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ALTAI CENTRE OF SCIENCE «THERMOSYNTHESIS» ON HIGH-TEMPERATURE SYNTHESIS OF NEW MATERIALS. CONDITION AND PROSPECTS

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Achievements of problem research laboratory SHS-material science founded in 1992 on the basis of I.I. Polzunov Altai State Technical University have been stated.

Discovery of self-propagating high-temperature (SH) synthesis (SHS) by A.G. Merzhanov in 1967 gave a new pulse for searching and understanding physicochemical combustion mechanisms in different aggregate states of a substance: first of all – combustion in solid phase. Method of SH-synthesis gave an opportunity of obtaining materials with unusual properties due to specific conditions of synthesis: considerable thermodynamic non-equilibrium and high temperature gradients. Altai state technical university is one of five scientific centers of the Russian Federation studying the processes of SH-synthesis. In 1992 the problem research laboratory of SHS-material science was established by our initiative and the task of the Department of Education [1–6].

Directions of research are determined: obtaining new materials by SHS method and spraying protective coats. Thus, the research carrying out was initiated. In 2003 the 5th regional Altai department of Scientific Council of combustion and blast of RAS in the field of investigation of processes of self-propagating high-temperature synthesis «TERMOSINTEZ» of non-traditional powder metallurgy, development of high technologies of obtaining composites, superalloys and products on their basis, detonating coating as well as development of measuring systems and diagnostic equipment on the basis of integral photodiode structures and microprocessor controller was established.

The research team consisting of about 30 employees includes 3 doctors and 12 candidates of science, scientists, engineers, graduate students, students. During the period of laboratory existence 5 doctor and 17 candidate theses have been written and defended. Works on the main sections are carried out:

- theoretical investigations of the processes of solid-phase combustion for developing principles of synthesis controlled mode, studying dynamics of development of combustion sources in the process of SH-synthesis, estimation of activation energy parameters, heat conductivity, investigation of weak structure formation in reacting powder system with admixture of inert material, examination of instability of combustion wave, determination of plasma influence on SHS-processes, solid-phase transformations in thin films, high-energy influences on reacting medium by concentrated energy flows etc.
- development and adaptation of new technologies of SH-synthesis, commercial development and implementation of technologies, for example, in manufacturing of filter elements and protective coatings.

A group of scientists put forward the concept of integral SHS-technologies and it is developed. Its matter is in the fact that the whole cycle of the development is carried out in respect to the general prototype (base sample) and «specialization» of materials to concrete operating conditions is implemented at the final stage by property modifying. If it is necessary to expand sample nomenclatures or change the requirements to the material only modification of basic sample is implemented. Methods of optical micro-pyrometry of dispersive media obtained considerable development.

The original experimental complex for studying thin thermal structure of combustion wave was developed (Fig. 1).



Fig. 1. *Experimental complex of micro-pyrometric and optical devices for studying processes of formation of thermal structures in the wave of SH-synthesis*

Complex technique for studying physical processes of structure-phase transformations in SHS-materials was developed. Its matter is in comparison of thermograms of high time and spatial resolution with diagrams of phase states as well as in determination of typical scales in thermal structure of combustion wave of SHS (Fig. 2).

Among the materials the technological basis of synthesis of which are the most developed the powders of refractory intermetallide compounds take special place. It is known that intermetallide compounds are characterized by a set of physicochemical properties which include high heat resistance, high-temperature strength, resistance to wear and corrosion at high temperatures, resistance to action of corrosive media etc. Synthesis was carried out in the systems Ni-Al, Ti-Al

