SHS for Space Exploration

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Abstract

For over past years, interest of leading space agencies (NASA, JAXA, ESA, RSA, etc.) in SHS experiments under microgravity conditions has been increasingly growing. The first SHS experiments during a parabolic flight in Russia and aboard the MIR Space station gave promising results. Similar studies are now being carried out in various countries. The obtained data and assimilated experience have shown that SHS reactions can be used for (a) synthesis of high-porosity materials and regulation of structure formation in combustion products, (b) preparation of skeleton structures by combustion of particles suspended in vacuum, (c) generation of thermal energy, (d) generation of incandescent radiation, and (e) for in-space fabrication and in-situ repair works (welding, joining, cutting, coating, near-net-shape production, etc.). However, the results of the above studies (strongly scattered in the literature) still seem insufficient for elucidating the mechanism of combustion in. Indeed, the experiments were carried out by different researchers for a dozen of systems and for strongly different duration of microgravity (drop towers, parabolic flight of a plane, parabolic flight of a spacecraft, in space stations). No correlation has been made with the available data of SHS studies (oriented largely on practical implementation) in conditions of artificial gravity.

In experiments, the combustion wave has enough time to spread over the sample while the structure formation, may not have. This implies that the process of wave propagation should always be identical, irrespective of the type of experimental technique and place of experiment.

SHS experiments in space are attractive because (a) of low energy requirements, (b) processing cycle is short, (c) of process simplicity, (d) of versatility (wide range of suitable materials, and (e) the use of in-situ resources possible. To date, SHS experiments has already been performed aboard the International Space Station (ISS).

Space technology has been developed for frontier exploration not only around the Earth orbit environment but also to the Moon, Mars, etc.

Introduction

Space experiments on Self-propagating High-temperature Synthesis (SHS) in the Russian segment aboard the International Space Station (ISS) in 2005-2010 were performed within the state Program of Applied Studies and Experiments. The experiments were undertaken in continuation of the investigations performed in 1997-1998 aboard the Space Station MIR. In 2005, the SHS experiments in space were carried out by S.K. Krikalev (mission ISS-11). Further experiments were performed during the ISS-13, ISS-16, ISS-18, ISS-19/20, and ISS-21/22 missions.

Self-propagating high-temperature synthesis (SHS) is some special kind of combustion in solid (powdered) mixtures that occurs in an autowave mode and yields valuable refractory compounds or materials as products [1, 2]. SHS is known to involve numerous physical processes, such as melting of reagents and products, spreading of melt, droplet coalescence, diffusion and convection in liquid metals and nonmetals, buoyancy of solid particles and bubbles in the melt, nucleation of solid prod-

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ucts, crystal growth, and sample deformation. Some of these processes are affected by gravity. There are two different SHS modes: one is a layer-by-layer (frontal) combustion and the other is a thermal explosion (volume reaction). In the former case, an exothermic reaction sets on upon initiation of combustion wave propagation along the sample. In the latter, a pre-pressed green sample is heated in a furnace up to self-ignition, and the reaction taking place over the entire sample volume. Therefore, the process can run at the minimum energy of external heaters, under any atmosphere or in vacuum and with simple technological equipment. The conditions of technological experiments aboard a spacecraft are restrained by the unavailability of powerful electrical sources and operational room. Thus, employment of SHS technologies is a perspective solution to technological problems of production of required materials and reductive in-situ repair. The main advantages of the proposed process are energy self-sustenance, i.e. generation of high temperatures without extra energy sources, indifference to the environmental conditions (oxidative or inert media, vacuum), and a wide range of synthesized materials (intermetallides, carbides, silicides, borides, and respective composites) [1, 2]. In a number of SHS systems, a high-temperature chemical interaction can be considered as a fast-running process. Therefore the first investigations of the microgravity influence on SHS reactions used the methods of 'parabolic flight' [3] and later 'drop tower' [4-6].

These methods allow creation of short-term microgravity conditions (from a few seconds to some tens of seconds) and can be used in investigations of fast processes that run for less time within which microgravity exists. However, duration of the SHS product structure formation can be much higher than the synthesis time and achieve some minutes.

