthesis, *etc.* (Charlot *et al.*, 1999; Popova *et al.*, 2013; Liss *et al.*, 2009; Curfs *et al.*, 2007). High density of the synchrotron radiation monochromatic beam and progress in detectors have resulted in obtaining one X-ray pattern per microsecond. In a classical registration scheme this is close to the limit determined by the time of charge assembly and relaxation in the detector. Owing to its properties, it is possible to use synchrotron radiation for phase and structural material analysis. The short wavelength of synchrotron radiation is considered to be important. Its tolerance is commeasurable to the interatomic distance in the points of the lattice. In the experiment conducted here, the wavelength is 1.505 Å.

The compound Ti_3Al was chosen for study because its α_2-phase alloys are widely used in aerospace engineering, the automobile industry, shipbuilding and electric power engineering due to their high heat resistance, durability, resistance to aggressive environments, corrosion stability, low density, and high resistance to fatigue failure and creep (Bartolotta & Krause, 1999; Leyens & Peters, 2003; Yamaguchi *et al.*, 2000; Valiev & Langdon, 2006; Kim & Froes, 1990). Reasoning from this, it would be useful to extend research of the structure and phase formation in the Ti–Al system, and also to control its synthesis, in order to obtain intermetallic compounds with targeted properties. The paper describes *in situ* experimental tests of the dynamics of phase formation of a mechanically activated 3Ti + Al powder composition with different timing during high-temperature synthesis in dynamic thermal explosion mode by means of heating the composition in fast-changing electromagnetic fields.

2. Experimental technique

In this experimental study, titanium powder with an average grain size of 50 µm was used as the object of study, as well as aluminium powder ASD-1 with an average size of 12 µm. The powder used had the composition 16 wt% Al + Ti. Mechanically activated treatment of the initial powder compound was carried out with the help of an AGO-2 planetary ball mill, its cylinder capacity being 160 cm^3, with a steel ball diameter of 8 mm and cylinder centripetal acceleration of 40 g. The ratio of the initial powder mass to the grinding medium mass was 1:20. To prevent corrosion, air was extracted from the cylinders, which were then filled with argon under a pressure of 0.3 MPa. The period of mechanical activation used was varied, *i.e.* 1 min, 3 min and 7 min. Upon mechanical activation the composition was taken out of the cylinders in an argon-atmosphere-filled box.

The next stage represented *in situ* study of the dynamics of mechanically activated composition phase formation during high-temperature synthesis by dynamic diffraction analysis in synchrotron radiation beams. We used radiation from the electron storage ring VEPP-3 of the 'Diffraction movie' 5b station in the Budker Institute of Nuclear Physics of Siberian Department, Russian Academy of Science (Novosibirsk, Russia). A diagram of the electron storage ring is presented in Fig. 1. The method of diffraction analysis is based on the radiation of electromagnetic waves with charged particles; the particles move with relativistic velocities in a uniform magnetic field (Shull & Cline, 1990; Clemens *et al.*, 2008).

From the injector (linear accelerator), pre-accelerated electrons with relativistic velocity (energy limit $\simeq$ 300 MeV) reach the circular orbit of the accelerator and are held there by the magnetic field of the bending magnets. The radiation is concentrated in a cone with a $1/Y$ angle and is tangential to the trajectory of the radiation point,

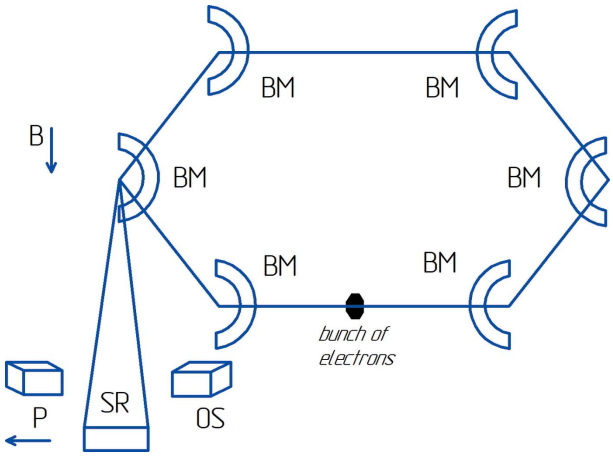


Figure 1
Electron storage ring structure: BM – bend magnets; B – magnetic field; P – photon polarization vector, radiated in electron orbit; OS – output slot, horizontally restricting synchrotron radiation beams; SR – synchrotron radiation.

$$Y \simeq mc^2/E, \tag{1}$$

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

In order to register the phase formation dynamics in the process of self-propagating high-temperature synthesis, an experimental complex was constructed based on high-frequency electromagnetic heating unit 6A that generates electromagnetic energy over a wide range of power (Fig. 2). The advantage of the induction method over classical heating methods (such as muffle furnace or electric filament) is that the induction method allows flash heating of the powder composition, which can be crucial for mechanically activated systems with a high content of non-equilibrium defects (Mukasyan *et al.*, 2011).

