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ABOUT ELECTRICAL CONDUCTIVITY OF FLUXES OF CaF_2–Ca SYSTEM

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New procedure of measuring electrical conductivity of metal-containing slags, used for ESR in chamber-type furnaces, is offered. Values of electrical conductivity of slag of CaF_2–Ca system are determined.

Key words: *electrical conductivity, procedure of measurement, CaF_2–Ca flux, ESR chamber-type furnace*

Mode of melting various metals and alloys in electroslag remelting using metal-containing fluxes (for example CaF_2–Ca) is differed greatly from that in ESR using standard industrial fluxes [1]. The presence of a metallic component in the slag leads to the change in electrical condition of melting that influences in its turn the speed of melting a consumable electrode, process power and formation of the ingot melted. This is, probably, first of all, associated with the change in a flux electrical conductivity. This is one of the main characteristics defining its major technological properties. It influences the thermal condition of a slag pool and stability of the electroslag process. Information available about the electrical conductivity of ESR industrial fluxes is too limited [2–8] and there is no data of electrical conductivity of metal-containing fluxes, in particular of CaF_2–Ca system, in literature. Mastering and optimizing of the ESR technology in a chamber-type furnace using fluxes of CaF_2–Ca system requires determination of this most important physical property of the slag.

Analysis of existing methods of measuring electrical conductivity of slag melts proves impossibility of their use for the determination of this parameter in Ca-containing fluxes. This is due to the fact that the metal calcium is characterized by a high chemical activity, including that with respect to the majority of materials, from which crucibles are manufactured, and by having a high partial pressure of vapours at real temperatures of the electroslag process. In open metallurgical units its evaporation and oxidation are occurred almost instantaneously. Therefore, it was necessary to develop a special procedure of determination of electrical conductivity of metal-containing fluxes on fluoride base during the real ESR process in a chamber-type furnace.

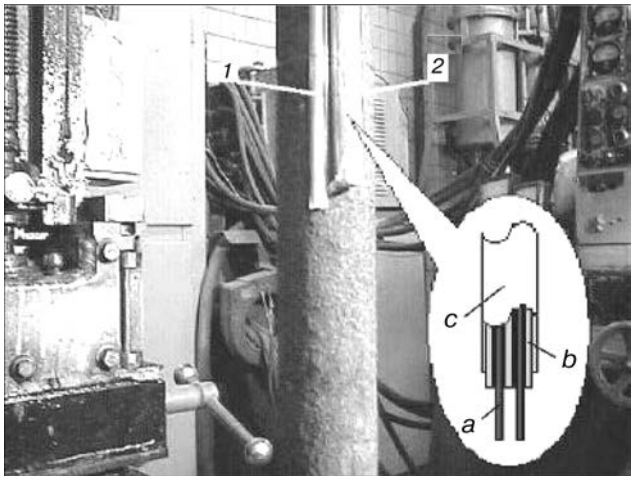

Figure 1. General view of arrangement of sensors on electrode in ESR furnace: *1* — sensor of temperature; *2* — sensor of electrical conductivity

At Donetsk National Technical University a procedure of measuring electrical conductivity has been developed and tested. It is based on a volt-ampere diagram with a two-electrode cell. The use of industrial ESR furnace in this case complicates a calibration of a measuring cell, but provides conditions of measurement which are close maximum to the real conditions. The main difference of the offered procedure from standard procedures consists in the fact that only a difference of potentials on the electrical conductivity sensors is recorded during measurements. And the value of electrical conductivity is calculated directly from the generator volt-ampere characteristic, obtained experimentally.

The electrical conductivity of slags was measured during the process of a chamber-type furnace electroslag remelting [9] of electrodes, made from St.50, of 50 mm diameter, 700 mm length in a water-cooled copper mould of 110 mm diameter in argon atmosphere and in air. Voltage and current of remelting were kept constant, 40 V and 2 kA, respectively, and slag mass was 1400 g. Flux of CaF_2–Ca system was produced by mixing of fluoride and metal calcium in different proportions. Slag pool was set with a «solid start».

To measure the electrical conductivity, a probe (Figure 1) was manufactured of 200 mm length and 15 mm diameter, consisting of metal electrodes (*a*), placed into ceramic straw (*b*), pressed-in into a quartz glass pipe (*c*). Distance between electrodes is similar along the entire length of the probe and equal to 4 mm. Slag temperature was measured by a thermocouple VR-5/20. As a power source of electrical conductivity sensor, the GZ-33 generator of high-frequency sinusoidal signals was used. At 20 kHz frequency of signals the minimum distortions caused by

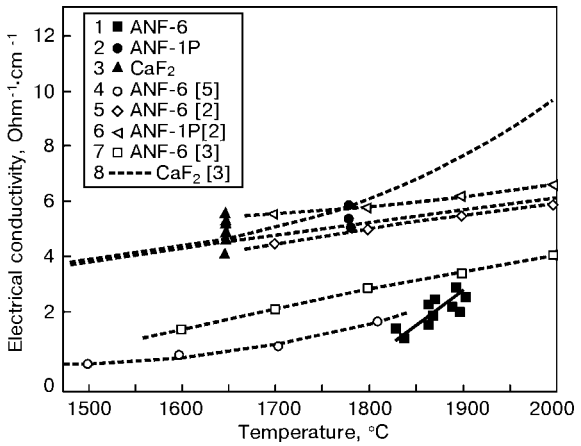

Figure 2. Values of electrical conductivity of industrial fluxes of ESR: *1–3* — obtained by authors; *4–8* — reference data

electrical current of remelting are provided. The indications were recorded by a selective millivoltmeter V6-4.

Probe and thermocouple were fastened on a consumable electrode at 200 mm distance from its lower edge. Auxiliary probe and thermocouple were located at 400 mm distance from the lower edge of electrode. Immersed probes were used during ESR in air to duplicate measurements.

After setting slag pool and formation of an ingot bottom part, the temperature and electrical conductivity were controlled constantly. Appearance of current in a measuring circuit in short-circuiting of electrodes through the slag was considered as a moment of probe contact with a slag.

Graduation of a measuring cell was made in ESR using standard fluxes ANF-1P and ANF-6, whose electrical conductivity is well-known. Constant of the measuring cell was defined as 6.

Results of measurements of the electrical conductivity of reference fluxes ANF-1P and ANF-6 using the offered procedure are comparable with data published earlier in works [2–5, 8]. This gives grounds to use the offered procedure for determination of electrical conductivity of slag of CaF_2–Ca system.

Values of electrical conductivity of known and experimental fluxes are given in Figures 2 and 3. As is seen, the adding of metal calcium to calcium fluoride leads to the increase in the electrical conductivity of

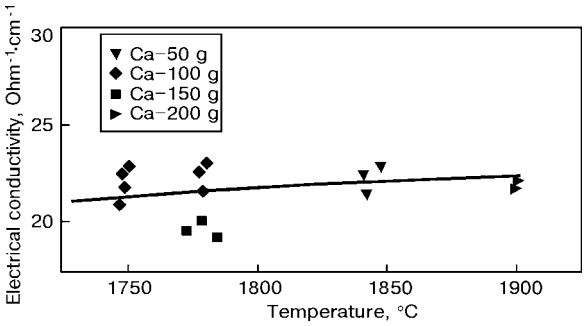

Figure 3. Values of electrical conductivity of Ca-containing fluxes

fluxes, here a significant dependence of electrical conductivity on mass of additions of metal calcium into the slag was not established, This is, probably, due to a limiting value of solubility of metal calcium in its fluoride which does not depend on its initial content in flux, but it is determined by the slag temperature and external pressure [10, 11].