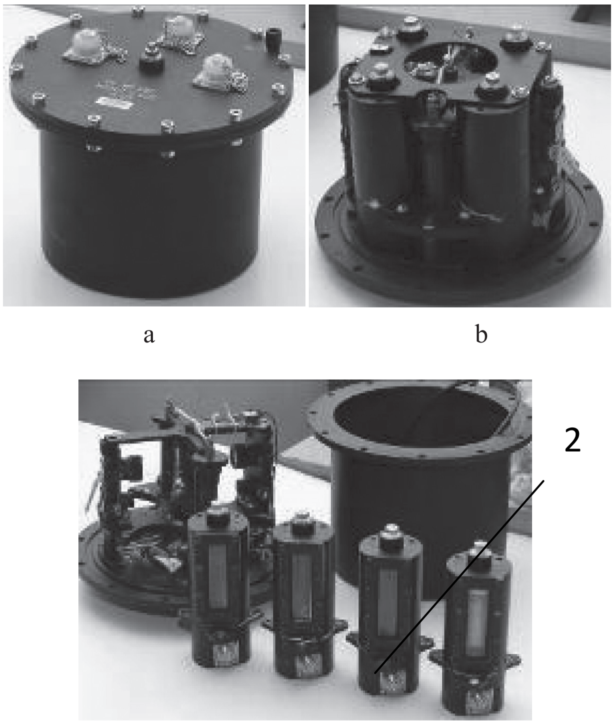
Long duration of the process is typical of highly caloric SHS systems with complete or partial melting of the components involved [7, 8]. Liquid products of such systems determine their high sensitivity to the effect of gravity. An interpolation-based diagnostics of SHS system behavior under microgravity conditions [9] showed that the sensitivity to gravity level increased with the liquid phase concentration in the reaction products. The first experiments on combustion of highly exothermic SHS systems under the conditions of flying space station were performed on an the Optizon-1 setup [10, 11, 14]. The setup allowed the sample ignition with the use of a powerful heat impulse generated by means of a light flow concentration on the sample surface. However, the setup had such drawbacks as large dimensions and weight, high energy consumption, long preparation time determined by the necessity of housing of an individual sample, sophisticated program of heating regimes etc., therefore a cosmonaut had to spend much time for the experiment to be carried out.

Some odd structural phenomena were found during investigation of the thermite-type SHS systems with completely molten combustion products (NiO + Ni + Al).

The task objective of SHS experiments aboard ISS was investigation of the influence of long-term microgravity on the processes of high-temperature synthesis and formation of product structure. The problems studied during the microgravity experiments included determination of the combustion regularities and formation of macro- and microstructure of condensed reaction products, the structure of the porous space and transition areas at the place of joining (welding) of the samples with various porosity and chemical composition. Besides, the mechanism and kinetics of formation of intermetallics and refractory inorganic compounds at the metal interface with the components of SHS systems were among the research goals. Solution of these problems will make it possible to synthesize refractory materials with a unique structure of foams of granular skeletons, which are efficient heat-insulating materials used in the space techniques involving technological tasks of the equipment mounting, demounting, and repair.

Experimental and Discussion

For SHS experiments in microgravity, a special research chamber was designed and manufactured in cooperation with the Central Scientific Research Engineering Institute (Korolev) [12]. The proposed reaction chamber allowed minimization of a cosmonaut's intrusion into the experiment preparation and essential simplification of the research process in ISS. The desired installation consisted of three key units: (1) an experimental chamber (1), a unit of communication and control (2), and a space complex *Telescience* for registration of the video signal (3). The reaction chamber was composed of an external protecting shell (Fig. 1a), with four independent experimental capsules (Fig. 1b, c). Two micro video cameras (3) and reflecting boards (4) gave the possibility of simultaneous video recording of two experimental capsules having transparent quartz windows (5) with one video camera (Fig. 1c). Such a construction provided maximum reduction of the area occupied by the unit and simplified the equipment transport into the orbital station and back upon returning to the Earth.



a b

c

Fig. 1. The experimental unit: an external view (a) that same with demounted protecting shell (b) and experimental capsules (c).

During microgravity experiments, the experimental process was registered with a video tape recorder. No special measuring means, pilot or any other equipment commonly required for the control, adjusting, and current repair was needed for the SE SHS performance on RS ISS.

The control over temperature and pressure inside the removable container, the green sample state, application of an initiating impulse, and the signal transfer from the video tape recorder onto the registering device was performed with the use of a supply and regulation unit. The experiments were in turn carried out within short time intervals (5-10 min) to let the containers be partially cooled.

The removable container that returned onto the Earth contained the experimental capsules with the material synthesized in the course of SE SHS and video cassettes depicting the data on the SHS process under the condition of the space experiment. When in the terrestrial laboratory, the SHS-produced samples underwent X-ray phase, microstructural, and chemical analyses. The material physicochemical properties (strength, hardness etc.) were also studied and the results obtained for the samples synthesized under terrestrial and space conditions were comparatively analyzed.

According to the program of the flight preparation, training studies of the future cosmonauts and their back-ups had training curses in the Gagarin Space Training Center.

The experimental results obtained within ISS-11 and ISS-18 were published in the home and foreign magazines and presented at conferences and symposia [8-26].

