Vladimir Isaakovich Yukhvid is one of the most famous scientists in the field of the theory and practice of self-propagating high-temperature synthesis (SHS), the leader of such directions as SHS-metallurgy of refractory compounds and hard alloys, SHS-surfacing, centrifugal SHS-casting. Based on the results of his research, V.I. Yukhvid has published more than 200 scientific papers, received more than 80 patents of the Russian Federation and copyright certificates of the USSR. His students have defended 10 candidate and 4 doctoral theses.

V.I. Yukhvid was born on October 18, 1946 in Ufa. In 1964 he finished secondary school in the village of Aksakovo, Bashkiria. After graduating from the Polytechnic Institute in the city of Samara in 1970, he took a post-graduate course and was sent to carry out research in A.G. Merzhanov's Laboratory in the branch of the Institute of Chemical Physics. In 1973 he defended his candidate thesis on the specialty 01.04.17 – Chemical Physics, including Physics of Combustion and Explosion. In 1975 he was hired by the Institute of Chemical Physics of the Academy of Sciences of the USSR. Since then his scientific activity has been aimed at studying materials-forming processes of combustion. This new scientific trend, created by the School of academician A.G. Merzhanov, was named self-propagating high-temperature synthesis (SHS) of refractory inorganic compounds. In 1990 V.I. Yukhvid defended his doctoral thesis, and in 1991he was awarded the title of Professor.

Experimental and theoretical studies of chemical transformations in high-calorific thermite-type systems conducted by V.I. Yukhvid revealed a new exciting picture of synthesis and allowed V.I. Yukhvid to develop the fundamentals of the process control. They also demonstrated the possibility of their implementation in auto-wave mode at a high temperature of 2500–4500 °C and obtaining combustion products (carbides, borides, silicides, metal and non-metal oxides, composite materials based on them, etc.) as cast.

In 1987, V.I. Yukhvid became head of the laboratory in the newly organized Institute of Structural Macrokinetics, USSR Academy of Sciences, where he continued his studies. This area of SHS research was named SHS-metallurgy. To test new materials in industry, under the leadership of V.I. Yukhvid there was designed and manufactured unique experimental equipment (centrifugal set-ups and reactors). These studies have the perspective of industrial application in the aircraft propulsion engineering and defense technology, metal working and ecology, raw hydrocarbons processing, etc.

On the basis of fundamental research V.I. Yukhvid has developed new technological approaches for solving a number of applications:

– obtaining cast materials – carbides, borides, silicides, metal and non-metal oxides, high-temperature alloys and composite materials;

– obtaining cast monolayer and multilayer pipes of metal, oxide and composite materials;

– SHS-surfacing of protective coatings from corrosion resistant and hard alloys to steel and titanium bases.

V.I. Yuhvid and the staff of his laboratory are actively involved in joint research with research institutes, universities and industrial enterprises to develop new materials to meet the challenges of aircraft propulsion engineering and defense technology, metalworking and ecology, raw hydrocarbons processing, etc.

Professor V.I. Yukhvid has repeatedly made reports at Russian and international conferences. Under his leadership the team of the Laboratory of liquid phase SHS-processes and cast materials has repeatedly received grant support from the Department of Chemistry and Materials Science RAS, Presidium of the Russian Academy, Russian Foundation for Basic Research, Civilian Research and Development Foundation, Federal Target Programs and other organizations. For many years V.I. Yukhvid has been actively engaged in teaching activity at the basic departments of Samara Polytechnic Institute and Moscow State University.

At present, V.I. Yukhvid is the head of the ISMAN Laboratory of liquid phase SHS-processes and cast materials, a member of the Scientific and Dissertation ISMAN Councils, a member of the Dissertation Council of Samara State Technical University, co-chairman of the ISMAN scientific seminar, a member of the editorial board of scientific journals "Combustion and Plasma Chemistry" and "Vestnik of Samara Technical University".

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SHS-Metallurgy: Fundamental and Applied Research

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Abstract

Currently, to create refractory ceramics, composite materials and alloys the industry uses long-time sintering and melting techniques in high-temperature set-ups. The School of Academician A.G. Merzhanov has developed a new technology to produce this group of materials based on combustion, called self-propagating high-temperature synthesis (SHS), which enables to reduce synthesis time (up to 1–2 minutes), eliminate complex high-temperature equipment and minimize energy consumption. One of the most promising areas in the SHS-processes research is SHS-metallurgy. To synthesize refractory inorganic materials SHS-metallurgy uses highly exothermic thermite-type mixtures. Such mixtures are capable of burning, and their combustion temperature may exceed 3000^0, which allows for producing a wide range of refractory materials as cast. The article provides an overview of the main results obtained to date in research on SHS-metallurgy. To implement SHS-metallurgy taking into account its specific features there has been developed and manufactured the unique equipment, reactors and centrifugal set-ups, which allow for carrying out and studying the processes under gas pressure and overload. In experimental studies, the influence of mixtures characteristics (ratio of components in the initial mixture, its density and calorific value, dispersion of reactants, the type of the reducer, so on), process parameters (pressure and overload), the scale factor (mass and geometry of the reaction volume), etc. on technological parameters (combustion rate, weight loss during combustion, complete phase separation), chemical and phase composition, structure and properties of the combustion products was found. In applied research there has been developed synthesis technology for a wide range of cast refractory inorganic materials (ceramics, alloys and composite materials), products and protective coatings.

Keywords

High-temperature synthesis; structural macrokinetics; combustion; ceramics; composite materials.