The mechanically activated composition was placed into a graphite melting pot, which is an open-top cylinder, and

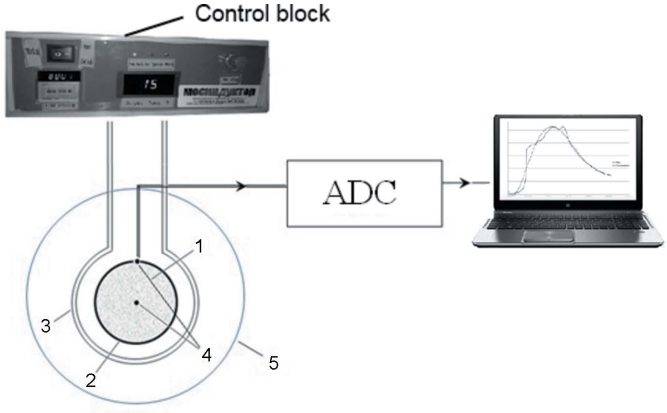


Figure 2
Experimental complex of high-speed induction heating: 1 – powder composition; 2 – graphite melting pot; 3 – induction filament; 4 – wolframite thermocouple units; 5 – vacuum lid.

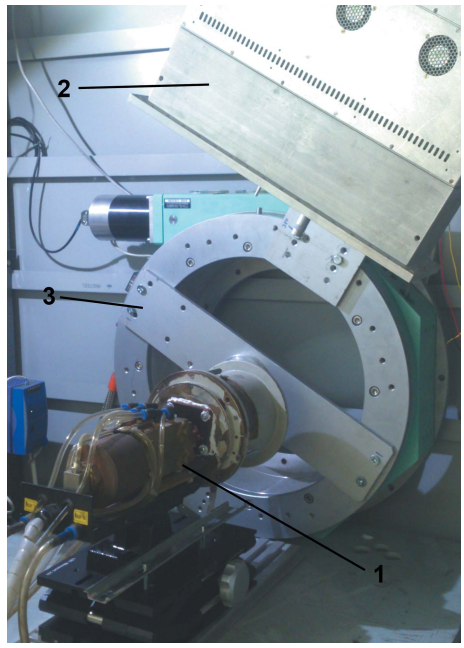


Figure 3
'Diffraction movie' experimental station 5b of the electron storage ring VEPP-3. 1 – experimental complex; 2 – detector; 3 – goniometer.

packed down. The melting pot was then isolated with an asbestos layer. A signal from the thermocouple unit was transmitted to an analogue–digital converter (ADC). The system was covered with a vacuum lid, with air being extracted and argon pumped in. The graphite melting pot was heated to high temperature by the high-frequency electromagnetic field, and the system heated the powder composition to >1500°C.

The experimental complex was adjusted for dynamic diffraction analysis using synchrotron radiation in order to study *in situ* high-temperature synthesis of mechanically activated 3Ti + Al composition in thermal explosion mode. Fig. 3 shows the 'Diffraction movie' experimental station 5b of the electron storage ring VEPP-3 with a mounted experimental device based on the inductive heating unit.

One peculiarity of the method of dynamic diffraction analysis is the fact that it needs composition materials with an open surface to which the synchrotron radiation beam can fall. The width of a synchrotron beam is ∼2 mm. To carry out the experiment a container was constructed, designed as a big hollow cylinder (Fig. 4). A specific feature of the reactor is an open surface of the composition to provide penetration and reflection of the synchrotron radiation beams, made possible due to the use of a thick metal plate with a 3 mm-wide and 30 mm-long slot. This is sufficient for a beam to fall on the furnace charge surface and to be reflected at an angle of 35–40°. The use of the metal plate provides additional fixation of the composition surface at one level during the heating and active chemical reactions.