Four samples were under study during the ISS-19/20 flight: (a) investigation of the influence of long-term microgravity on the processes of voluminous SHS reactions, formation of SHS coatings and porous functional materials (samples #1 and #2) and (b) investigation of the prolong influence of weightlessness on the interaction of molten SHS products and metallic supports (samples #3 and #4).

To gain the purpose the fastening eclipses were improved (Fig. 2), the green compositions were optimized, and the experiments were pilot-tested under terrestrial conditions.

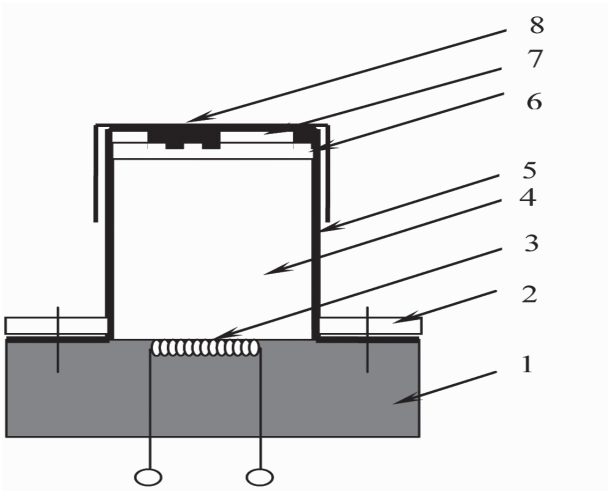


Fig. 2. The scheme of the synthesis of porous functionally graded materials in space: 1 – platform; 2 – sample-fastening ring; 3 – igniting wire; 4 – sample (lower pellet); 5 – Al foil (protecting the sample from damage); 6 – sample (intermediate pellet); 7 – sample (upper pellet); 8 – fastener of the upper pellets.

The combustion reaction was initiated for 5-7 s with an electric impulse fed onto the igniting tungsten wire. A self-propagating exothermic reaction induced by the red-hot wire in the lower pellet was accompanied with bright glowing and resulted in a voluminous reaction (thermal explosion) in the intermediate and upper pellets.

The exothermic reaction yielded molten products which under the gravity effect could form unique porous structure and phase composition. Sample # 1 was composed of three pellets wrapped into an aluminum foil: lower (basic) 0.35NiO + 0.66Ni + 0.29Al + Ti, intermediate Ti – Al, and upper Ti – Ni ones.

It was expected that the experiment with the given sample would bring about a porous functional material based on the TiNi alloy and the influence of prolonged microgravity on the formation of a protecting coating from titanium alloys would be studied under the conditions of thermal explosion.

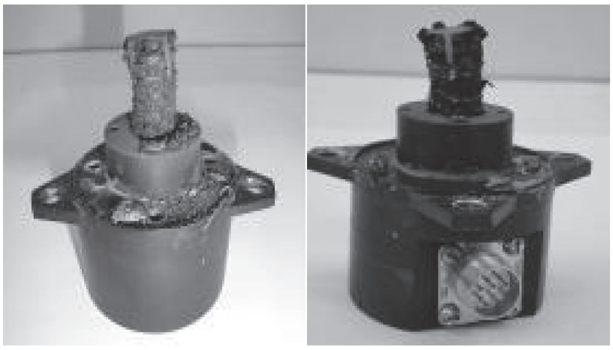
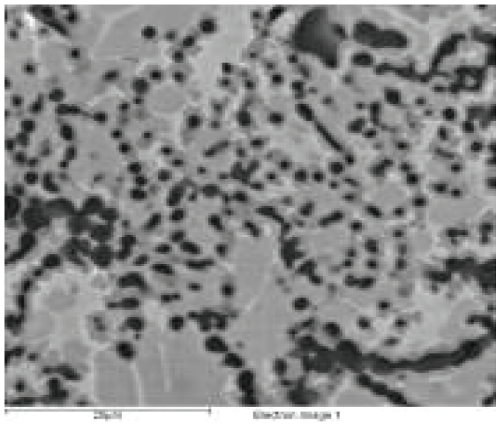


Fig. 3. An exterior of samples #1 and #2 after capsule disclosure.

Sample #2 was also composed of three pellets, each being wrapped into an aluminum foil. However, in this case, investigations were aimed at the in fluence of long-term microgravity on the formation of a coating from aluminum-based alloys under the condition of thermal explosion. A particular attention should be made to fundamental study of the influence of long-term microgravity on the main reaction processes (liquid phase formation, impurity gas release, crystallization) under thermal explosion.

A porous composite material characterized with a globular structure was synthesized in the experiments with the use of a basic pellet.