Introduction

The fundamentals of SHS-technology were developed in 60-80s of the 20th century by the school of Academician A.G. Merzhanov [1–4]. In the framework of these studies in 1975–1985 there was formed one of the directions called SHS-metallurgy [5–11]. In these studies the main directions of the fundamental and applied research were formulated, including synthesis of a wide variety of cast refractory inorganic materials: ceramics, alloys and composite materials, as well as manufacturing products made of them and facing protective coatings. SHS-metallurgy uses a mixture of powders of one or more metal oxides with a metal reducer (Al, Mg, Ti, etc.) and nonmetal (C, B, S, etc.) capable of burning as initial feedstock. Aluminum is most commonly used as a metal reducer.

In the independent research in 1980–1982, O. Odawara with his colleagues (Japan) conducted a study of iron-aluminum termite combustion and developed the technology for producing large-sized pipes [12]. In 1990 S. Vuytitsky (USA) designed a radial centrifugal set-up and conducted the first experiments to produce cast hard alloys based on tungsten carbide [13]. Later centrifugal SHS-technology was developed by Lai Ho-Yi and his colleagues and others (China) and G. Cao (Italy) with his staff and others [14].

At present SHS-metallurgy is researched in Russia, Armenia, Kazakhstan, Georgia, Europe, USA, Japan, Turkey and others [15–17]. The work is carried out in the following directions:

– fundamental research (experiments, thermo-dynamic calculations, process modeling);

– applied research (synthesis of refractory materials important for practice, facing protective coatings, obtaining products);

– industrial implementation (joint developments).

To perform research new set-ups are designed and manufactured and new research methods are developed.

The article aims to review the main results obtained up to date in the research on SHS-metallurgy.

Objects, Methods of Research, Phenomenology

Before the combustion, mixtures are placed in refractory molds (made of quartz or graphite) and compacted. Typically, combustion is initiated by an electrical coil, which heats the surface layer of the mixture up to ignition temperature. After ignition the combustion front is formed, which is spread over the mixture.

In the combustion front chemical transformation of the initial mixture into the final products proceeds. In general, the scheme of chemical conversion can be written as:

$$\sum v_i A_i + v_2 R + v_3 B \rightarrow v_4 C + v_5 R_x O_y + \text{impurities},$$

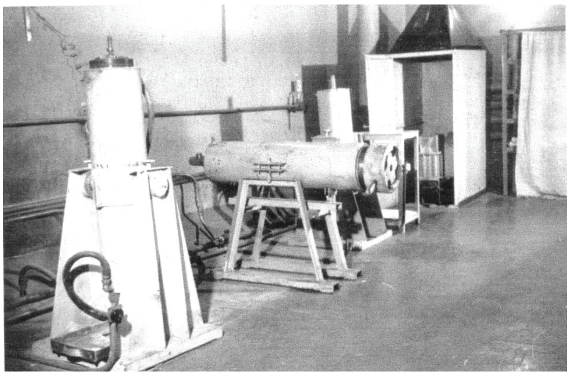
where A_i – metal oxide; R – reducer; B – nonmetal; C – compound, R_xO_y – reducer oxide. Impurities may be condensed (residues of initial reactants) and gaseous (vapors and suboxides). Below are the examples of reactions.

$$WO_3 + 2Al + C \rightarrow WC + Al_2O_3;$$

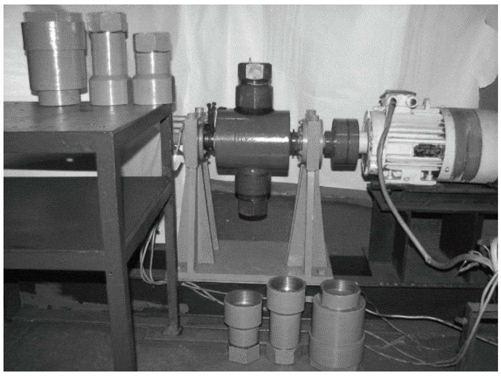
$$3MoO_3 + 6SiO_2 + 14Al \rightarrow 3MoSi_2 + 7Al_2O_3;$$

$$3NiO + 2Al + 3B \rightarrow 3\ NiB + Al_2O_3.$$

High temperature of thermite mixtures combustion (up to 3000–4000 °C) allows you to obtain the products of combustion in a liquid phase state. Under the influence of gravity the separation of melts of metallic and oxide phases of combustion products occurs. High combustion temperature also leads to intensive gas formation and spread of the melt at atmospheric pressure. High pressure and centrifugal effect allow suppressing the spread, therefore SHS-metallurgy is carried out in reactors (Fig. 1 *a*),



a)



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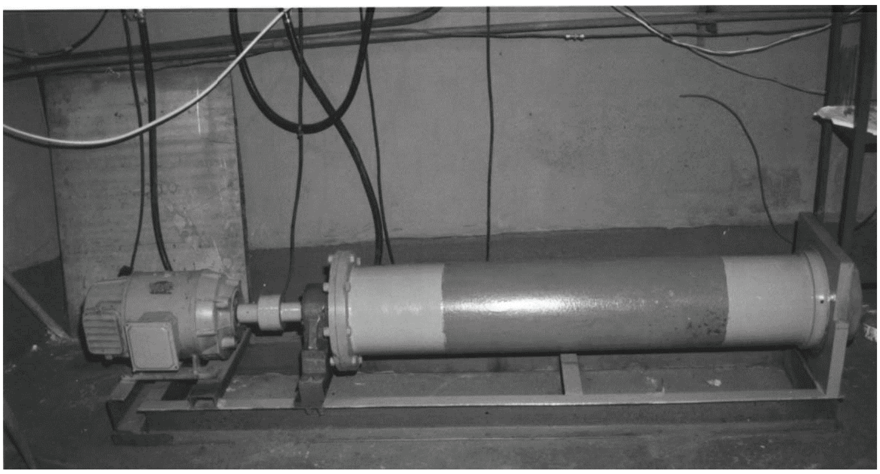


c)

Fig. 1. Equipment for SHS-metallurgy and study of its regularities:
a – reactors for synthesis under gas pressure; *b* – radial centrifugal set-up for carrying out the synthesis under the effect of overload; *c* – axial-centrifugal set-up for producing tubes

under nitrogen or argon pressure of 0.1 to 10 MPa and centrifugal plants with overload ranging from 1 to 1000 g (Fig. 1 *b*, *c*).