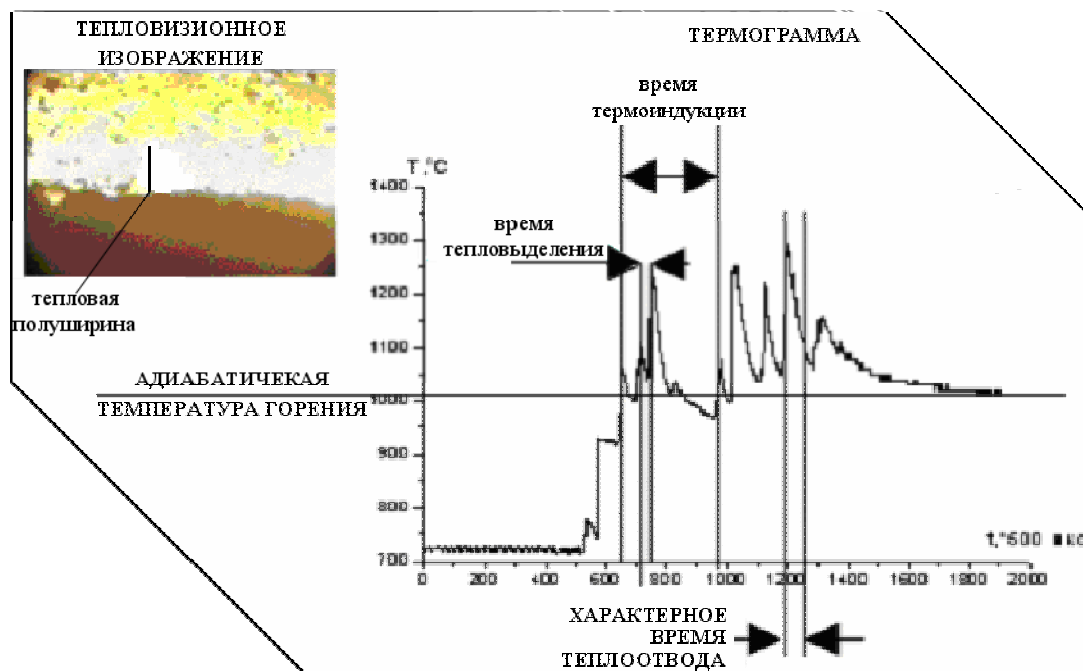


Fig. 2. Thermal structures in combustion wave front
 Тепловизионное изображение – Thermal image; Термограмма – Thermogram; Время термоиндукции – Time of thermoinduction; Тепловая полуширина – Thermal half-width; Время тепловыделения – Time of heat-segregation; Адиабатическая температура горения – Adiabatic combustion temperature; Характерное время теплоотвода – Typical time of heat extraction

the product of which were intermetallides of various stoichiometry and the influence of batch composition, powder dispersivity, heating temperature on physicochemical properties of the obtained materials was studied. Using the concept of integral technologies alloying of binary systems Ni-Al, Ti-Al was carried out. They are basic samples, elements of IV subgroup (Fe, Cr, Ti, Ni) as well as nonmetals Si, B, C. The factor determining properties is the existence of high-temperature eutectics of one of the components of basic system with alloying element. The sets of powder materials of intermetallide class for spraying supporting high level of coating operating characteristics were developed. The technology of detonation-gas spraying (DGS) of composite SHS-powders, for example, titanium boride and aluminide was created for development of integral technology concept. DGS technology characterized by high speed of mist flow (to 2000 m/s) and temperature (to 3000 K) allow supporting high level of adhesion and obtaining almost compact covering material with porosity about 0,5...6 %.

To study technologies of spraying new materials in AltSTU the experimental stand for studying pulse gas-dispersed flows on the basis of multichannel analyzer of thermal particle spectrum of condensed phase and optoelectronic time-of-flight meter of mist flow rate was developed (Fig. 3).

The processes conducting by formation of new phases occur on the surface of the base at spraying and elements of bundle take part in the reaction. As a result of exothermic processes the quality of surface link with the base improves. Thus, high temperatures and pressu-

res increased in the process of DGS may stimulate the processes of secondary structure formation or the processes of new phase formation at spraying multicomponent system. Indication of temperature of dispersed stream and base surface enables controlling the processes of structure-phase-formation, reduces significantly power inputs and material consumption for unsystematic search for optimal modes of DGS, opens wide opportunities of predicting results of spraying process carried out in some condition or other. In the process of detonation-gas spraying the issue of the dynamics of heating surface of base contact with evaporated layer is very important as contact temperature determines to a large extent the adhesive properties of coating. A special microthermocouple sensor, Fig. 4, was constructed in the laboratory of SHS-material science for controlling outside surface temperature, temperature of base inner surface and temperature of gas-dispersed flow.