A globular structure (Fig. 4) is formed under microgravity: rounded grains with a predominant phase of titanium monoxide (dark phase) is uniformly distributed in the sample. Along the grain boundaries there is titanium nickelide (gray circular structure) and a Ti-Ni-Al solid solution (light phase). Such material microstructure allows suggestion of good strength characteristics of the synthesized material. However, the main experimental objectives were the processes of thermal explosion and formation of coatings and compounds under the long-term microgravity. As mentioned above, formation of coatings on the base of Ti alloys was studied in the case of the first sample. After a voluminous reaction (thermal explosion), the Ti-Al pellet with pre-pressed metallic wires, foils, and plates of various metals (Ta, Mo, Ti, Nb) was a porous material based on the TiAl and Ti_3Al alloys.



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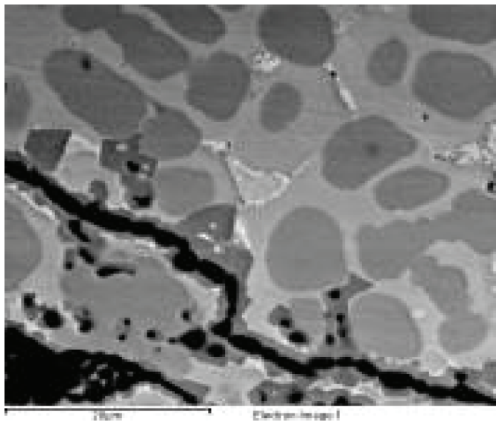


b

Fig. 4. Microstructure of samples #1 (a) and #2 (b) synthesized under microgravity.

The formation of a porous structure was an obstacle to the formation of a coating with good adhesion properties (Fig. 5). The coating formation on the Nb, Mo, and Ta foils was not detected and the reaction resulted in the formation of porous TiAl thus being evidence to the reaction completion (Fig. 6).

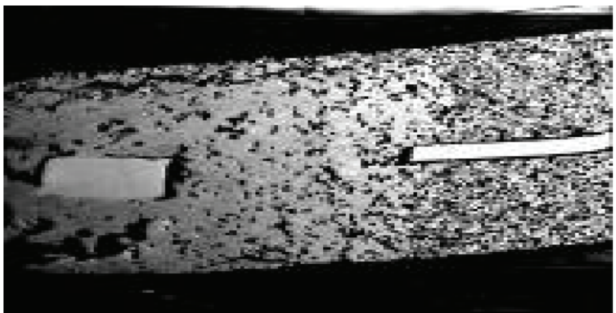
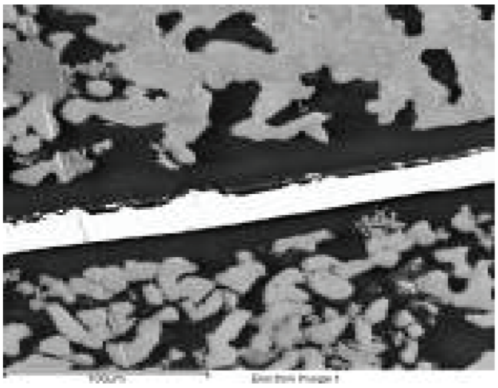


Fig. 5. Microstructure of the Ti-Al sample with pre-pressed Ti and Mo foils.

Coatings are seen on some small sites of titanium (Fig. 6b) formed of Ti_3Al due to partial reaction of titanium. When the reaction of thermal explosion takes place in the Ti-Ni sample, the coatings formed on the Mo and Ta supports exhibit good adhesion properties (Fig. 7). This proposition is well evidenced by the presence of the $TiNi_2$ grains respectively enriched with the Mo and Ta compounds.



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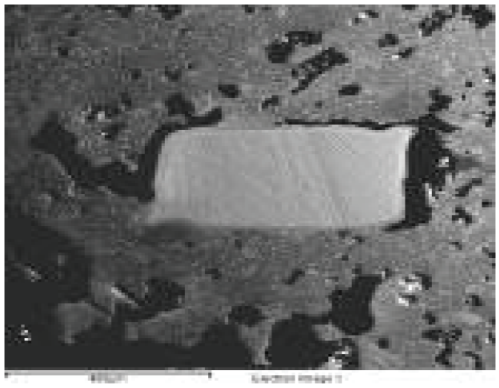