Phenomenological studies have shown that the process of SHS-metallurgy proceeds stage by stage. There are 3 main successive stages (Fig. 2). In the first stage (Fig. 2 *a*) combustion occurs, the product of which is a two-phase melt. In the two-phase melt the droplets of metal phase are distributed in oxide medium. In the second stage (Fig. 2 *b*) due to the difference in densities, separation of metallic and oxide phases (phase separation) is carried out under the action of gravity field. In the third stage (Fig. 2 *c*) combustion products are cooled and crystallized.

Combustion in high-calorific mixtures of metal oxides with reducers and non-metals at atmospheric pressure is accompanied by strong spread of combustion products and proceeds in the explosive regime. Increased gas pressure (of argon, nitrogen, air) and overload (centrifugal effect) inhibit the spread.

After combustion, gravity separation of metalic and oxide phases and subsequent cooling the combustion products have a two-layer cast cylinder form, with a clear separation of metal and oxide layers (Fig. 3 *a*). The total height of layers is 3–4 times less than the height of the layer of the initial mixture, because the density of the molded products is higher than the density of the initial mixture.

To describe the processes proceeding during synthesis, 3 characteristic times can be introduced: combustion time (t_1), the time of gravity phase separation (t_2), the time of melt cooling (t_3). $t_1 = H/u$ is determined by the ratio of the height of the initial mixture layer (H) and the linear combustion rate (u). $t_2 = h/v$ is determined by the ratio of the melt height of combustion products (h) and the velocity of the metal drops movement in the oxide melt (v), where $v = (\rho_2 - \rho_1)\, d_m^2 a/18\mu$, μ – the viscosity of the oxide phase, $(\rho_2 - \rho_1)$ is the difference in the densities of metallic and oxide melts, a is the value of the overload (in the case of experiments on the centrifugal plants $t_3 \sim d^2/\chi$, where d is diameter of the reaction volume, χ is thermal diffusivity. To provide complete yield of metal phase in the ingot the following conditions must be satisfied:

– a two-phase melt must have high temperature exceeding the temperature of metalic and oxide products;

– the time of separation of metallic and oxide phases must be greater than the cooling time up to the crystallization temperature of the end products;

In the experiments the influence of mixtures characteristics (ratio of components in the initial mixture, its density and the calorific value, dispersion of reactants, the type of the reducer, etc.), process parameters (pressure and overload), the scale factor (mass and geometry of the reaction volume), etc. on process parameters (combustion rate u, weight loss during combustion η_1, completeness of phase separation of the ingot η_2), chemical and phase composition, structure and properties of the combustion products was estimated.

Process parameters were calculated according to the formulas: $u = h/t_g$, $\eta_1 = [(M_1 - M_2)/M_1]100$ % – relative weight loss during combustion, $\eta_2 = (m/m_p)100$ % – the completeness of the yield of

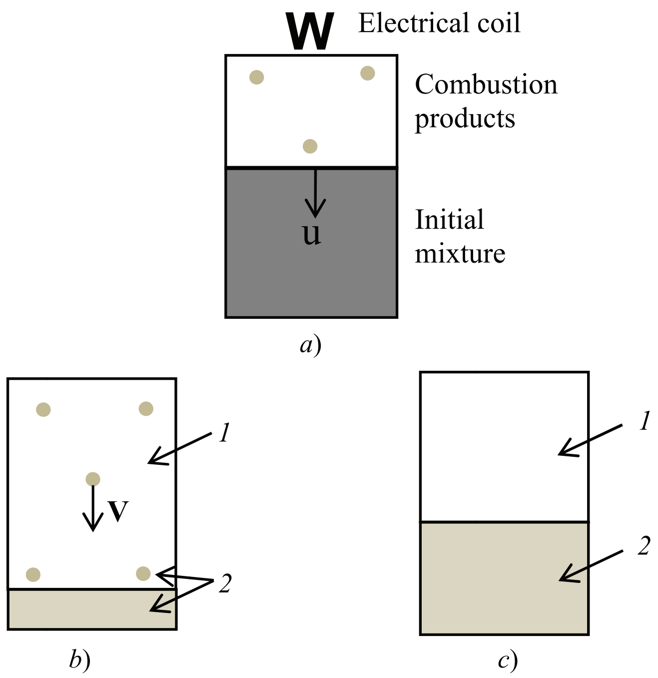


Fig. 2. The main stages of SHS-metallurgy:
a – combustion and chemical transformation;
b – gravity phase separation;
c – cooling and formation of a crystalline structure;
1 – oxide phase *2* – metallic phase

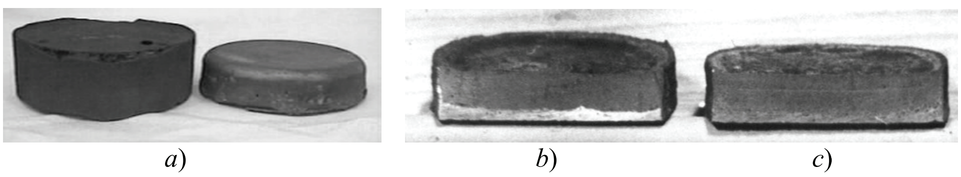


Fig. 3. Macrostructure of the combustion products in:
1 – complete separation; *2* – partial separation; *3* – complete non-separation

target elements in the ingot, where h is the height of the layer of the initial mixture, t_g is time of layer combustion, M_1 and M_2 are the masses of the initial mixture and combustion products, m и m_p – experimental and calculated masses of the ingot.