Thus, the procedure offered can be used for measuring electrical conductivity of different slag systems in the process of ESR in the chamber-type furnace.

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APPLICATION OF NON-CONSUMABLE ELECTRODE ELECTROSLAG PROCESS FOR MELTING, REFINING AND TREATMENT OF METALS. Report 2

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Examples of use of the non-consumable electrode electroslag process for processing of a non-compact metal-containing charge to produce different ferroalloys and master alloys, metal refining and cladding are given.

Key words: *non-compact charge, non-consumable electrode, electroslag melting, ferroalloys, master alloys*

Examples of application of the electroslag process with non-consumable electrodes for heating and refining of molten metals were described in report 1 [1]. In the mentioned technologies the temperature of the slag melt is maintained above the temperature of melting of the metal being treated. Therefore, it became logical to develop the electroslag technologies in which the overheated slag is used not only as a means of heating of the preliminary prepared molten metal, but also as a means of melting when the initial metal is in a solid state. In this case the charge, fragmented and bulky in the form of chips, small pieces, shot, pellets, coarse dust, etc. is fed gradually from the top to a slag pool which is set in a water-cooled or lined vessel using non-consumable electrodes (Figure 1). Usually, the parameters of the electroslag process are selected so that the metal could be melted completely during passing through the thickness of the slag melt. During melting in the water-cooled mould the molten metal is solidified gradually in the form of an ingot, while in melting in a skull or lined vessel the metal is accumulated in a molten state and poured out periodically into various moulds. The selection of the melting diagram, including also the lining material, is defined in each definite case separately with allowance for the composition and form of the charge, requirements to the quality of final products, efficiency and economy of the process.

The peculiar feature of the electroslag melting (ESM) methods considered is the fact that the charge, which is fed to the slag melt and having a higher density, is immersed into this melt and melted more often, not already contacting the surrounding atmosphere. This gives an opportunity to decrease significantly the metal losses for smoke and makes it possible to melt many highly-active materials even without an additional protection of the slag mirror. Owing to the developed surface of fragmented charge the intensive heat transfer occurs from the hot slag melt to the material being remelted, thus providing the high melting efficiency. One more advantage of the ESM consists in that the charge being remelted is subjected simultaneously to purification from harmful impurities and a non-metallic component. The feasibility to vary the slag composition and its temperature within the wide ranges, to add deoxidizers and refining additions into a slag during melting can interfere actively in the physical-chemical processes proceeding in the slag–metal system. And this in its turn gives opportunity to reduce the oxides, contained in the charge, and to remove selectively or, vice versa, to add different elements. Therefore, this process is often called in the Russian technical literature as electroslag refining or electroslag melting and refining.

Technologies of utilization and processing of slag-metal wastes of non-ferrous metallurgy, containing from 50 to 80 % of metal in the form of biscuits were the first technologies where the capabilities of the electroslag process with non-consumable electrodes for melting of a non-compact charge were realized successfully [2]. Melting of these wastes in induction and shaft furnaces is difficult, mainly due to a comparatively high content of a non-metallic component, and accompanied by irrevocable losses both in base metal (copper) and also in alloying elements. However, the ESM, owing to its specifics, can separate a slag component from a metal component, moreover, the copper and nickel are removed completely, and the losses in zinc from brass wastes are reduced to minimum at an active use of the reduction processes. The ESM is also more effective, as compared with other types of melting, in processing of purely metallic wastes of copper and copper alloys in the form of chips. ESM of non-ferrous metals is usually performed in a vessel, lined with C-containing materials, the metal is accumulated in a molten state under the slag and poured out periodically or continuously from the melting vessel. In this case, the high quality of the

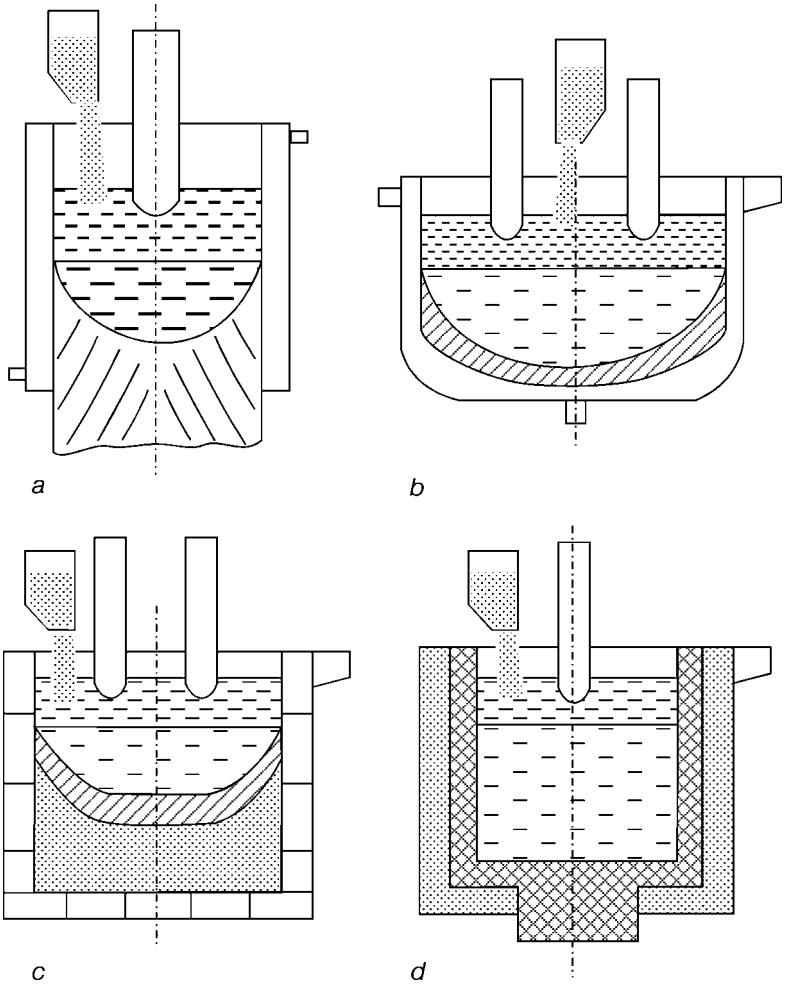

Figure 1. Technological schemes of electroslag melting of a non-compact charge: *a* — in mould; *b* — in skull furnace; *c* — in autoskull furnace; *d* — melting in lined vessel

metal makes it possible not to be limited by melting of a charge billet, but provides an opportunity to produce a final casting of the required shape.

At the different stages of the metallurgical processing and at a subsequent treatment of the metal products, a large amount of various wastes is formed in the form of chips, dust, slime, scrap, fragments and other small non-compact metal-containing materials. These wastes are characterized by a high oxidation and contamination with a non-metallic component, including organic compounds, that limits their application as an efficient charge. The maximum use and return of these low-grade wastes into production, especially those containing valuable alloying elements such as chromium, nickel, cobalt, molybdenum, tungsten, niobium, vanadium and others is rather actual problem.

In this connection, the ESM with non-consumable electrodes can serve one of the most acceptable method of their processing, owing to its above-mentioned features. For example, at such utilization of chips and metal dust of high-temperature alloys the losses in alloying elements do not exceed 2–3 % in spite of the fact that a part of the metal is oxidized. At the same time when these wastes are subjected to processing in electric arc furnaces the losses in alloying elements reach 20 % and more. In practice, the full preserving of main alloying elements such as chromium, molybdenum, tungsten and nickel, is provided during ESM of wastes of high-speed and stainless steels. The low content of foreign impurities in metal of charge billets and castings allows the most part of the wastes to be used in a technological cycle of production without deterioration of the final product quality.