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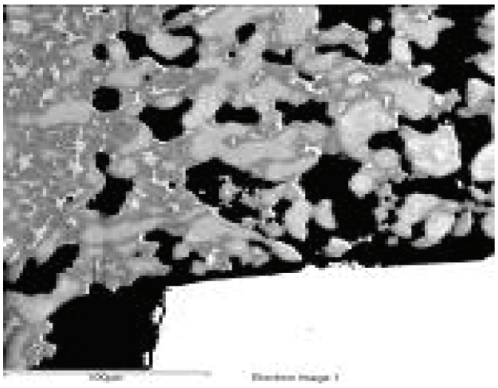
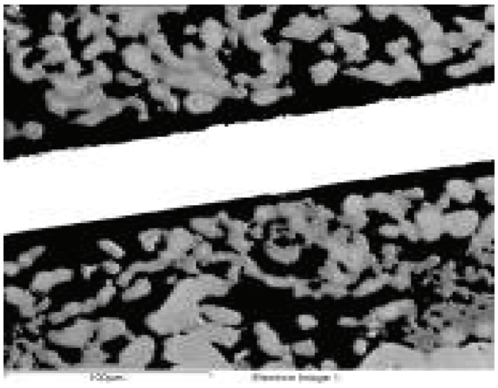
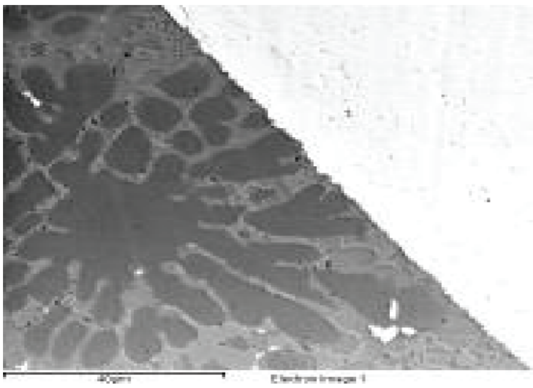


c



d

Fig. 6. The Ti-Al sample microstructure with foils of Ni (a), Ti (b), Mo (c), Ta (d).



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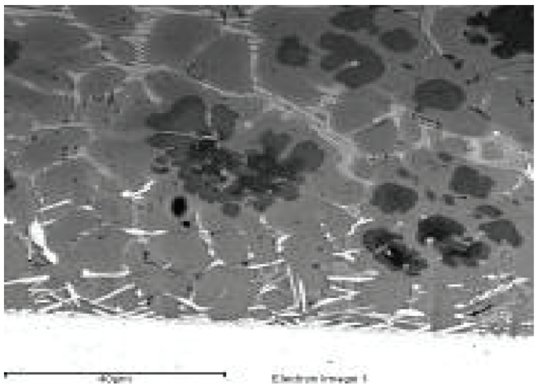

b

Fig. 7. Microstructure of the Ti–Ni sample with Mo (a) and Ta (b) foils.

Besides, near the Mo support dendrite structures based on Ti-Ni-Al are formed. This can happen only under the condition of microgravity, since in the given reaction aluminum could diffuse into the upper pellet from the lower one. Indeed the pellet was initially pressed from the mixture of only titanium and nickel with added metal foils.

Sample #2 was used in the study of formation of coatings based on Al alloys under thermal explosion. Opposite to sample #1, a reaction-induced joining of NiAl and TiAl pellets was observed in this case. This can be explained by a high content of the liquid phase and its rather durable lifetime (Fig. 8).

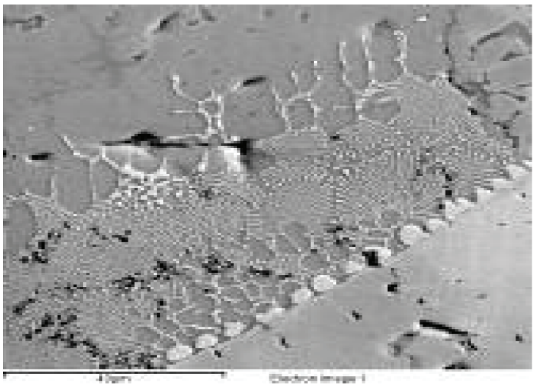

Fig. 8. Microstructure of the reaction-induced joining of two samples.

The microstructure of the gradient material produced with the method of thermal explosion owing to the in-reaction joining of two samples of aluminum nickelide (the lower part of the photo and titanium nickelide ($TiNi_2$). In the center of the photo, one can see an intermediate layer formed due to aluminum diffusion from the lower pellet into the upper one, which could happen only under the condition of microgravity.

Under thermal explosion coatings with good adhesion are formed on molybdenum and tantalum supports in the Ni–Al system (Fig. 9a, c). In contrast to the samples synthesized under the terrestrial conditions, the formation of a Ni_3Al-based coating on the molybdenum foil under the microgravity is followed by the formation of a NiAl-based coating, which is characterized with a higher coefficient of refractoriness.