To study the microstructure and chemical composition of cast alloys we used a field-emission scanning electron microscope with ultrahigh resolution Zeiss Ultra plus on the basis of Ultra 55 equipped with an X-ray microanalyzer and JCXA-733 "Superprobe", JEOL. To study phase composition we used X-ray diffractometer ARL X'TRA (Basic X'TRA System with Peltier Detector) [17–22].

Laws of SHS-Metallurgy: Process, Composition and Synthesis Products Structure Control

Regularities of combustion. Combustion of high-calorific mixtures of metal oxides with reducers and non-metals under atmospheric conditions is accompanied by a strong spread of melt and proceeds in explosive mode. Increased gas pressure (of argon, nitrogen, air) and overload (centrifugal effect) suppress the spread. Combustion takes place in the frontal mode. The average linear combustion rate ranges from a few millimeters to a few centimeters per second. The velocity of the combustion front movement can be changed several times, by varying the pressure, overload, dispersion of the reagents and ratio of reactants in the mixture.

With increasing pressure and overload, the rate of combustion of thermite-type mixtures, generally increases according to power law. In some cases, these relationships are more complex.

The phenomenology of combustion essentially depends on the dispersion of the reactants. In most experiments, dispersion of reagents of thermite mixtures is from 1 to several micrometers. Observation and video filming showed that combustion of mixtures having reactants particle size from 1 to several micrometers proceeds in a steady mode with practically planar front which moves through the mixture at a constant rate.

With an increase in the dispersion of aluminum and non-metals (carbon, boron or silicon) up to 0.1–1.0 mm the combustion front becomes uneven. In the process of movement the front shape changes continuously, but the front unevenness is substantially less than the height of the tablet.
The nature of the influence of particle size of the reducer (aluminum) and nonmetal (carbon and boron) on the combustion rate is opposite: with an increase in the size of aluminum particles combustion rate decreases, and with increasing particle size of nonmetal it increases.

Regularities of phase separation. The completeness of the yield of metallic phase (carbides, borides, hard alloys, etc.) into an ingot can be controlled by overloading the phase separation process and cooling two-phase melt by inert additives. This allows obtaining three classes of materials: separated, gradient (partially separated) and cermet (mixed). All of them are used in practice. Fig. 3 *a*, *b* and *c* present the results of separation completeness control of metallic and oxide phases in the range from complete separation to complete non-separation. These approaches are universal and used for a wide range of mixtures.

Regularities of formation of chemical composition of cast products. In general, three phases are formed under combustion: a high-temperature melt, dispergat and gaseous products. After crystallization of the melt, in the case of complete phase separation a two-layer ingot is formed, in which the metallic and oxide phases and clearly separated.

For example, the chemical composition of the phases formed during combustion of a mixture of oxides of chromium, titanium and nickel with aluminum and carbon is presented in Fig. 4. Each phase

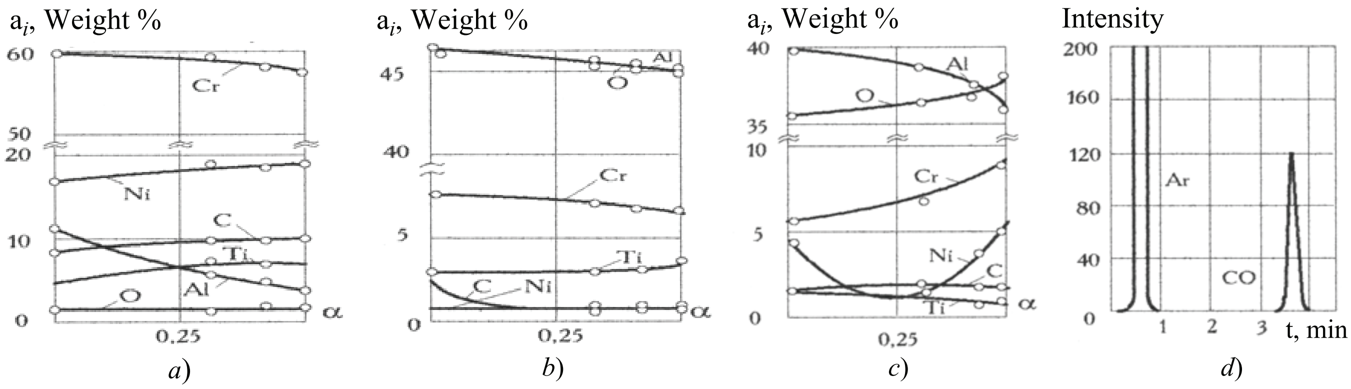


Fig. 4. Chemical composition of the final products of mixture $CrO_3 - TiO_2 - NiO - Al - C + \alpha Cr_2O_3$ combustion:
a – a metallic layer; *b* – oxide layer; *c* – dispergat; *g* – gas phase

contains the entire set of source elements, but their mass fractions are different. The metallic phase mainly comprises chromium, titanium, nickel, carbon, and Al as the main impurity. The oxide layer and dispergat have a similar composition and consist mainly of Al_2O_3. Chromatographic analysis showed that the gaseous phase of combustion products in an argon atmosphere contains CO after cooling. Suboxides formed according to thermodynamic calculations during combustion are condensed (with subsequent decomposition) on the cold wall of the reactor.

Optimization of the composition of the initial mixture of reactants dispersion allows for obtaining the calculated content of elements in the ingot.