The efficiency of the ESM of a non-compact charge was confirmed in producing ferrotitanium, including 70 % one, from titanium and steel chips [3]. The thing is that the processing of the titanium chips into ferrotitanium in open induction furnaces is difficult due to high reactivity of titanium and a low bulk density of chips, and the melting in different vacuum units requires a careful preparation of the charge and is not economically rational often. At the same time, when the condition of looseness is kept, the chips, having a relatively small thickness and developed surface, are rather convenient material for the ESM. The ESM of ferrotitanium is realized in a water-cooled mould by feeding titanium and steel chips into a slag pool from two hoppers through a weigher (Figure 2). Entering the slag melt, the chips are melted and a metal pool is formed. Its composition and, consequently, the composition of ferrotitanium being melted are determined by a ratio of materials, fed for melting, to physical-chemical processes in the slag–metal system.

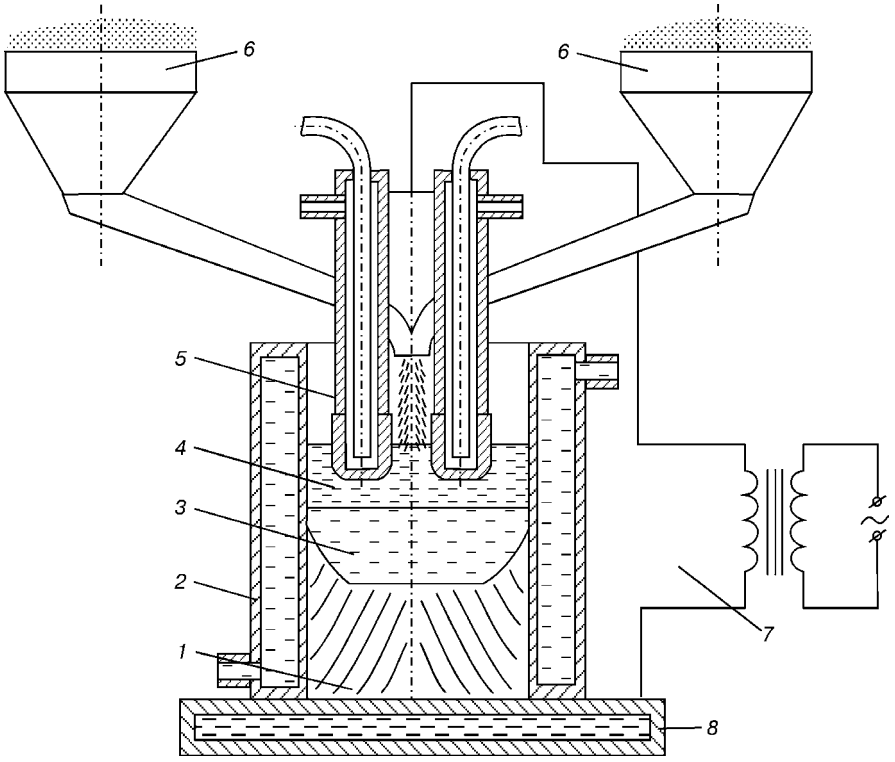

Figure 2. Scheme of process of electroslag melting of ferrotitanium from titanium and steel chips: *1* — solid metal; *2* — mould; *3* — metal pool; *4* — slag pool; *5* — non-consumable electrode; *6* — hopper with steel and titanium chips; *7* — power source; *8* — bottom plate

The quality of this ferrotitanium much exceeds the quality of the similar ferrotitanium produced by an aluminothermic reduction from Ti-containing concentrates.

Similar to ferrotitanium melting, the melting of low-grade wastes of production of ferroalloys, ferrous and non-ferrous metals in a slag pool in various combinations and proportions gives an opportunity to produce special master alloys and alloying elements. There is an experience of ESM of such compounds as FeMnTi, FeTiAl, SiMn, AlTi, FeCrTi, MnSiTi and others. In spite of use of cheap low-grade wastes, the master alloys produced have a low content of harmful impurities, owing to a refining capability of the slag, and can be used successfully in different productions, in particular in melting superalloys.

ESM can be used not only for the utilization and processing of different wastes, which was above-described, but also as an effective method of refining metals. For example, the need arises in use of manganese with a low content of impurities in the production of some precision and special alloys. The purest manganese is produced by electrolysis from sulphate solutions, but it has an increased concentration of sulphur and gases due to specifics of the production. For example, in manganese of Mr-0 grade at content of base element of not less than 99.5 % the sulphur concentration is up to 0.1, oxygen — 0.1, nitrogen — 0.15, and hydrogen — 0.06 %. Therefore, this manganese is purified preliminary by a vacuum annealing or induction melting with a slag treatment.

At the E.O. Paton Electric Welding Institute the technology of electroslag refining of the electrolytic manganese has been developed and realized successfully in industry. This manganese represents a fragmented material in the form of flakes of several millimeters thickness and it was remelted in the layer of a molten slag [4]. Due to strict requirements to the purity of the metal being remelted, in particular to carbon, in this case the non-consumable metal water-cooled electrodes of a special design were used for the electroslag process unlike the previous process where the graphitized electrodes were used, and the melting itself was realized in a water-cooled mould. The application of highly-basic slags of CaF_2–CaO–Al_2O_3 system with adding during melting of refining and deoxidizing components allowed us to decrease the sulphur content in the manganese melted to 0.005–0.01 %. This is 2–3 times lower as compared with content attained in induction refining. Simultaneously, the metal saturation with a gas is also decreased by one order (Figure 3). It is also important that in use of melting by the new technology a 5-fold increase in productivity is achieved and irrevocable losses in manganese are 3–4 times decreased. The use of manganese, passed the electroslag refining, in melting precision alloys, in particular thermal bimetals, could reduce rejections as to chemical composition, stabilize the properties and increase the production profitability. During recent years the technology of ESM of highly-damping Mn–Cu alloys was mastered on the basis of the above-described method of manganese refining [5]. Here, the results obtained in purity, deformability and damping properties of metal in combination with high through yield of efficient metal give grounds to consider the electroslag refining

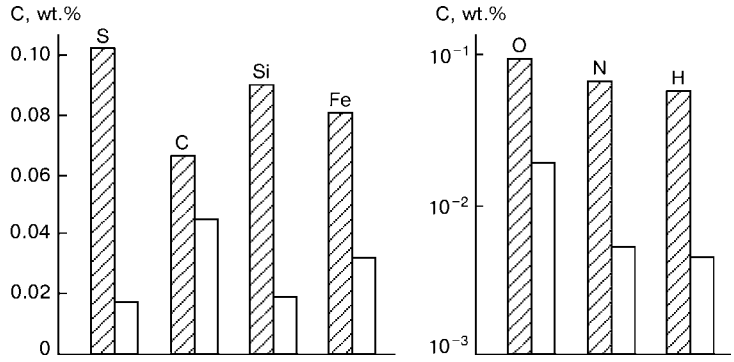

Figure 3. Concentration of impurities in electrolytic manganese: ▨ — initial; ❐ — after electroslag refining

method as promising for melting alloys of Mn–Cu system, used for manufacture of high-quality virboabsorbing devices.