The coating formed on the niobium foil is not firm; however, an intermediate layer is seen to be formed in some sites (Fig. 9c). Thus, the experiments performed showed the possibility of synthesizing gradient intermediate-based materials, compounds, and coatings by the method of thermal explosion. Optimum compositions (Ni–Al, Ti–Ni) usable for the formation of compounds and coatings on the molybdenum, tantalum, and titanium supports were experimentally found. The experimental results obtained suggest the mechanism of the coating formation. It is known that nickel does not practically melt in molybdenum but readily diffuses along the grain boundaries. It can be supposed that under the conditions of the thermal explosion reaction the metal grains are torn off from the support to be further dissolved in the melt. This is evidenced by uneven foil-coating interfaces and large formations enriched with the foil grains. Besides, the concentration of the support-forming metals in the coating is seen to monotonically decreasing from the interface to its surface.

a b c

Fig. 9. Microstructure of the Ni–Al sample with foils of tantalum (a), niobium (b), and molybdenum (c).

The experiments with samples #3 and #4 included determination of the conditions of synthesis of molten inorganic materials formed during SHS and their interaction with the metallic supports resulting in the formation of coatings (Fig. 10). One of the experimental goals is investigation of the influence of weightlessness and phase composition of the SHS products on the characteristics of the coating formed on the surface of the metallic supports.

Samples #3 and #4 produced from the Ni–Al–NiO system were intended for the formation of molten SHS products of the following compositions:

$$Al_2O_3\text{–}NiAl \text{ and } Al_2O_3\text{–}NiAl_3.$$

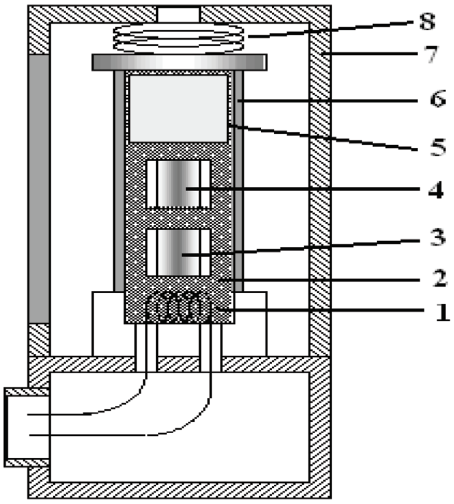

Fig. 10. Experimental setup for studying the interaction of molten SHS product with metallic substrates in microgravity: 1 – igniting wire; 2 – reaction mixture; 3, 4 – metallic substrates (titanium, dural); 5 – heat insulator (SiO_2); 6 – quartz tube; 7 – chamber shell; 8 – steel wire fastening the quartz tube in the chamber.

In the case of sample #3, the grater attention was paid to the influence of microgravity on the formation of oxide phases, while sample #4 was an illustration to the formation of intermetallide phases (Fig. 11).

Fig. 11. An external view of samples #3 and #4 after the capsule disclosure.

Investigation of sample #3 showed that exothermic SHS reactions yielded fused intermetallic and oxide compounds which when interacting with the metallic supports are spreading along their surface form a non-joinable compound. Under the space conditions the surface of the titanium support becomes the place of participation of a foam-like SHS product consisting of molten drops of an intermetallide and aluminum oxide (Fig. 12).

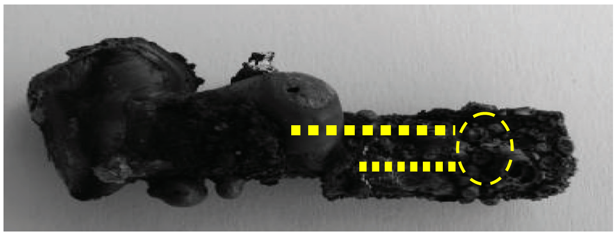

Fig. 12. A titanium support covered with the SHS product formed under microgravity.

The following specific features of formation of the microstructure of welded joining of the titanium support and the SHS product were defined using the obtained experimental results.

Figure 13 shows a microstructure of the SHS product consisting of intermetallic alloys based on Ni_2Al_3 (a light phase), $TiAl_3$ (a grey phase), and aluminum oxide (a dark phase). The intermetallide phases look like a branched dendrite structure, while the oxide phase is presented by highly dispersed inclusions. It should be noted that the particles of aluminum oxide are uniformly distributed in the volume of the intermetallide-base alloy, in spite of the fact that the oxide density is two times lower than that of the intermetallide alloy. Uniform distribution of dissimilar compounds different in their density over the melt volume is determined by the conditions of microgravity. It is microgravity which provides the conditions unfavorable for the multiphase melt deformation because of the absence of an effect of the Arrhenius force.