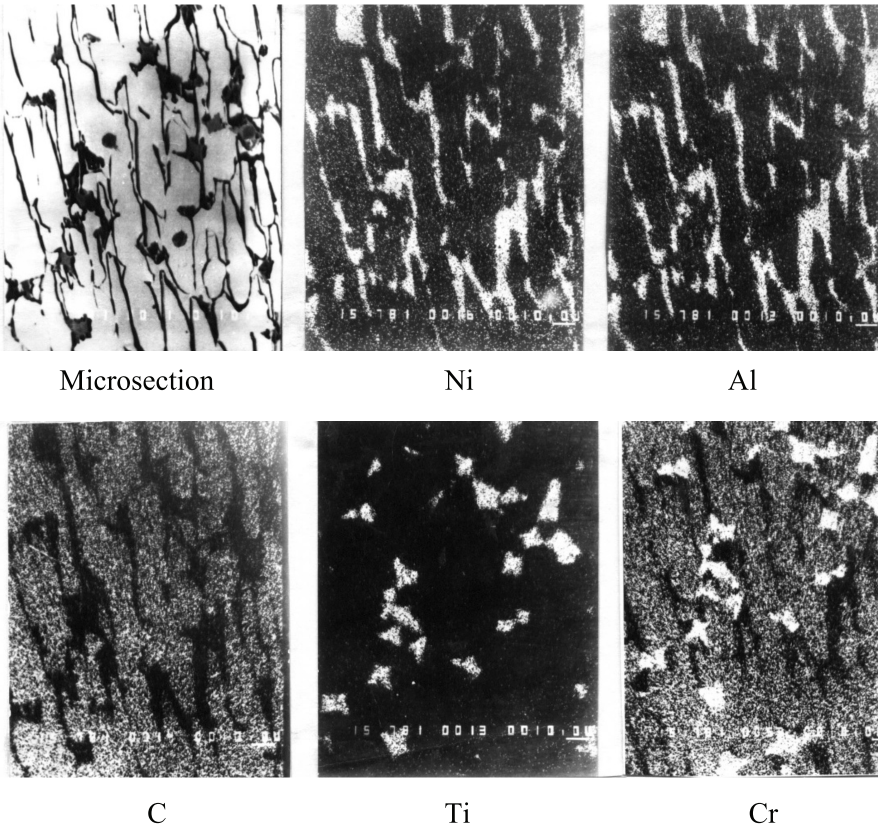


Fig. 5. Microstructure and composition of the structural components of the composite material Cr–Ti–C–Ni–Al

Formation of the phase composition, macro- and microstructure of cast products. One of the main factors determining the macrostructure of the cast material obtained from the thermite mixture is the degree of gravity separation of the metallic and oxide phases. It was shown above that by regulating the degree of gravity separation, three types of macrostructure can be obtained: 1 – a two-layer structure with a clear separation of the metallic and oxide layers; 2 – a cermet structure in which the metallic phase is distributed as particles in the oxide matrix; 3 – a gradient structure in which some part of the metallic phase is precipitated in a layer and the other part is distributed in the oxide matrix. This section focuses on the formation of the microstructure of the metallic layer under complete separation of the metallic and oxide phases.

Fig. 6. Microstructure of a composite material Cr–Ti–C–Ni–Al–Mo

The most complex microstructure is that of multicomponent composite materials. For example, from the melt Ti–Cr–C–Ni–Al matrix of Ni–Al is formed, in which "large" chromium carbide plates and “fine grains” Ti–C are distributed (Fig. 5).

When molybdenum is introduced into the system, it is dissolved mainly in the titanium carbide grains which take spherical shape (Fig. 6). Under the effect of overload there proceeds a strong grinding of carbide grains, both on the basis of chromium carbide and that of titanium carbide. A similar effect of overload was found for many refractory compounds and composite materials on their basis [23–35].

Applied Problems and Technologies

In the process of combustion of mixtures of metal oxide, reducer and nonmetal there occurs chemical transformation of the initial mixture, and a two-phase high-temperature melt of combustion products is formed. It consists of metallic phase and a reducer

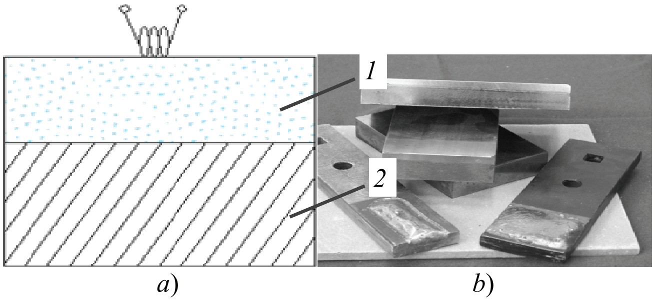


Fig. 7. Surfacing scheme:
a – under the gas pressure of hard alloys on steel basis;
b – surfaced steel samples; *1* – initial mixture; *2* – and steel base

metal oxide. Using the liquid-phase state of synthesis products, three classes of applied problems can be solved and the following products can be obtained: 1 – cast refractory compounds and composite materials; 2 – cast protective coatings; 3 – cast products.

Processing plants (reactors, centrifuges) allow for producing large ingots and products with a weight of up to 5–10 kg and overlaying the protective coatings as well.

The flow diagram of preparation and carrying out the synthesis and operating time of product batches is similar and includes: drying, dispensing and mixing of components, filling in the mold, synthesis in the reactor, removing the mold and extracting cast material or a product. If necessary, mechanical treatment of ingots is performed. Powders and metal oxides of different purity, including crude ore are used for synthesis.

Production of cast materials. Experiments in a laboratory reactor with low masses of the initial mixture (from 20–30 to 100–150 g) have shown the influence of gas pressure, mixture composition, reagents dispersion on the process characteristics and chemical composition of combustion products. In the synthesis of large ingots similar effect was found. However, experiments with large masses (from 1 to 10 kg) in a pilot scale reactor showed a strong effect of the scale factor on the process parameters and composition of synthesis products. Thus, for example, while obtaining chromium carbide it was found that with increasing mass of the initial mixture the completeness of phase separation and carbon content in the ingot increases considerably. The pressure in the reactor increases considerably, from 4 to 12 MPa. Change in the chemical composition of large ingots requires adjustment of the initial mixture, and a rise in pressure limits the mass of the initial mixture in the reactor.