Electroslag cladding is one more successful application of the electroslag process with non-consumable electrodes. Its principle consists in an initial heating and prefusion using electroslag heating of surface of billet being processed and subsequent fusion of a base with a metal supplied through a slag, thus forming a deposited layer. Among the numerous variants of similar cladding, one of the most typical is electroslag restoration of hot extrusion dies [6]. For its realization the die in a vertical position with an engraving at the top is placed into a mould or is confined by water-cooled panels in perimeter and a slag pool is set in the cavity formed over its worn-out surface using non-consumable electrodes. At the expense of heat generated in a slag pool the upper part of the die is melted for a depth required for removing cracks and other existing here defects. Then, the chips of die steel are fed to the overheated slag. The chips are melted and enter the metal pool of the prefused die (Figure 4). Metal is supplied in the amount necessary for restoration of the die height. The ESM provides a reliable fusion of base and deposited metal at the high quality of the deposited metal. The service life of the restored dies is usually higher than that of the new dies made from a forged metal of a conventional production.

Similar to the realization of the die restoration, it is possible to produce the bimetal billets, for example, by cladding of a layer of stainless steel or copper alloy on non-alloyed steel by depositing metal of another composition on the base metal. Variants of cladding can be different, the same as the billets subjected to cladding, namely flat, round at their horizontal and vertical position.

The present article does not cover all the examples of application of the electroslag process with non-con-

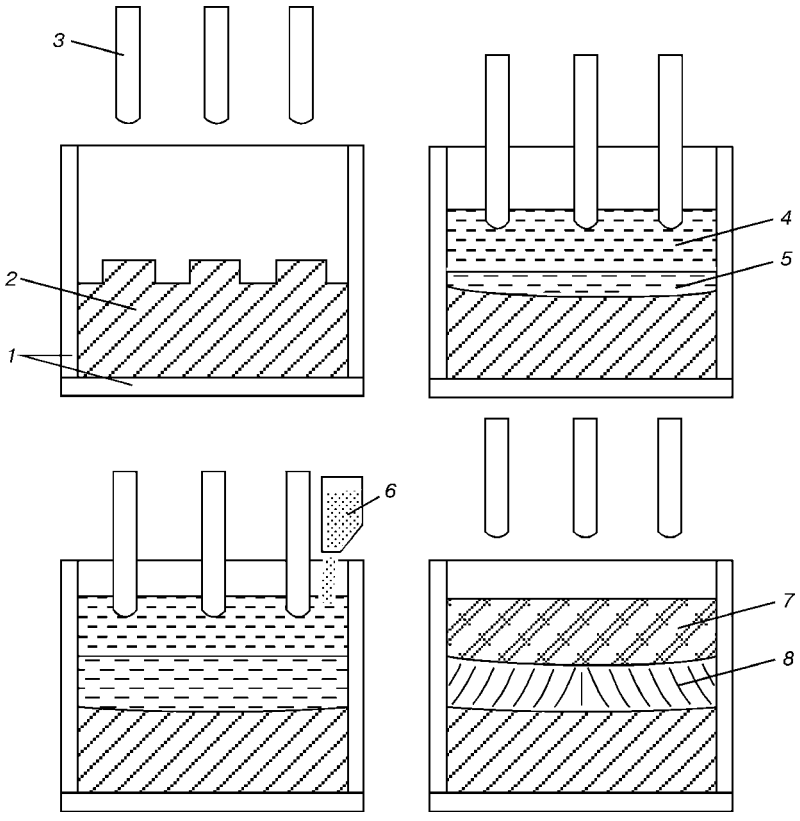

Figure 4. Scheme of electroslag cladding of dies: *1* — water-cooled panels; *2* — worn-out die; *3* — non-consumable electrode; *4* — slag pool; *5* — metal pool; *6* — hopper with chips; *7* — solid slag; *8* — deposited layer

sumable electrodes for casting ingots, refining of metals, melting from a non-compact charge of ferroalloys and master alloys, utilization of wastes of non-ferrous metallurgy and others, but it proves the wide possibilities of the technologies. It is natural that the comprehensive study of specific features and laws typical of this type of the electroslag treatment of metals, such as heat transfer from slag to metal, including in passing of a solid charge through a layer of a molten slag, carbon behaviour in use of graphitized electrodes, effect of slag conditions on metal refining from impurities and also many phenomena occurring in gas–slag–metal system was necessary for their realization and successful application. The investigations in this field are continued, the technologies and equipment are updated, the efficiency is increased and fields of application of the electroslag process with non-consumable electrodes are widened for production of various metal products. Thus, during recent years the electroslag technology is used in larger scales in processing of slags, concentrates, ashes, ores and other mineral charge containing many expensive and scarce metals in different compounds, mainly in the form of oxides. The ESM of the above-mentioned charge in combination with selective reduction of either elements makes it possible to produce, in particular, ferrochromium, ferronickel, ferrotitanium, ferromolybdenum, and also other ferroalloys and master alloys.

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TITANIUM ALLOYS FOR AIRCRAFT INDUSTRY OF UKRAINE

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Distribution of alloying elements and impurities in length and section of 400 mm diameter ingot from titanium alloys of VT6, VT22, T-110 grades, produced by the method of electron beam melting with an intermediate crucible (EBMC) is investigated. Macro- and microstructures of forgings from these alloys are examined. Mechanical properties of alloys after deformation and heat treatment are determined.

Key words: *titanium alloys, chemical composition, distribution of alloying elements, semiproducts, macro- and microstructures, mechanical properties*

As is known, the application of titanium alloys in aircraft construction is stipulated by their high specific strength, corrosion resistance, specifics of thermophysical properties of titanium. In structures of «AN» aeroplanes the mass of parts, made from titanium alloy, is at present 8–9 % of the mass of airframe parts. These are, first of all, braces and load-carrying cylinders of chassis, forks, brackets of control system, load-carrying parts of wing mechanization, cargo tracks of flooring, pipelines, compensators, heat exchangers and other elements.

Efficiency and reliability of titanium alloy parts and sub-assemblies are mainly defined by sensitivity of their mechanical properties to deviations of technological parameters during the process of treatment. It refers not only to the technology of manufacture of different parts and sub-assemblies, but also greatly to the technology of manufacture of semiproducts. Thus, for example, the service life of chassis parts of alloy VT22 at low-cycle tests is varied within the range of 17783–110880 cycles depending on the quality of semiproducts, in particular, on the presence of non-metallic inclusions in the semiproduct metal. Therefore, to have the wider application of titanium alloys in aircraft structures it is necessary not only to create new Ti-based materials with higher service characteristics, but also to improve further the production of titanium semiproducts. It should be noted here, that such defects of the titanium alloy semiproducts as non-homogeneity of chemical composition and structure, inclusions of high and low density, increased content of gaseous impurities (oxygen, nitrogen and hydrogen) are trapped into metal even at the stage of ingot melting and preserved in it during all next processing [1–7]. Moreover, if the inclusions in titanium alloy ingots are formed as a result of contamination of charge materials, then the occurrence of chemical non-homogeneity and increase in concentration of impurities in metal (as compared with their content in charge) are caused by specifics of vacuum-arc melting (VAM), being the main industrial method of production of titanium ingots [4–6].