Use of contact microthermocouple sensor allows solving the problem of diagnostics of disperse flow and controlling temperature of surface layer contact with the base that is not possible when using methods of optical pyrometry.

Recently the interest to the method of electric-spark alloying in respect to hardening of tillage machine members has increased. At present alloys VK are used as electrodes. Materials on the basis of titanium boride obtained by the method of SHS are alternative to them. Synthesis was carried out in the mixture Ti+3B+Ni under pressure 10 MPa, initiation was carried out by direct transmission of electric current. As a result of treatment of leading edge of tillage machine plowshare by the

method of electric-spark alloying using the obtained SHS-material on the basis of TiB_2 the coating with microhardness to 17 GPa was obtained. Alloys on the basis of titanium aluminides are widely applied in aircraft construction for manufacturing gas turbine engines and parts owing to their low specific weight, high strength to breakage, pressure, action of corrosive media at high temperatures. In AltSTU phase formation is studied at synthesis of titanium aluminides in thermal explosion mode that is of great engineering importance as it gives an opportunity to control synthesis reaction. Reactor for carrying out SHS-synthesis with tiristor control unit of heating and system of batch forced cooling as well as the device of temperature diagnostics of SH-synthesis in thermal explosion mode were developed.



Fig. 3. Experimental stand of studying pulse gas-dispersed flows

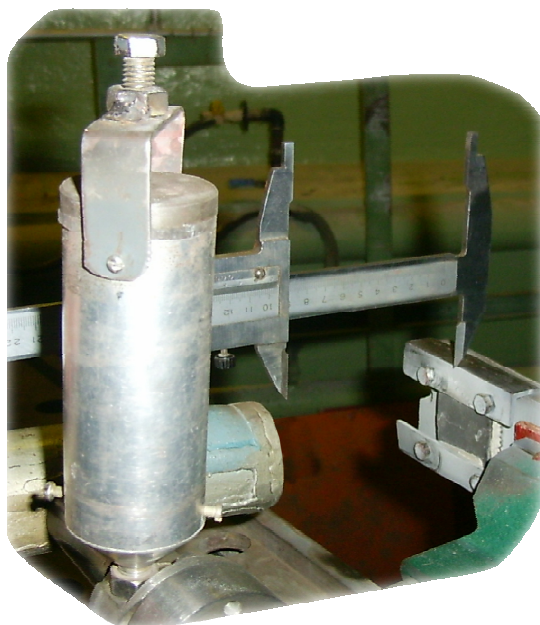


Fig. 4. Sensor for controlling temperature of spraying base surface and flow temperature

SHS-reactor was constructed for studying dynamics of heating reactive mixture in thermal explosion mode, Fig. 5.

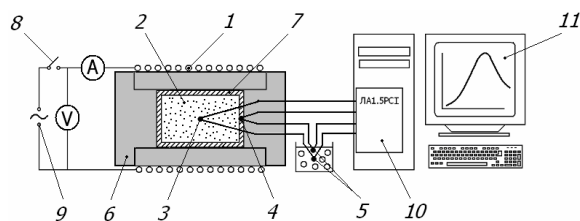


Fig. 5. Flow chart of SH-synthesis device in thermal explosion mode. 1) heating element, 2) reactive mixture, 3) thermocouple for controlling dynamics of heating mixture, 4) thermocouple for controlling wall temperature, 5) compensating thermocouples, 6) steel reactor, 7) heat-insulating asbestos layer, 8) switch, 9) voltage source, 10) DAQ board LA 1.5 PCI, 11) monitor

Porous permeable materials obtained by SHS method have been studied by the team for the last ten years. By the results of investigations the manufacturing of filters of nickel aluminides for water purification systems of power plants, ceramic filters on the basis of aluminum and iron oxides used for purification of air-gas mixtures, emulsions and solutions was mastered. Layerwise character of SHS-synthesis reaction conditions the directed character of formation of porous frame of the obtained materials that allows making permeable materials with low hydraulic resistance at retention of high filtering properties. Presence of «wall effect» allows implementing the mode of surface filtration and self-regeneration for SHS-filters at purification of gas-and-dust media with high dust condition. Efficiency of emulsion and water purification is not less than 95 %, air-gas mixtures and carbon – not less than 95...98 %. Retention factor of copper, chromium and nickel compounds in galvanic and etching solutions is not less than 95 %. Efficiency of filters varies in wide ranges from 1 to 1000 l/h. Lifetime is not less than 1 year; prevention is carried out by simple water flush or at ultrasonic device.