SHS-surfacing. Experimental studies have shown that in the combustion of high-temperature mixtures of oxides with aluminum and nonmetals under gas pressure on the surface of a steel sample (Fig. 7 *a*) a cast coating is formed, which is uniformly distributed and tightly bound to the base (Fig. 7 *b*). The uniformity of spreading on the base is affected by the mass of the initial mixture, the temperature of combustion, gas pressure, etc. The minimum thickness of SHS-coatings is 2–3 mm and the maximum thickness depends on the ratio between base masses and charge material layer and is limited by melting of the sample.

Using SHS-surfacing technique, considerably hard coatings of hard alloys based on carbides and chromium and titanium borides were obtained (Fig. 8 *a*). Local X-ray spectral and X-ray phase analysis revealed 3 zones in the surfaced sample: the coating itself, transition zone and a steel base (Fig. 8 *b*). The coating itself has a composition structure in which in the matrix of the solution of Fe, Cr, Ti in Ni the grains TiC, Cr_3C_2 и $(Cr,Fe)_7C_3$ are distributed. It is obvious that Fe gets into the coating from the base. Its content is from 10 to 30 wt. %. The elements that make up the cast coating are evenly distributed along its height (Fig. 8 *b*). Under the overload one can accomplish the surfacing of thin (up to 1 mm) coatings in which the iron content does not exceed 5–6 wt. %.

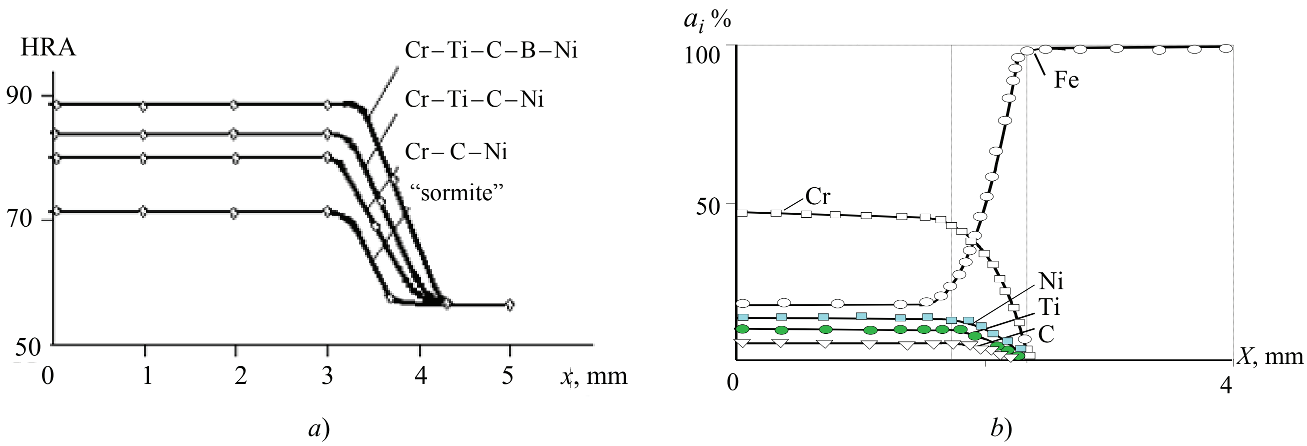


Fig. 8. Hardness distribution (*a*) and cumulative distribution of elements (*b*) along the height of coatings on a steel plate

SHS Centrifugal option. SHS-metallurgy in centrifugal set-ups allows us to solve a wide range of applied problems, such as:

– obtaining refractory compounds and composite materials from the thermite-type mixtures with low thermal effect, and also in the case when specific weights of metallic and oxide phases are close;

– filtration SHS-impregnation by metal melts of highly porous combustion products of elemental mixtures;

– manufacturing products of cylindrical and tubular shape, applying protective coatings in pipes, etc. [36–51].

Using SHS-Metallurgy to Solve Practical Problems

After synthesis and phase separation metallic and oxide products of SHS-metallurgy take the form of cast layers with a clear boundary between them and are easily separated. Strong alloys and composite materials can be used for manufacturing products. Brittle carbides, borides, silicides, oxides and others are ground in the disintegrator and classified. The ground materials are used as raw materials for various applications of powder metallurgy. Pilot technologies and technology regulations on a wide range of cast materials and powders of them have been developed to date.

Application of SHS-materials in aircraft propulsion engineering. In order to increase operating life and ensure the competitiveness of domestic gas turbine engines (**GTE**) it is necessary to create new heat-resistant alloys for manufacturing engine parts. The turbine blades of GTE which have complicated outer shape and geometry of the internal cavity (Fig. 9 *a*, *b*) experience the strongest damaging effect.

Hollow turbine blades are manufactured by casting in corundum molds. Heat-resistant alloys enter into chemical reaction with the mold walls, which results in a defect layer (waste). To form the cavity shape generating cores are used whose removal after casting is a difficult task. Development of new refractory materials for casting GTE blades is an important task.

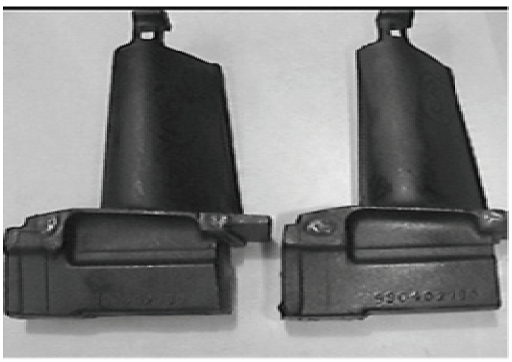
Below are SHS-metallurgy facilities to produce high-temperature heat-resistant materials.