At the first stage of the experiment, 3Ti + Al powder composition was placed into the graphite melting pot and pressed using the thermocouple unit under a laboratory press. The melting pot containing the pressed powder composition

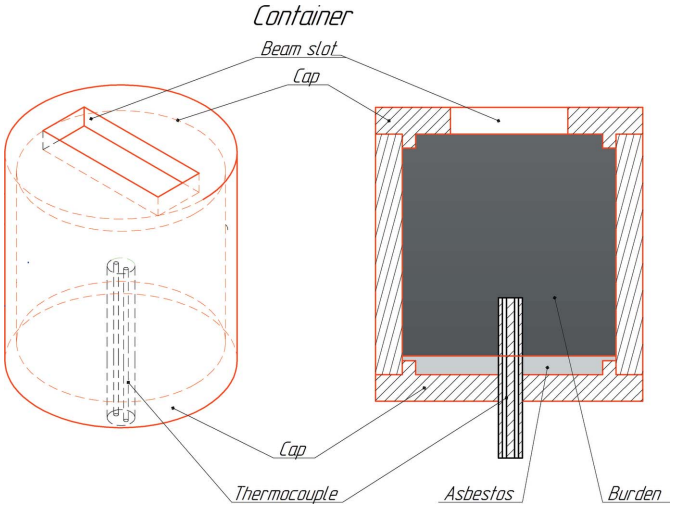


Figure 4
Container, adjusted for the study using synchrotron radiation.

and the thermocouple was put into the inductor, and then into the container set up in a vacuum chamber, from which air was extracted and argon delivered. Finally the experimental complex was mounted onto the 5b synchrotron radiation station of the VEPP-3 electron storage ring (Fig. 5).

Nonstop recording of the diffractograms was carried out with monochrome radiation, of wavelength $\lambda = 1.505$ Å, with a 30–70° range of scanning angle and 10 s frame interval. In the experiment we used an OD-3 single-coordinate detector with a focal length of 350 mm. It has 3328 channels, a registration angle of ∼30°, a minimum frame accumulation time of 1 µs, and a maximum number of frames of 64. The powder composition ratio corresponded to 3Ti + Al (16 wt% Al + Ti).

High-temperature synthesis was facilitated by means of increased inductor capacity obtained by heating the composition in an electromagnetic field. A PC was used to register the synthesis temperature, equipped with an LA 2.0 USB multichannel ADC card, to which a VR-5/20 type thermocouple unit was connected. Simultaneously with the synthesis initiation a 'diffraction movie' type automatic registration was started. The radiator generates a synchrotron radiation beam that passes through a beryllium window to the surface of the reacting furnace charge, located in the induction unit. The reflected beam reaches a detector. The construction is provided with a cooling system. The complex described above is used for synchrotron radiation beam formation and represents a unique device.

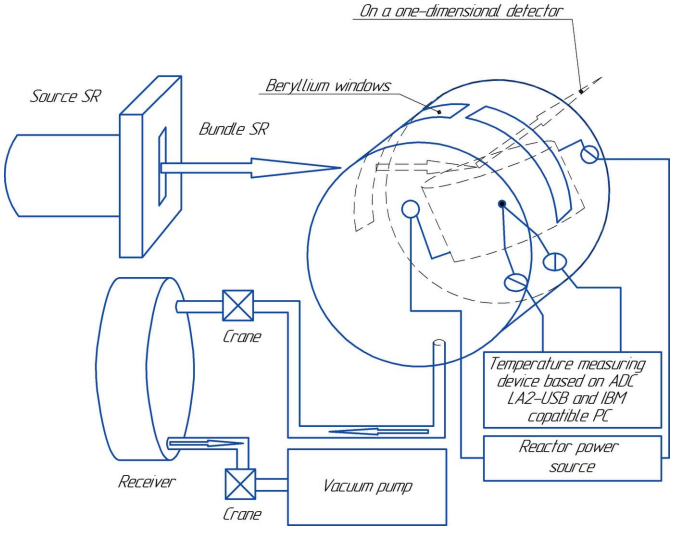


Figure 5
Experimental complex for the registration of phase formation of titanium aluminide in the process of inductive heating in the thermal explosion mode.

The application of the designed experimental complex is possible not only for the registration of the dynamics of phase formation of powder SHS Ti–Al systems in the thermal explosion mode but also for fuel–air explosions in any SHS system due to the fact that induction-heated powder composition can be 1500°C and more, which is enough for SHS formation in most systems.

Phase analysis of the obtained *in situ* diffractograms was carried out using the *dqv2* program developed in INP SB RAS (Novosibirsk, Russia). The percentage of the recorded phases was calculated by semi-quantitative methods according to the ratio of maximum peaks on diffractograms without taking into account mass absorption coefficients of phases (the accuracy of the calculations is 1–3%).