Effective method of prevention of formation of the above-mentioned defects in cast metal is the electron beam melting with an intermediate crucible (EBMC) [8–11]. This is stipulated by the fact that EBMC is realized at higher, than in VAM, vacuum and there is no noticeable saturation of metal with gaseous impurities during the melting process. In EBMC the molten titanium is held for a long time in a liquid state, that promotes its degassing (hydrogen removal) and the chemical composition levelling. Simultaneously with degassing the molten metal is also subjected to refining from foreign particles of high and low density, which form inclusions in a solidified ingot at the absence of the intermediate crucible. Thus, EBMC makes it possible to increase greatly the quality of titanium ingots [9–12]. However, in this case a problem exists in producing ingots of a preset chemical composition, as the melting in a comparatively high vacuum contributes to a selective evaporation of alloying elements of a high vapour pressure [12, 13]. Therefore, the industrial electron beam units are used mainly for the production of ingots of commercial titanium [6, 8] and ingots of the first remelting from titanium alloys, which are the electrodes for next melting in vacuum-arc furnaces [10, 11].

The task of industrial production of titanium alloy ingots exclusively by the EBMC method is actual and appropriate investigations are carried out both in our country and abroad [12, 14]. Their positive result is very important for the aerospace complex of Ukraine, because our metallurgical industry has no available vacuum-arc furnaces for titanium melting.

Theoretical and experimental works, carried out at the E.O. Paton Electric Welding Institute of the NAS of Ukraine, were directed to producing ingots of high-alloyed alloys of titanium using the EBMC method. These works represented a complex of investigations including the mathematical modelling of evaporation of alloying elements depending on the EBMC parameters and the static analysis of results of experimental meltings. Thus, the quantitative relationship was established between the losses of al-

Table 1. Distribution of alloying elements and impurities in length of ingots of titanium alloys produced by EBMC

Grade of alloy	Sampling place	Content, wt.%								
		Al	Mo	V	Nb	Fe	Cr	Zr	O	N
VT6	Top	6.37	–	4.05	–	0.13	–	–	0.09	0.026
	Middle	6.32	–	4.27	–	0.15	–	–	0.11	0.024
	Bottom	6.45	–	4.18	–	0.18	–	–	0.09	0.026
GOST 19807–91		5.3–6.8	–	3.5–5.3	–	< 0.3	–	–	< 0.2	< 0.05
VT22	Top	5.17	4.30	4.90	–	0.95	1.44	–	0.11	0.012
	Middle	5.23	4.67	5.13	–	1.03	1.32	–	–	–
	Bottom	5.13	4.31	5.10	–	1.01	1.35	–	–	–
GOST 19807–91		4.4–5.9	4.0–5.5	4.0–5.5	–	0.5–1.5	0.5–2.0	< 0.3	< 0.2	< 0.05
T-110 (conditional grade)	Top	5.47	1.03	1.33	5.04	1.62	–	0.36	0.09	0.02
	Middle	5.29	1.10	1.48	5.32	1.60	–	0.35	–	–
	Bottom	5.35	1.16	1.37	4.93	1.58	–	0.32	–	–
Temporary TS		5.0–6.0	1.0–1.5	1.2–2.0	4.5–5.5	1.5–2.0	–	0.3–0.5	< 0.15	< 0.04

loying elements, first of all aluminium, and melting conditions, type of charge, chemical composition of the ingot melted. Technological charts of EBMC of titanium alloys were created on the basis of relationships obtained. Their use in blending and selection of parameters of melting process provides the preset chemical composition of ingot metal. Besides, the effect of different methods of blending on uniformity of distribution of alloying elements in the ingot length was studied. A diagram and special fixture were developed for a continuous feeding of alloying elements into the charge (in a preset amount) in the course of its melting. The use of this procedure of alloying ensures the uniform distribution of alloying elements both along the ingot and also in its section.

As examples, Tables 1 and 2 show the distribution of alloying elements in length and cross-section of 400 mm diameter ingots, made from two grades of titanium alloys, used widely in structures of «AN» aeroplanes, namely VT6 (Ti–6Al–4V) and VT22 (Ti–5Al–5Mo–5V–1Cr–1Fe), as well as of the ingot, made from experimental high-strength multicomponent alloy T-110 (conditional grade), which was developed by the specialists of the PWI and Antonov Scientific and Technical Complex and is the first national titanium alloy. The given data confirm that the EBMC technology provides a sufficiently uniform distribution of alloying elements in volume of titanium alloy ingots. Thus, for example, the difference between the maximum and minimum concentration of aluminium in ingot metal (see Tables 1 and 2) does not exceed 0.25 wt.%. This is 4 times lower than in Ti–6Al–4V alloy ingots produced by EBMC in work [14]. It should be noted that in the near-surface layers of VAM ingots the aluminium concentration is usually by 1.0–1.5 wt.% higher that in their central part [4, 5].

Examination of macro- and microstructures of ingots metal confirmed the high efficiency of EBMC from the point of view of prevention of the formation of inclusions. The inclusions of high and low density were not revealed neither in experimental ingots which were subjected to the comprehensive examination, nor in semiproducts from serial-produced industrial ingots.

There are no long columnar crystallites in the ingots produced by EBMC. Their macrostructure consists of polyhedral β-grains, equiaxial or slightly elongated in the direction of heat removal (Figure 1). The length-to-width ratio of the elongated grains is within 1.5–3.0. There are no defects in the form of cavities and pores in the cast metal.

Ingots of alloys VT6 and VT22, melted by both methods, were subjected to forging for rods of 80 and 70 mm, respectively, and the ingot of alloy T-110 produced by EBMC, was forged for plates of 50 mm thickness. Forging of ingots from alloys VT6 and VT22 was realized according to the industrial technology [15]. The forging of the ingot of T-110 alloy started at temperature 1050 °C (in β-region) and finished at 850 °C (in α + β-region). Ratio of reduction per one heating was 30–40 %. Value of macrostructure grain of forging metal was equal to 4–5 mark of 10-mark scale of macrostructures for titanium alloys [16], while microstructure was equal to the 6th type of scale of microstructures (Figure 2). This satisfies completely the requirements of the aircraft industry, specified to the structure of titanium semiproducts.

Single-type stampings for «AN» aeroplane were manufactured from forgings (Figure 3). Tables 3 and 4 give the mechanical properties of forged pieces and stampings manufactured from them. These data enable us to make conclusion that mechanical properties of semiproducts from alloys VT6 and VT22 correspond to the requirements of standards independently of the method of melting ingots.

It should be noted that alloy T-110 is experimental. It was aimed at the development of high-strength titanium alloy with a good weldability for heavy-loaded parts and sub-assemblies of the aerospace engineering. Therefore, even at the initial stages of in-

Table 2. Distribution of alloying elements in section of ingots of titanium alloys produced by EBMC

Grade of alloy	*Ingot part*	*Sampling place**	*Content, wt.%*						
			Al	*Mo*	*V*	*Nb*	*Fe*	*Cr*	*Zr*
VT6	Top	A	6.28	–	4.19	–	–	–	–
		M	6.39	–	3.87	–	–	–	–
		P	6.35	–	3.96	–	–	–	–
	Middle	A	6.41	–	3.91	–	–	–	–
		M	6.30	–	4.15	–	–	–	–
		P	6.36	–	4.25	–	–	–	–
	Bottom	A	6.53	–	4.03	–	–	–	–
		M	6.72	–	4.14	–	–	–	–
		P	6.48	–	4.18	–	–	–	–
VT22	Top	A	5.21	4.32	4.85	–	0.92	1.40	–
		M	5.12	4.26	4.95	–	0.90	1.42	–
		P	5.25	4.33	5.07	–	1.03	1.50	–
	Middle	A	5.20	4.41	5.13	–	1.00	1.39	–
		M	5.11	4.59	5.21	–	1.05	1.35	–
		P	5.14	4.73	5.10	–	1.04	1.28	–
	Bottom	A	5.16	4.42	5.13	–	1.02	1.32	–
		M	5.08	4.18	5.06	–	1.00	1.39	–
		P	5.16	4.23	5.10	–	1.01	1.33	–
T-110	Top	A	5.48	1.02	1.18	5.32	1.63	–	0.40
		M	5.52	1.01	1.41	4.91	1.61	–	0.35
		P	5.57	1.06	1.40	5.02	1.60	–	0.33
	Middle	A	5.45	1.02	1.50	5.45	1.58	–	0.37
		M	5.30	1.12	1.49	5.26	1.60	–	0.31
		P	5.14	1.14	1.45	5.27	1.61	–	0.37
	Bottom	A	5.54	1.01	1.42	4.98	1.59	–	0.32
		M	5.43	1.12	1.39	4.78	1.69	–	0.34
		P	5.21	1.31	1.33	4.84	1.47	–	0.30

*A — near ingot axis; M — near radius middle; P — in periphery zone (about 10 mm from ingot surface).