One of perspective directions in the work with porous permeable materials is processing waste of metallurgical industry at metal reduction from oxides with purity to 95 % and synthesis of low-exothermal compositions under the influence of electric current. Advantages of SHS on traditional metallurgical methods of obtaining materials occur when the processes of transferring heat excess in reactionary medium exceeds diffusion scale. In this case the most interesting properties of materials appear as the conditions for occurrence of several phases on the boundary of the original metals are created owing to high concentration gradients and temperature.

Talking about the unique set of physicochemical properties of materials obtained by SHS method the unique technological reproducibility and stability of these properties which is obviously the effect of autoinhibiting character «phenomenon of wave localization of solid-phase reactions» should be mentioned. That is at similar initial conditions the SHS reaction should automatically come to one and the same combustion mode. It is expected that at SHS upgrading the scales of heterogeneity not only of micro- and mesostructures but nanolevel as well are achieved when growth of specific surface of interphase results in occurrence of new opportunities

and properties. Such properties of materials may support at nanostructure level, for example, the effects of high-temperature superconductivity or «structure» memory.

In the whole the government program of supporting the development of this direction is required for SH-synthesis industrialization.

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AUTOMATED EXPERIMENTAL COMPLEX FOR RESEARCH AND CONTROL OF DETONATION STREAM AT PARTICLE SPRAYING

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The opportunity of application of image input system to PC on the basis of PZS with electronic shutter and exposition time 35,5 mks in structure of complex of optical control of particle spraying detonation stream characteristics on installation «Katun-M» has been shown. The results of inspection of particle speeds by the length of their tracks on image, dynamics of gas fuse formation at the initial moment of stream occurrence on section of installation shaft and root angle of the stream are given.

Search for the ways of intensifying the process of coating the surface and automated control of the process requires the development of the bases of detonation spraying technologies. The most important stage of detonation spraying process is propagation of detonation wave (DW) in detonation set shank and interaction of powder with DW and high-speed gas flow of combustion products. The analysis of physical phenomena at this stage allows approaching more substantiated to selection of spraying modes. The examination of literary sources shows that occurrence of detonation spraying processes was studied both experimentally and theoretically. In spite of significant amount of issues devoted to detonation coating there are almost no theoretical and experimental investigations of two-phase flow dynamics, course of two-phase flow occurring at output from the channel to submerged space and its interaction with the barrier (substrate where the powder is coated) that allows determining process energy parameters. The comparative analysis of some characteristics and processes of binding materials coated by gas-thermal coating on the basis of works [1–3] confirms the advantages of detonation-gas coatings among which high cohesive strength with sprayed surface and low porosity may be singled out.

The process of spraying differs in pronounced non-stationarity the level of which influences significantly the selection of technological parameters of equipment. Examination of domestic and foreign issues gives the information on technology of detonation coating far from being complete. There are considerable divergences in values of spraying parameters in the modes of obtaining coatings from one and the same material. It can be explained by the fact that detonation spraying represents complex multiparameter process. Separate experimental one-way dependences given in a number of issues are made for some concrete conditions of spraying or at certain equipment therefore, they do not always correctly and fully reflect regularities inherent to the studied process. Besides, the given references are difficult to be applied at changing conditions and equipment. The analysis of laws peculiar to the process of detonation coating should precede the selection of technology and equipment [2].

M.Kh. Shorshorov and Yu.A. Kharlamov [3] put forward the concept of coating formation at detonation spraying. It considers dislocations as active centers within which chemical interaction is implemented. The authors put forward the supposition according to which the increase of pressure in contact zone results in inten-