To conduct a phase analysis of the final synthesis product we used a DRON-6 X-ray diffractometer with Cu $K\alpha$ radiation (λ = 1.5418 Å). The diffractograms were registered under similar conditions with a scanning interval of 0.05° and time exposure of 3 s at each point. Processing and analysis of the data were performed by means of the *PDWin* program pack, designed for automatization of X-ray pattern processing (Loginova *et al.*, 2018*b*).

3. Experiment results and discussion

Fig. 6 shows diffractograms of the initial 16 wt% Al + Ti powder composition and identical compositions mechanically activated for 1, 3 and 7 min. The comparison study of the diffraction analysis showed the interdependence of the mechanical activation time and the intensity of the diffraction reflections of the powder components: the longer the time, the lower the intensity, and the wider the peaks become, proving the existence of non-equilibrium defects of the ground material and smaller microcrystals. Any other additional compositions are not formed upon mechanical activation.

The next stage is an *in situ* synchrotron study of the high-temperature synthesis of activated compositions in thermal explosion mode using the experimental complex adjusted for synchrotron radiation. Fig. 7 presents synthesis thermograms obtained by inductive heating of the 3Ti + Al composition with different mechanical activation periods.

Filming of the *in situ* dynamics of the phase formation ('diffraction movie') was started from the beginning of the system heating. The heating was switched off when the reacting furnace charge reached its maximum temperature,

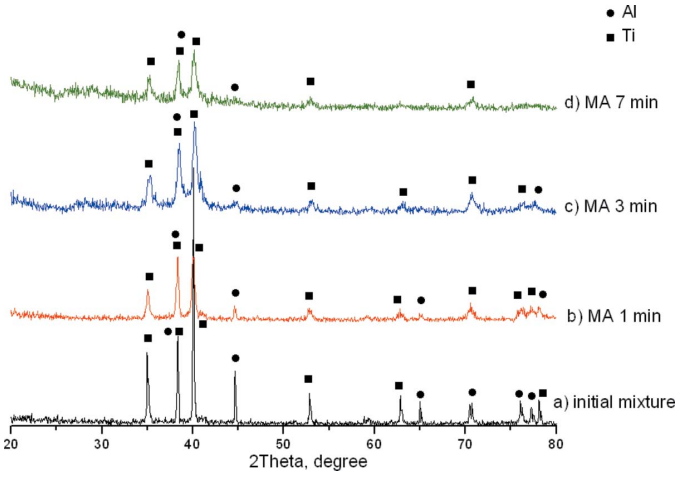


Figure 6
Diffractograms of the initial (*a*) and mechanically activated (*b–d*) 3Ti + Al composition: (*b*) 1 min, (*c*) 3 min, (*d*) 7 min.

with the consequent cooling to room temperature. As follows from the thermograms, for 1 min of mechanical activation of the mixture the reaction start temperature is T = 603°C; for 3 min, it is T = 442°C; and for 7 min, it T = 359°C. Maximum combustion temperatures are: for 1 min of mechanical activation, T_{max} = 1080°C; for 3 min, T_{max} = 1003°C; for 7 min, T_{max} = 820°C. Thus, we can see a relation between the temperature decreasing at the beginning of the composition reaction and the time of the mechanical activation. A longer mechanical activation time influences the thermal parameters of the composition combustion: the temperature decreases and the rate of component interaction grows. The mechanical activation increases the reactivity of the powder components, thus facilitating longer chemical reactions, with the synthesis in the solid-phase combustion mode.

Fig. 8 represents the *in situ* dynamics of the phase formation under inductive heating conditions of the mechanically activated 3Ti + Al composition with different periods of activation. The first frame of the 'diffraction movie' for the powder mixture activated for 1 min was registered at T = 40°C.

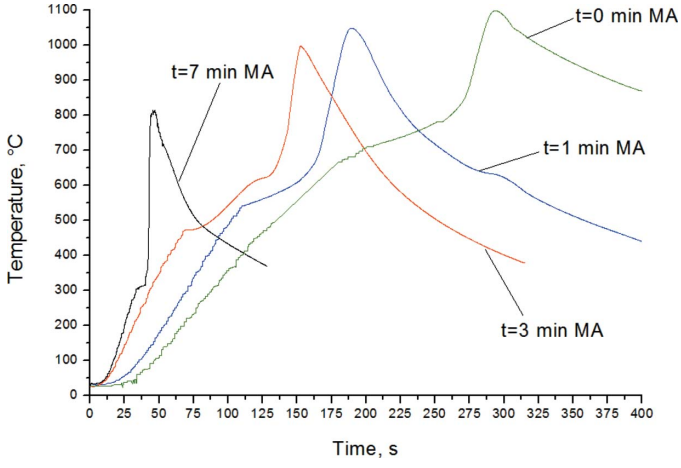


Figure 7
Thermograms of the synthesis of 3Ti + Al composition.