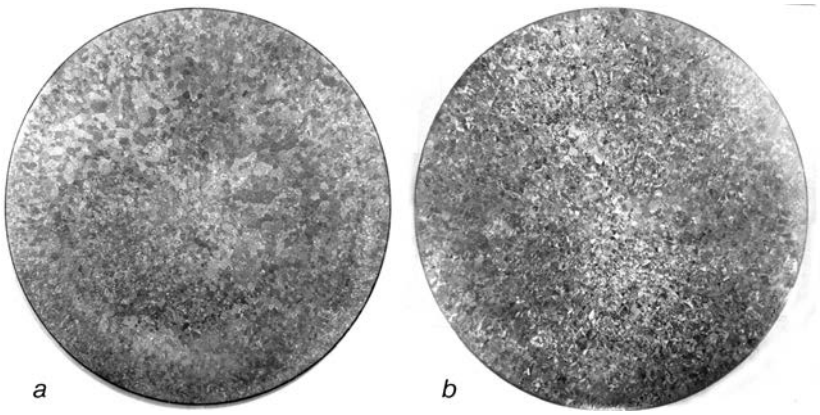

Figure 1. Macrostructure of transverse section of 400 mm diameter ingot from alloys VT22 (*a*) and T-110 (*b*)

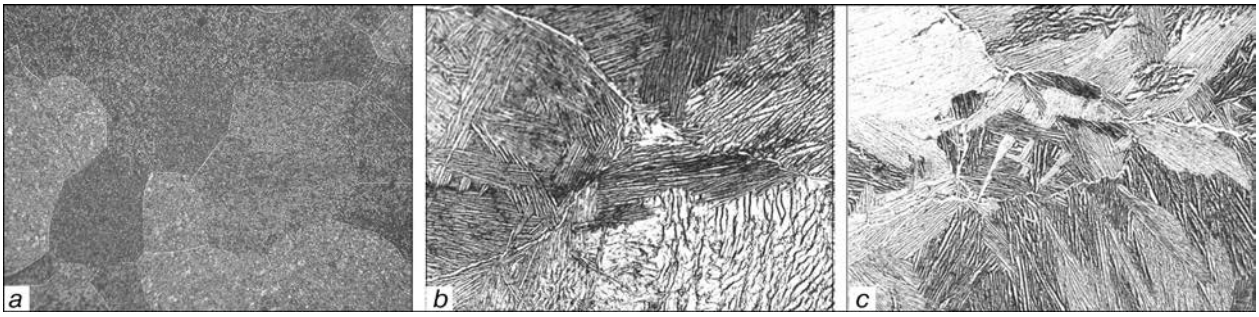

Figure 2. Microstructure of forgings from alloys VT22 (*a*), T-110 (*b*) and VT6 (*c*) (×400)

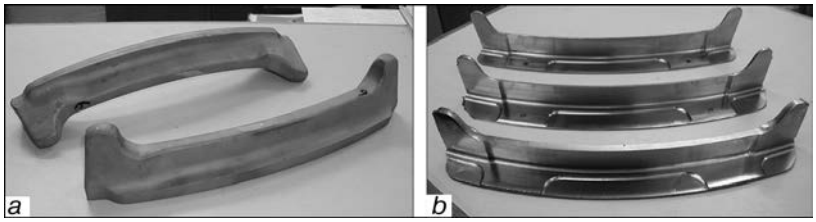

Figure 3. Stampings manufactured from ingots of titanium alloys produced by EBMC before (*a*) and after (*b*) mechanical treatment

Table 3. Mechanical properties of semiproducts from ingots of titanium alloys VT6 and VT22 produced by different methods

Alloy	*Type of semiproduct*	σ_t, *MPa*	$\sigma_{0.2}$, *MPa*	δ, %	ψ, %	*KCV*, J/cm^2
VT6 (VAM)	Forging, 80 mm dia.	972–998 / 985	916–932 / 927	10–15 / 12	34–39 / 36	30–35 / 32
	Stamping	1040–1060 / 1054	960–978 / 965	12–16 / 14	36–41 / 38	33–38 / 34
VT6 (EBMC)	Forging, 80 mm dia.	954–979 / 970	893–918 / 905	14–18 / 15	39–47 / 42	39–43 / 40
	Stamping	1005–1021 / 1012	958–964 / 961	14–17 / 15	44–49 / 46	41–48 / 45
VT22 (VAM)	Forging, 70 mm dia.	1150–1180 / 1163	1070–1100 / 1085	12–17 / 14	46–49 / 47	23–28 / 24
	Stamping	1176–1190 / 1180	1120–1139 / 1128	15–18 / 17	42–50 / 45	27–32 / 29
VT22 (EBMC)	Forging, 70 mm dia.	1178–1216 / 1189	1100–1124 / 1106	13–16 / 14	43–47 / 44	22–30 / 25
	Stamping	1175–1220 / 1195	1096–1118 / 1110	16–19 / 17	45–49 / 47	24–31 / 27

Note. Above the line the minimum and maximum values are given, under the line the mean values are given.

Table 4. Mechanical properties of semiproducts of titanium alloy T-110

Alloy	*Type of semiproduct*	σ_t, *MPa*	$\sigma_{0.2}$, *MPa*	δ, %	ψ, %	*KCV*, J/cm^2
T-110 (EBMC)	Forging	1071–1099 / 1085	1034–1047 / 1038	16–19 / 17	38–41 / 39	39–42 / 40
	Stamping	1165–1180 / 1173	1124–1145 / 1130	20–23 / 21	49–53 / 51	28–31 / 29

Note. 1. Above the line the minimum and maximum values are given, under the line the mean values are given. 2. Heat treatment was made using the following conditions: for forging — heating 780 °C ± 10 °C, 2 h soaking, air cooling; for stamping — heating 870 °C ± ± 10 °C, 0.5 h soaking, furnace cooling to 800 °C ± 10 °C, soaking 15 min, furnace cooling to 750 °C, 1 h soaking, air cooling, heating to 380 °C, 8 h soaking, heating to 570 °C, 2 h soaking, air cooling.

vestigation the ingots from this alloy were melted exclusively by using EBMC, as the most challenging method which is characterized by the absence of defects in cast metal. It is assumed that it will replace VT22 alloy in future. Comparison of mechanical characteristics of these alloys makes it possible to state that at actually equal strength the ductility of the alloy T-110 is higher.

Thus, the investigations showed that the properties of semiproducts, manufactured from ingots produced by the EBMC technology developed, meet all the requirements specified by the aircraft industry to the quality of the titanium alloys.