Heat-resistant alloys. To develop new multicomponent alloys based on cobalt and nickel aluminides with the alloying additions Cr, Nb, W, Mo, Ti, C, Si, etc., SHS-centrifugal technique was used. Technological centrifugal set-up is shown in Fig. 10 *a*. Mixtures of oxides with aluminum and nonmetals are used as the initial charge. Optimization of the initial mixtures composition, the overload level allowed for obtaining SHS-alloys which are similar in chemical composition to industrial aviation heat-resistant alloys ZhS6U and HTN 61.

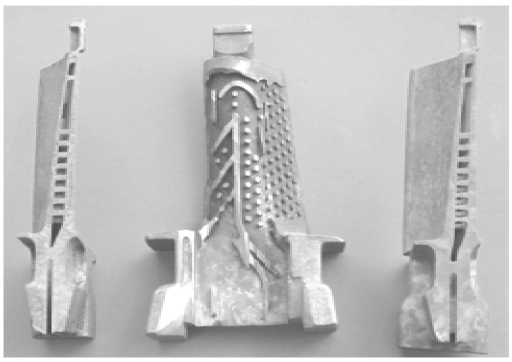
Having the same chemical composition industrial alloy HTN61 and SHS-alloys have different structures. Industrial alloy is not uniform in its volume, it has large inclusions (up to 100 μm) of strengthening phases (carbides Nb, Cr, and intermetallides). On the contrary, SHS-alloy is a homogeneous structure and has a small size of the strengthening phase. The size of the structural components has decreased more than 10 times (Fig. 10).

This noticeable difference of microstructure is due to the peculiarities of SHS-process (high synthesis temperature ~2500 °C and intensive mixing of the metal melt caused by gravity convection).

Oxide solid solutions. In the joint research of ISMAN and FSUE MMPP "Salut" it was shown that SHS-metallurgy allows for synthesizing cast solid solutions based on corundum with different content of chromium oxide in them. Similar results were obtained in the synthesis of cast solid solutions based on quartz with different content of chromium oxide in them. Tests have shown that both materials have a high

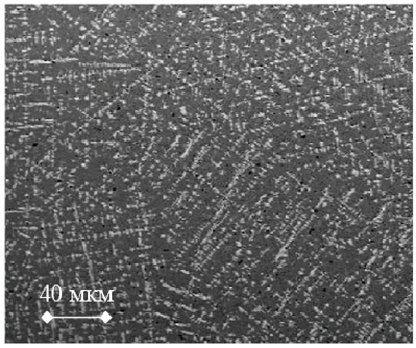


a)



b)

Fig. 9. Cast hollow gas turbine engine blades made of industrial nickel-based superalloys:
a – appearance; *b* – internal cavity of a complex shape



a) *b*) *c*)

Fig. 10. Pilot centrifugal SHS-set-up (*a*), ingots of nickel and cobalt-based superalloys (*b*) and microstructure of the ingots (*c*)

resistance to high temperature metallic nickel-based melts and can be used for fabricating cast molds and shape generating cores in the production of GTE blades. Detailed studies revealed that the first material, $Al_2O_3 \times Cr_2O_3$, is promising for making molds, can significantly improve the quality of GTE blades made of alloy ZhS6U:

– surface cleanliness from 5–6 to 6–7 class;
– grain size in the blade wall 4–6 times;
– casting material strength by 20 %.

The second material, $SiO_2 \times Cr_2O_3$, is promising for the production of mold cores, forming cavities in GTE blades. This material has a unique amorphous structure, it well sinters and has the following advantages over the commercially used corundum:

– high strength (10 %);
– smaller k.t.r. (10 times);
– can be easily removed from the blade by alkali.

Powders for applying protective coatings. In industry coatings are produced by plasma and detonation spraying, gas-thermal and electric arc surfacing. For plasma spraying the powders with dispersion of 40–80 μm are used, and for surfacing – 300 μm. In the manufacture of surfacing strips and wire the range of powders dispersion can be considerably wider. In this regard, a study on the grinding process of SHS-material ingots was undertaken.

Grinding of cast SHS-materials is carried out in two stages. In the first stage the ingots are passed through a jaw crusher to obtain large granules, and in the second stage – the granules are ground in the set-up in a ball mill, followed by classification. Brittle materials, such as carbides, borides and silicides most easily lend themselves to grinding. When a binder is introduced into the cast material, grinding becomes complicated, and if its content is sufficiently large, grinding becomes impossible. Such materials require different, more severe methods of grinding.

A number of cast SHS-materials have been tested as a basis for protective coatings. Characteristics of the coatings are shown in Table. 1. The coatings have superior properties and can significantly increase the operating life of details used in heavy wear.

Granules with high catalytic activity. SHS-metallurgy technique with subsequent chemical activation has been used to create new catalytic materials – polymetallic alloys Ni–Co–Fe–Mn–Al with a highly-developed Raney surface structure. These catalysts have shown high efficiency for solving environmental problems to neutralize combustion products of hydrocarbon fuels.

In the combustion of hydrocarbon fuels environmentally harmful impurities, such as, carbon monoxide, various hydrocarbons and nitrogen oxides

Table 1

Characteristics of gas-thermal coatings

Coating material	Adhesion strength, $*10^{-7}$, н/m^2	Porosity, %	Microhardness, H 10^{-7}, н/m^2	Coating thickness, μm
Cr_3C_2–Ni		8–10	1500	350
Cr_3C_2–Ni–Al	5,6	5–10	2500	–
Cr–Ti–Si	1,2	6–7	700–1050	350
Pink corundum	1,3–2	3–12	2100	350