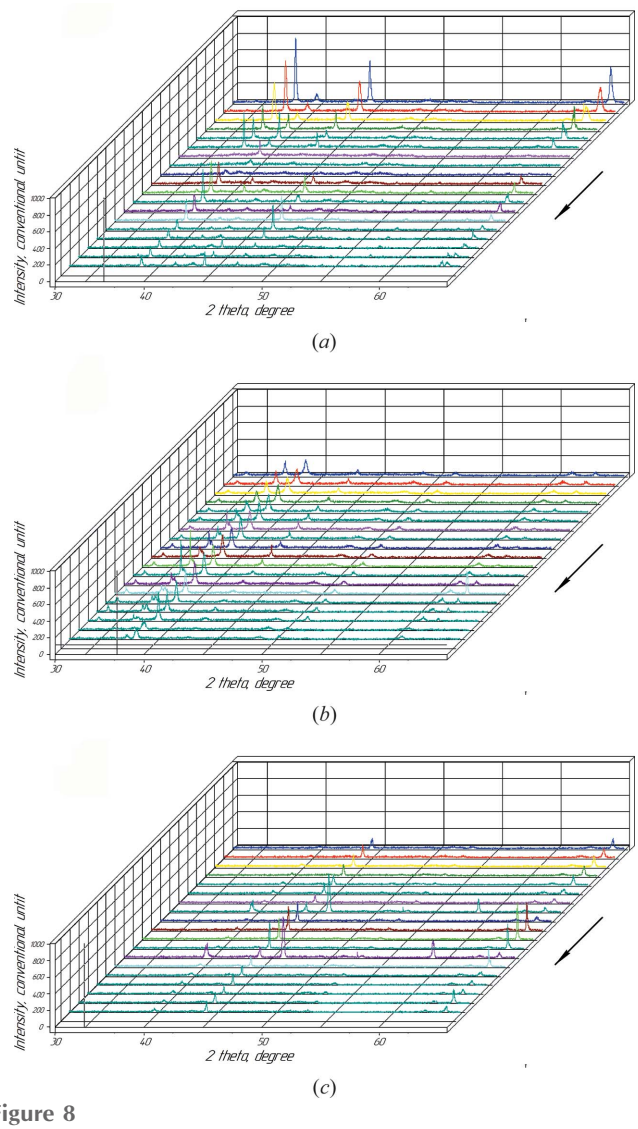


Figure 8
'Diffraction movie' synthesis of the mechanically activated 3Ti + Al composition (the arrow indicates the sequence of obtaining diffractograms during synthesis reaction): (*a*) 1 min, (*b*) 3 min, (*c*) 7 min.

Analysis of the dynamics of the phase formation during high-temperature synthesis for the 1 min-activated 3Ti + Al powder composition proves that the reflection on the first diffractogram (the first frame of the 'diffraction movie') corresponds to the initial titanium and aluminium components (Fig. 9). Up to T = 320°C the pattern of the diffraction field practically does not change.

Next comes the initial structure formation stage. Changes of the initial composition diffraction reactions take place as well as their gradual broadening with a shift to lower angles. This indicates the average parameter increment of crystalline grids. Then, starting from a temperature of 564°C, processes of phase formation with their subsequent transformations begin. Thus, at the stage of initial structure formation, 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 system exits to thermal explosion were identified. Dominating this stage is the compound $TiAl_3$. At T = 603°C the system