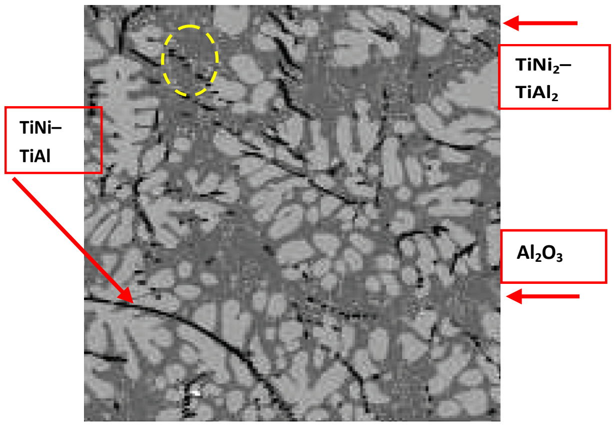

Fig. 13. The microstructure of the SHS product consisting of the alloy based on Ni_2Al_3 (light phase), $TiAl_3$ (grey phase), and aluminum oxide (dark phase).

A typical microstructure of the surface layer of the titanium support that is adjacent to the SHS product is presented in Fig. 14. Inside the support interaction proceeded via the reactive diffusion mechanism and as a result a product composed of alternating needle-like grains was formed in the solid phase.

A layer of molten eutectics was formed on the surface of the support. The data of the quantitative analysis showed the eutectics to consist of the following phases: $Ti_3Al + Ti_3Ni + 2Ti$. The composition and microstructure of the SHS product changed with the departure from the titanium support. The main portion of the SHS product layer was an alloy based on Ni_2Al_3 (bright phase) and $TiAl_3$ (grey phase).

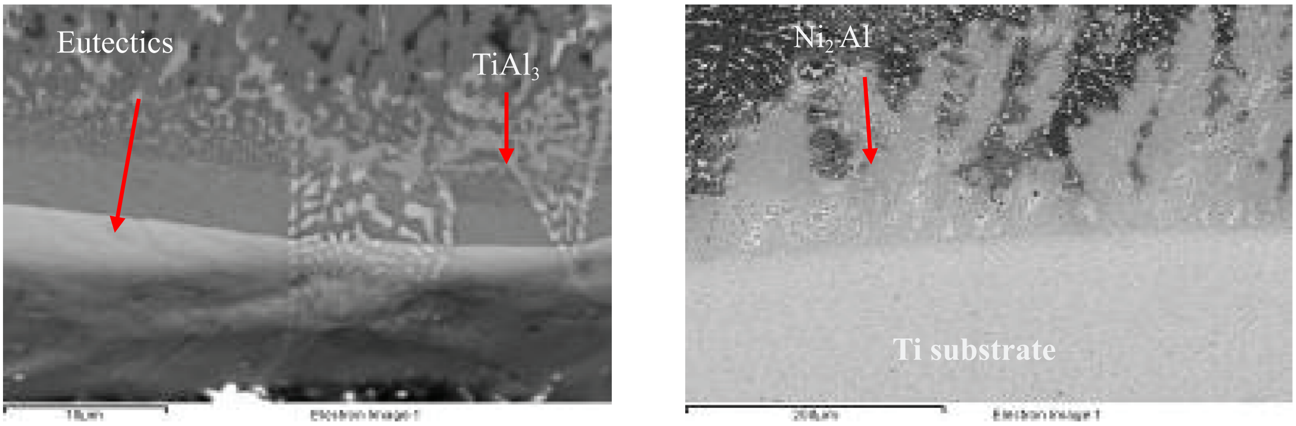

Fig. 14. The microstructure of the surface layer of the titanium support (to the left) and the SHS product on the support (to the right) obtained under microgravity.

It should be noted that there is a peculiar feature in the formation of a microstructure of the SHS product layer. Figure 15 shows the microstructure of the surface layer of the SHS product obtained under the space conditions.

It is seen that the closer was the surface of the intermetallide layer the less was the size of the structural component. An average size of the particles in the 10 μm-thick surface layer was 30-50 nm. As shown by the experimental results, a multiphase SHS product was formed under the microgravity conditions. Dissimilar compounds with a much different density were uniformly distributed over the product bulk.