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FORMATION OF QUASI-CRYSTALLINE STRUCTURES IN ANNEALING OF MICROLAYER COATINGS (Ti, Cr)–Si

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Structural changes in microlayer coatings (Ti, Cr)–Si in the process of isothermal annealing at temperatures 900, 1000 and 1100 °C are studied. Microlayer structure was produced by electron beam evaporation from two ingots (Ti–Cr and Si) and condensation in vacuum of non-mixing vapour flows. It is shown that in diffusion interaction between the layers the microlayer structure is destroyed and the structure on the base of icosahedral phase (or approximate to it) is formed.

Key words: *electron beam evaporation and deposition, microlayer coatings, Ti–Cr–Si system, quasi-crystalline structure*

Alloys with a quasi-crystalline structure (or approximate, close to it by a local atomic packing) represent interest from the point of view of creation of materials on their base, having unique combination of properties, such as low heat conductivity and electrical conductivity, high hardness and wear resistance, low coefficient of friction, high corrosion and oxidation resistance and others [1]. The use of the materials with a complex crystalline structure (CCS) as coatings can widen significantly the functional capabilities of products made from traditional materials. Many works are devoted to the study of methods of producing films and coatings with CCS on the base of different metal systems [2, 3].

Thus, to form CCS on the base of aluminium systems an application of plasma methods of coating deposition found the wide spreading [3]. It is shown in our work [4] that these coatings can be formed for a single technological cycle directly from the vapour phase in electron beam evaporation and condensation of ingot of an optimum composition in vacuum. It was established that the microstructure of coatings, formed here, is characterized by a high degree of perfection that promotes the increase in their service properties (for example, microhardness of coatings is increased from 7.5 up to 9.5 GPa in transition from plasma to electron beam method of the coating formation).

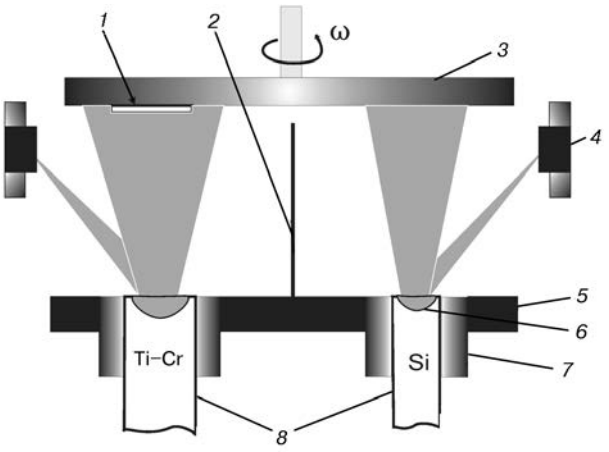

Figure 1. Scheme of deposition of microlayer coating (Ti, Cr)–Si: *1* — substrate; *2* — separating shield; *3* — rotating holder of substrates; *4* — electron beam gun; *5* — water-cooled plate; *6* — pool; *7* — water-cooled crucible; *8* — ingots Ti–Cr and Si

The another method of CCS formation can be based on annealing of nano- or microlayer condensates, produced using the technology of a physical deposition of vapour and consisted of separate components of the alloy in a preset ratios. For the first time the feasibility of formation of a metastable quasi-crystalline phase as a result of proceeding of solid-phase reactions during annealing of microlayer condensates Al–Mn, Al–Mn–Si was shown in [5]. Similarly, CCS in other systems were also produced, for example metastable quasi-crystalline phases in Al–Cr [6] and Al–Co [7], and also equilibrium icosahedral phase in system Al–Cu–Fe [8]. The similar technology was also successfully used earlier for producing intermetallics in system Ti–Al [9, 10].

Practical application of coatings with CCS on aluminium base is limited by their comparatively low temperature of melting (1100 °C). From this point of view the alloys on titanium base Ti–TM–Si (TM = = V, Cr, Mn, Fe, Co, Ni), in which one of the most wide classes of quasi-crystalline structures was revealed, are more promising. The icosahedral phase in most these alloys is metastable and a fast cooling from the melt is used, as a rule, for its formation. The examination of the quasi-crystalline phase in system $Ti_{68-x}Cr_{32}Si_x$ ($6 < x < 18$) [11] using differential calorimetry showed that during heating it does not undergo an abrupt transition into a crystalline phase. This makes it possible to assume that the icosahedral phase in the above-mentioned system can be stable [11]. Moreover, CCS was revealed in system $Ti_{75-x}Cr_{25}Si_x$ ($10 < x < 20$) representing 1/1 rational approximation (bcc lattice, a = 1.314 nm), in which a local atomic packing is close to the atomic packing in the icosahedral structure [12].

In the present work the feasibility of formation of coatings with CCS on the base of Ti–Cr–Si system in the process of annealing microlayer condensates produced by electron beam deposition in vacuum and consisting of alternating thin layers of Ti–Cr and Si is studied.

Method of manufacture of samples. Microlayer coatings (with up to 0.1 μm layer thickness) were produced by evaporation of Ti–Cr (70 mm diameter) and Si (50 mm diameter) ingots from two water-cooled crucibles in vacuum and subsequent condensation of vapour on a rotating substrate. Ingots of Ti–Cr alloy were manufactured by the induction melting of titanium (99.9 % purity) and chromium (99.99 % purity).

Scheme of deposition of a microlayer condensate is presented in Figure 1. A separating shield mounted between crucibles makes it possible to avoid mixing of vapour flows from two sources. By the substrate rotating around vertical axis it is possible to provide the formation of structure of the coating with alternating layers of Ti–Cr and Si. The rate of evaporation of ingots was selected so that the content of Ti–Cr and Si corresponded to the stoichiometric composition $Ti_{60}Cr_{25}Si_{15}$, in which the CCS formation was observed earlier. The thickness of microlayers was varied by changing the rate of the substrate rotation. Thus, the samples obtained were subjected to annealing in vacuum furnaces at 10^{-6} Pa pressure in chamber during the preset period of time. Structure of coatings was examined using scanning microscope CamScan equipped with an energy-dispersed system of a local analysis Energy 200. Phase composition was determined with the help of X-ray diffractometry of surface of the coatings in a general-purpose diffractometer DRON-4 in a filtered radiation of iron anode.

Results and their discussion. Figure 2 presents the typical microstructures of a transverse section of a microlayer coating (Ti, Cr)–Si in initial state and after annealing at temperatures 800, 1000 and 1100 °C. It is seen that in initial state the microstructure of coating consists of alternating layers (light and dark) of Ti–Cr and Si. Two systems of lines corresponding to phases α-Ti and ε-"$TiCr_2$" are observed on diffractograms taken from these coatings (Figure 3, *a*). It can be assumed on this basis that during the process of deposition of a vapour flow formed by Ti–Cr ingot, the microlayers are formed, consisting of two phases: intermetallic ε-"$TiCr_2$" and solid solution on titanium base. The absence of diffraction lines from silicon can be stipulated by a high dispersity of its grained structure.