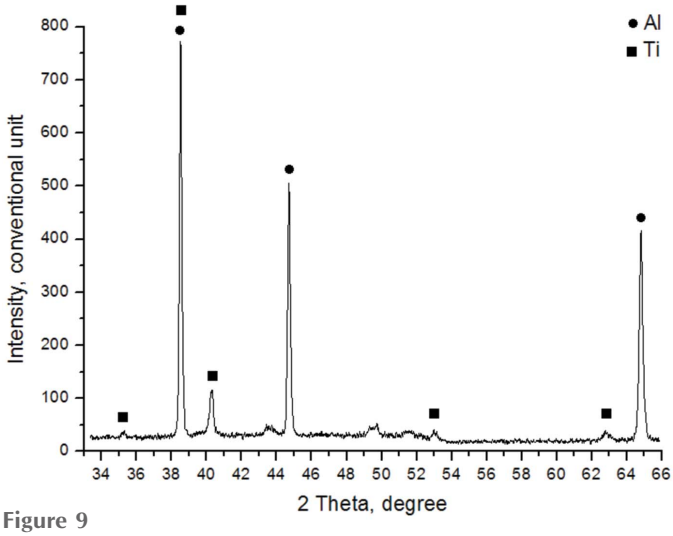


Figure 9
Diffractogram, made *in situ* during the synthesis of 1 min activated 3Ti + Al composition.

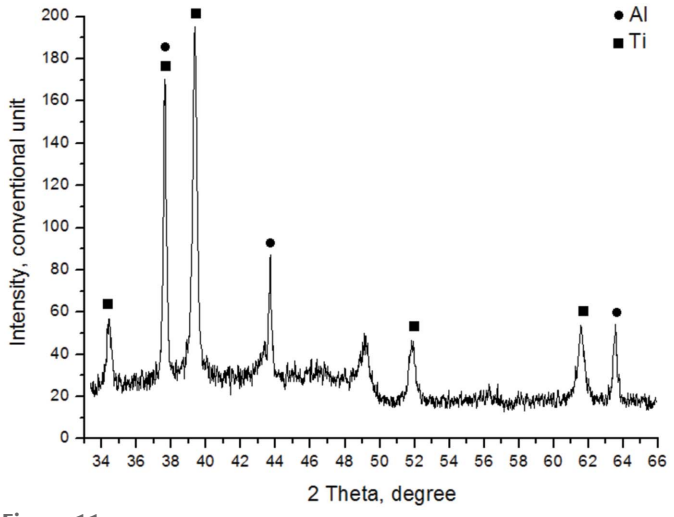


Figure 11
Diffractogram, made *in situ* during the synthesis of 3 min activated 3Ti + Al composition.

goes to thermal explosion. Phase transformations continue. At the moment of reaching maximum charge temperature of the synthesized product, $TiAl_3$, Ti_3Al and $TiAl_2$ are registered, as well as a small amount of metastable phases Ti_5Al_{11} and Ti_9Al_{23}. After turning off the heater ($T = 1080$°C), compounds Ti_3Al, $TiAl_3$, $TiAl_2$ and intermediate phases Ti_5Al_{11}, Ti_9Al_{23} emerge as reaction products due to the quick temperature decrease of the furnace charge. In the synthesized product the main phase is $TiAl_3$ (~78%); other phases are Ti_3Al (12%), $TiAl_2$ (7%) and intermediate phases Ti_5Al_{11}, Ti_9Al_{23} (3%).

In order to work out the phase content of the final product we recorded a diffractogram of the synthesis product upon cooling (Fig. 10). As we can see, dissipation of the metastable phases Ti_9Al_{23}, Ti_5Al_{11} takes place in the process of furnace charge cooling. There are three phases present in the final product: Ti_3Al, $TiAl_3$ and $TiAl_2$, but predominantly $TiAl_3$ phase of the intermetallic compound.

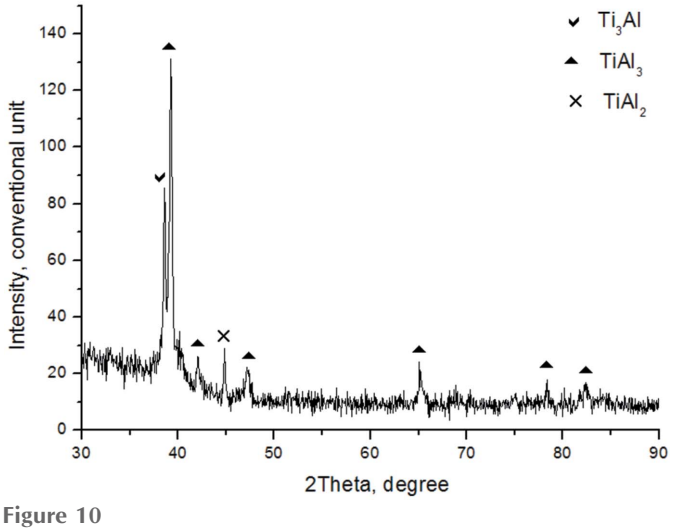


Figure 10
Diffractogram of the synthesis final product of 1 min mechanically pre-activated 3Ti + Al composition.