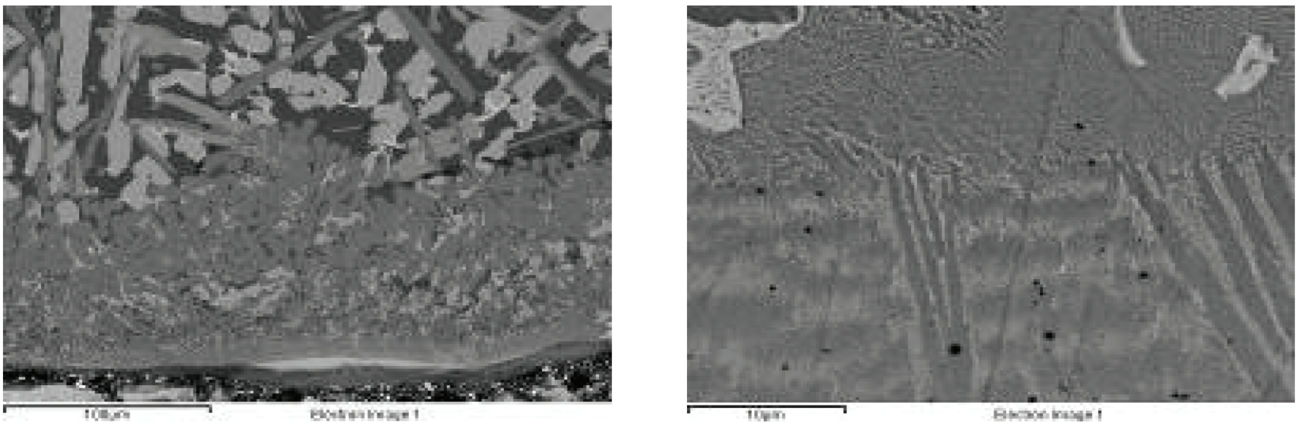

Fig. 15. The microstructure of an upper layer of the SHS product synthesized under microgravity.

The influence of gravitational forces onto both the formation of the surfaced layer on the Ti-support surface and its chemical composition was also studied. It was shown that at the interface of the molten SHS product and the titanium support an intermediate layer composed of the eutectics based on the $Ti_3Al + Ti_3Ni + 2Ti$ phases. A mutual influence of microgravity and thermal regime of the SHS process resulted in the formation of a surface welder with an average particle size of 30-50 nm.

Thus, the experiments performed in ISS-19/20 showed the potentials of employment of the thermal explosion process in the synthesis of intermetallide-based gradient materials, compounds, and coatings under microgravity conditions. The mechanism of SHS formation of coatings based on titanium aluminides and nickelides on the titanium supports was studied in the thermite NiO–Al systems.

Within the period of the ISS-21/22 flight, the space experiment ‘Self-propagating high-temperature synthesis’ was intended for the further investigations of the influence of long-term microgravity on synthesis of porous gradient materials, interaction of molten SHS components and products with various metals as well as the processes of capillary spreading.

At the same time, a larger number of the systems under study and improved design of the sample fastening allowed harvesting of much more information as compared with the previous experiments. Investigation of multilayered samples composed of a few pellets of different composition, density, and shape provided the possibility of simultaneous run of several reactions in one capsule. Thus, the available facility was used at its utmost efficiency when catching out a maximum achievable volume of scientific and practical information.

Conclusions

The program of the space experiment ‘Self-propagating high-temperature synthesis’ was enlisted into the schedule of the scientific and applied investigations and experiments that were planned to be performed in the Russian section of ISS was successfully fulfilled.

New uppermost data of fundamental and applied importance were obtained for the mechanism of the combustion process, formation of the phase composition and structure of the final products. The possibility of employing technological processes of synthesizing new materials, compounds, and coatings by the methods of SHS, SHS welding, and SHS soldering was demonstrated under the conditions of microgravity.

The obtained experimental results allowed the following conclusions to be made:

- the combustion velocity, the phase and chemical compositions of the products are practically independent of the gravity in the systems under study;
- under the conditions of microgravity, phase separation is observed in the synthesis products;
- the sample shape and surface, all structural components of the combustion products synthesized in the terrestrial and space experiments are significantly different;
- the main reasons of the observed differences are the conditions of gravity and the subsequent convective component of heat and mass transfer, i.e. slower rates of the product crystallization.

Materials with granular skeletons, foam-like materials based on SHS systems were synthesized within the frames of the undertaken experiments. Besides, experiments on obtaining compounds and coatings by the methods of SHS, SHS welding, and SHS soldering were performed under the space conditions. Analysis of the materials synthesized by SHS method under prolong microgravity showed the perspective horizons for production of highly porous foam-like materials and coatings applicable to aerospace industry under weightlessness. As shown by the experimental results SHS welding, SHS-surfacing, and SHS soldering can be used in the solution of technological and repair-curing problems under microgravity on board ISS.

Investigations in the given direction can be used in further conquering the cosmic space both for material synthesis and as heat generators (heat sources), light sources, and technological approaches to problems of repair.

Acknowledgments

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