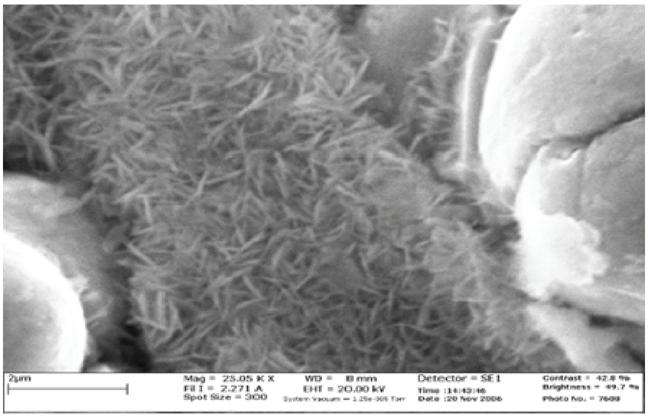


Fig. 11. Microstructure of the surface of the catalyst particles of the polymetallic alloys Ni–Co–Fe–Mn after leaching Al

are formed. For their neutralization precious metals – platinum, palladium and ruthenium are used. Precious metals have high catalytic activity, they are effective neutralizers but are very expensive, therefore their replacement by polymetallic alloys with comparable catalytic efficiency is a perspective task.

The development of polymetallic catalysts was performed in three stages: stage 1 – auto-wave synthesis of ingots of multicomponent nickel-based intermetallides with high Al content; stage 2 – obtaining polymetallic granules by grinding an ingot; stage 3 – leaching Al from the alloy and creating highly active skeleton structure.

After leaching, the surface of the granules acquired unique nanoscale Raney structure (Fig. 11). Studies have shown a high catalytic activity of the resulting granules, comparable with the activity of platinum catalysts. Full conversion of carbon monoxide and propane was achieved at a temperature of 300 °C. Filters based on polymetallic granules are currently used in Moscow municipal services at the reserve diesel power plants in order to ensure the environmental safety of the city.

Multilayer materials and cast protective covers. The technique of SHS-surfacing protective coatings of solid tungsten-free hard alloys has been

Table 2

Comparison of SHS-surfacing operating life and its analog

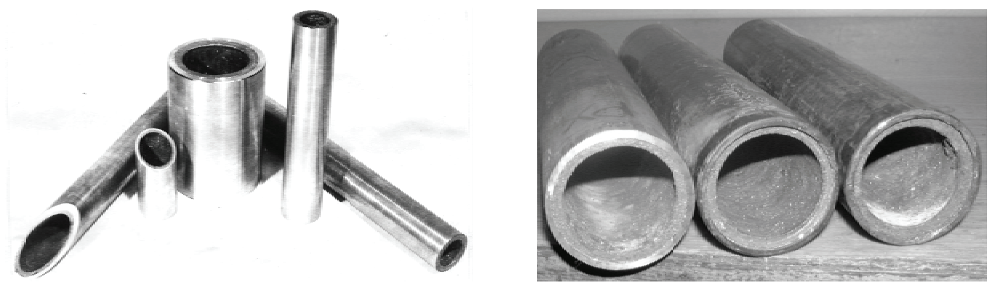
Part name	Analogue materials and SHS-surfaced layer	Increasing operating life
Mixer blade	Cr–Ti–C–Ni–Mo Ст. Г–35	20
Bit	Cr–Ti–C–Fe Sormayt	3–5
Landside	Cr–Ti–C–Fe Sormayt	2,4–2,8
Plow share	Cr–Ti–C–Fe Sormayt	2,7–3,0
Cryogenic plant valve	Cr–Ti–C–Ni–Mo Ст. 4011	15

effectively used to increase the operating life of parts under conditions of intense friction and wear (mixer blades in the production of refractory bricks, bits of machinery for road construction and parts of agricultural machinery for soil cultivation, etc.). SHS-surfaced coatings have high hardness and wear resistance, significantly exceeding the specifications of industrial surfaced coatings.

Tests of different parts with SHS-surfacing under ndustrial conditions showed that their operating life increased from 3–5 to 15–20 times (Table 2).

Cast pipes. In the mid 70-s the creators of SHS-metallurgy showed the possibility of producing double-layer pipes and protective coatings in pipes, using centrifugal SHS-technology (Fig. 12).

In recent years, ISMAN together with Pipe Plant “Stroy-profil” have developed pilot SHS-technology for producing protective coatings for steel pipes, using metallurgical production wastes as raw material. The objects of research were standard, commercially available welded tubes with a diameter of 57, 76 and



a) *b*)

Fig. 12. Cast two-layer pipes (*a*) and steel pipes with corundum coating (*b*) obtained in the centrifugal SHS-set-ups

108 and 220 mm. For each type of tubes there were created optimal conditions for applying protective wear resistant coatings of corundum with a thickness of 5 to 15 mm (Fig. 12 *b*).

Tests of double-layer pipes with an inner layer of cast corundum on the test bed under prolonged abrasion with abrasive slurry showed that their wear resistance is 20–30 times higher than that of steel pipes [52–64].

Conclusion

To implement SHS-metallurgy of refractory inorganic materials taking into account its specific features, the unique equipment, reactors and centrifugal set-ups have been developed and manufactured. They allow for carrying out and studying the processes under the effect of gas pressure (nitrogen and argon, to 12 MPa) and overload (up to 1000 g) .

In the experimental studies, the effect of thermite-type mixtures characteristics (ratio of components in the initial mixture, its density and calorific value, dispersion of reactants, the type of the reducer, etc.), process parameters (pressure and overload), the scale factor (mass and geometry of the reaction volume), etc. on the operational parameters (combustion rate, weight loss during combustion, complete phase separation), chemical and phase composition, structure and properties of combustion products have been found. Based on the results of the research, there was developed a system of control of the composition, structure and properties of refractory inorganic compounds.

In the applied research there have been developed technologies for synthesis of a wide range of cast refractory inorganic materials (ceramics, alloys and composite materials), products and protective coatings.

The proposed approach extends methodological possibilities of synthesis of cast refractory inorganic materials, allows for reducing synthesis time, eliminating complicated high-temperature equipment and minimizing energy consumption.

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