In the 3 min pre-activated composition (by contrast with the 1 min pre-activated composition) we could observe decreased intensity and broadening of the diffraction maximum of titanium and aluminium and a shift to lower angles from the very beginning of the filming process with temperature of 40°C, acknowledged by the first diffractogram of the 'diffraction movie' (Fig. 11).

In 10 s from the start of filming, along with shifted peaks Al and Ti, reflections corresponding to $TiAl_3$ and Ti_2Al_5 compositions were identified. Peak broadening is due to decreased fields of coherent scattering (crystallite sizes) and increased micro-stress in the crystalline grids. From $T = 200$°C, additional reflections corresponding to Ti_3Al, $TiAl_2$, TiAl and metastable phases Ti_9Al_{23}, Ti_5Al_{11} are registered on the diffractograms. Further, the phase formation mechanism will be much different from that of the 1 min activated composition. In the synthesis process at the stage of initial structure formation, the main phase is $TiAl_3$, as well as compounds Ti_3Al, $TiAl_2$, TiAl and metastable phases Ti_9Al_{23}, Ti_5Al_{11}, Ti_2Al_5, Ti_3Al_5. By the end of the reaction, a multiphase product consisting of $TiAl_3$, Ti_3Al and $TiAl_2$ is synthesized. The main compound is $TiAl_3$. According to the quantitative calculation its content is 60%, with the content of Ti_3Al being 24% and that of $TiAl_2$ being 16%.

Structure maxima corresponding to the Ti_3Al, $TiAl_3$ and $TiAl_2$ phases are identified in the diffractogram of the product obtained as the result of cooling (Fig. 12).

As for the 7 min activated composition, the very first diffractogram (Fig. 13) with temperature T = 40°C showed peak broadening corresponding to the Ti_2Al_5 and Ti_5Al_{11} metastable phases along with maxima corresponding to initial Ti and Al components. That is, in this case, similar to the 3 min mechanically activated composition, the transition from initial components to the final product is so quick that it cannot be registered by the 'diffraction movie' with 10 s framing.

The further process of phase formation does not bear any significant difference from the cases described above.

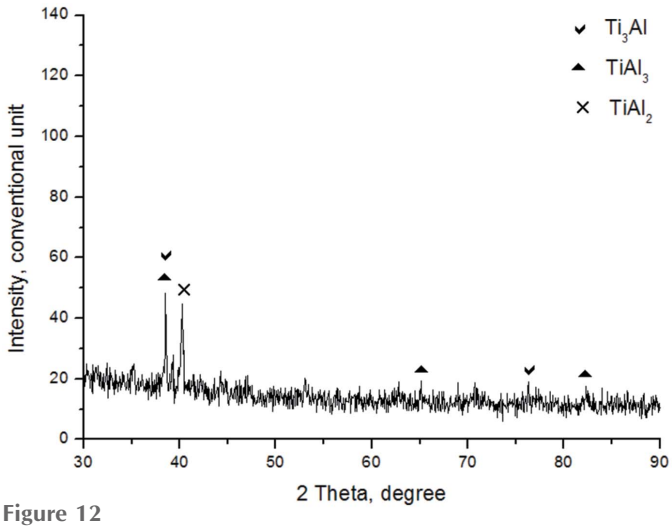


Figure 12
Diffractogram of the synthesis final product of 3 min activated 3Ti + Al composition.

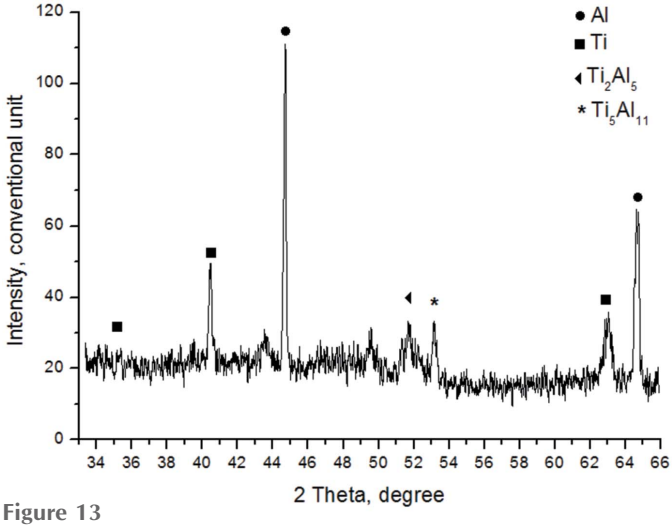


Figure 13
Diffractogram made *in situ* during the synthesis of 7 min activated 3Ti + Al composition.

However, increasing the time of mechanical activation accelerates the phase transformations processes. The formation and disappearance of metastable phases takes place until the system reaches thermal explosion (T = 359°C). Metastable phases Ti_9Al_{23}, Ti_5Al_{11}, Ti_2Al_5 and Ti_3Al_5 do not influence the synthesis of Ti_3Al, $TiAl_3$, $TiAl_2$ and TiAl compositions. The stable compositions and metastable virtual phases form before the synthesis at the stage of initial structure formation and are preserved after the heater is turned off. At the moment of reaching maximum temperature of charge T = 820°C the product is synthesized, showing a dominant content of Ti_3Al, in contrast to the previous two cases. The content of Ti_3Al in the synthesized product is about 68%. The presence of 26% $TiAl_2$ and 6% $TiAl_3$ was also recorded.

The diffractogram of the final product formed after cooling of the system to room temperature is presented in Fig. 14. We can identify reflections corresponding to the Ti_3Al, $TiAl_3$ and $TiAl_2$ phases of the intermetallic compounds, with Ti_3Al being the predominant phase.

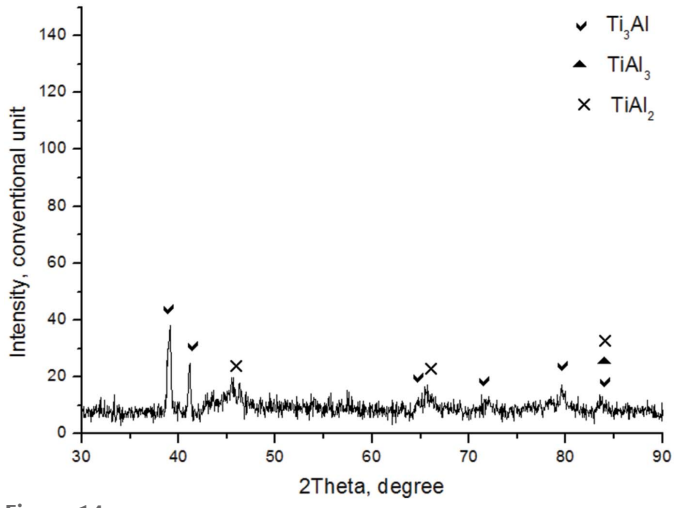


Figure 14
Diffractogram of the synthesis final product of 7 min activated 3Ti + Al composition.

4. Conclusion

The designed experimental complex is based on inductive heating and adjusted to self-propagating high-temperature synthesis by means of synchrotron radiation. It has enabled us to carry out *in situ* synchrotron research of the dynamics of phase formation during the synthesis of mechanically activated 3Ti + Al composition. The following conclusions are made:

(i) The increased time of mechanical pre-activation of powder composition 16 wt% Al + Ti results in the transformation of the thermal parameters of combustion: an increase of the speed of burning and a decrease in the time and temperature of the synthesis initiation. For 1 min, the mechanical pre-activation (MA) reaction starting temperature in the mixture is T = 603°C; for 3 min, MA T = 442°C; for 7 min, MA T = 359°C. The maximum burning temperatures are: for 1 min, MA T_{max} = 1080°C; for 3 min, MA T_{max} = 1003°C; for 7 min, MA T_{max} = 820°C.

(ii) The preliminary mechanical alloying processing of powder mixture composition 16 wt% Al + Ti makes it possible to accelerate the time of chemical reactions in high-temperature synthesis. The formation of stable compounds Ti_3Al, $TiAl_3$, $TiAl_2$ and TiAl and metastable phases Ti_9Al_{23}, Ti_5Al_{11}, Ti_2Al_5 and Ti_3Al_5 occurs before the system exits to thermal explosion mode, at the stage of primary structure formation. In the synthesis of 7 min MA powder mixture, already at T = 40°C the formation of compounds (Ti_2Al_5 and Ti_5Al_{11}) was observed.

(iii) Preliminary mechanical activation for 7 min of powder mixture 16 wt% Al + Ti contributes to the formation of a product with a dominant content of intermetallic compound Ti_3Al, constituting 68%.

(iv) As a result of pre-activation, the high-temperature synthesis reaction of the composition under study proceeds without liquid phase in the actual solid-phase combustion mode, proving the emergence of new diffusion and mass

transfer mechanisms in the solid-phase combustion of mechanically activated compositions.

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