Analysis of coating microstructure after annealing at temperature 800 °C (Figure 2, *b*) shows a significant diffusion stirring of components forming multilayer structure. The structure formed is characterized by the presence of two components: dark phase on titanium base and light phase on the base of ε-"$TiCr_2$" (Table). The appearance of peaks corresponding to solid solution ε-"$Ti(Cr, Si)_2$": $Ti_{39-42}Cr_{60-x}Si_x$, x = 9–10 is observed on a diffraction pattern (Figure 3, *b*). The other peaks can be identified as Bragg's reflexes from solid solution $(Ti, Cr)_5Si_3$: $Ti_{62-x}Cr_xSi_{38}$ ($0 < x < 44$) on the Ti_5Si_3 base. Structure of such solution has a hexagonal singony with lattice parameters: a = 0.519 nm, c = 0.489 nm that corresponds to

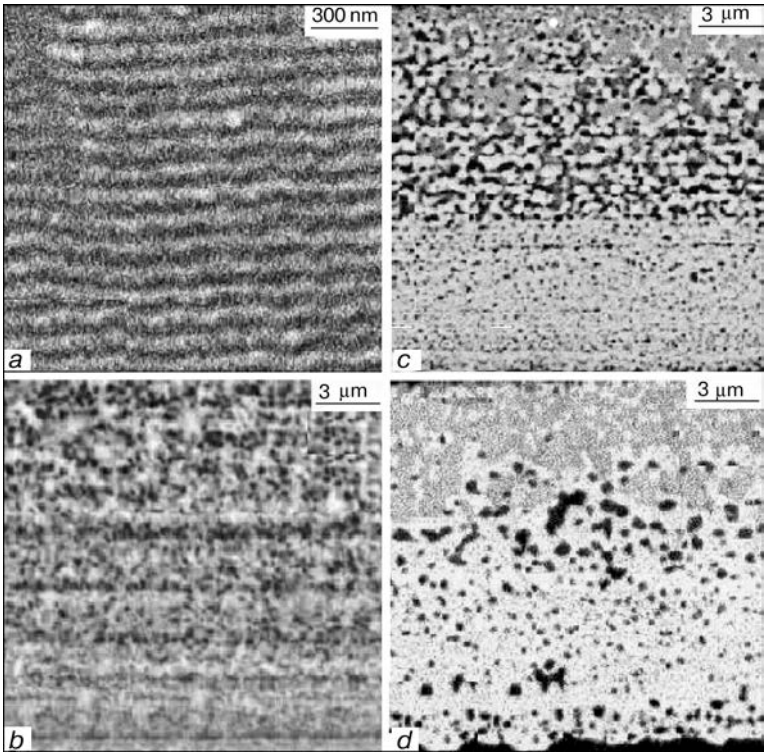

Figure 2. Microstructure of transverse section of coating Ti–Cr–Si: *a* — after deposition; *b, c, d* — after annealing at 800 °C for 16 h, 1000 °C for 2 h and 1100 °C for 2 h, respectively

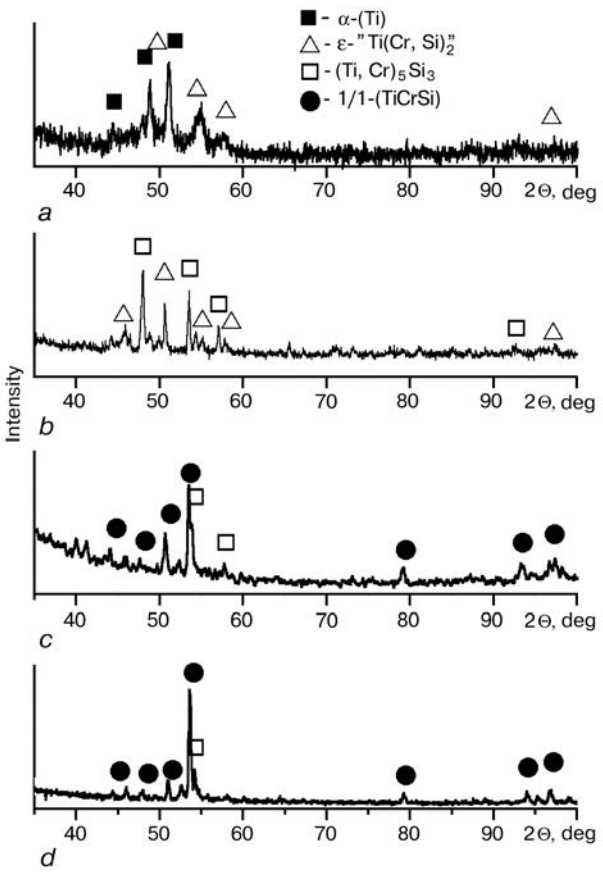

Figure 3. Roentgenograms taken from the surface of Ti–Cr–Si coating: *a* — after deposition; *b, c, d* — after annealing at 800 °C for 16 h, 1000 °C for 2 h and 1100 °C for 2 h, respectively (Co-K_α radiation)

x = 20–25 [13]. Peaks of titanium, not included into solid solutions, are almost not seen.

The further annealing at temperature 1000 °C (Figure 2, *c*) leads to coarsening grains of phases. In this case a new phase (of grey colour) is appeared, whose composition is close to optimum composition of an approximate phase $Ti_{75-x}Cr_{25}Si_x$ ($10 < x < 20$). Compositions of dark and light phases are changed negligibly. X-ray diffraction pattern of coatings after annealing at 1000 °C (Figure 3, *c*) proves about formation of a complex structure, close to the structure of an approximant, described in [12]. Several peaks are also seen which can be identified as peaks $(Ti, Cr)_5Si_3$. The observed dominating of peaks from phase similar to the approximate structure is explained by the fact

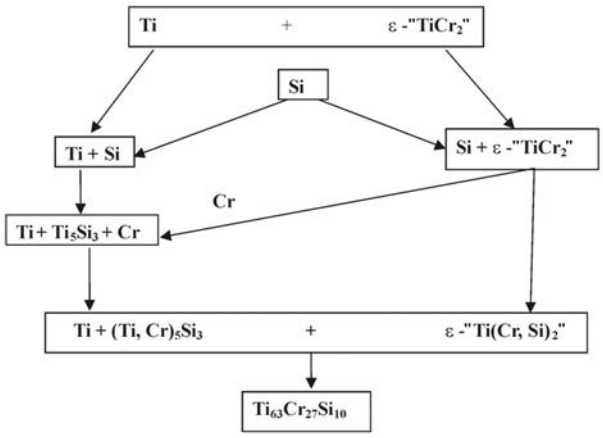

Figure 4. Scheme of proceeding solid-phase reactions in the process of annealing of microlayer coatings

Characteristic of microstructures of coatings forming in the process of annealing

Annealing temperature, °C	Compositions of phases		
	(Ti) + (Ti, Cr)$_5$Si$_3$ (dark)	ε-"Ti(Cr, Si)$_2$" (light)	Approximant (grey)
800	$Ti_{77.5}Cr_{15.5}Si_{7.0}$	$Ti_{49.3}Cr_{42.4}Si_{8.3}$	–
1000	$Ti_{76.7}Cr_{8.3}Si_{15.0}$	$Ti_{41.3}Cr_{49.8}Si_{8.8}$	$Ti_{63.7}Cr_{27.0}Si_{9.3}$
1100	$Ti_{64.8}Cr_{13.9}Si_{21.3}$	$Ti_{37.7}Cr_{53.5}Si_{8.8}$	$Ti_{61.9}Cr_{27.6}Si_{10.5}$

that this phase is located mainly near the sample surface.

Annealing at temperature 1100 °C (Figure 2, *d*) leads to the further increase in a volume fraction of phase of a grey colour, here the composition of this phase is not almost changed. Compositions of dark and light phases are changed negligibly. Roentgenogram of this coating (Figure 3, *d*) has peaks on the same angles in principle as after the annealing at 1000 °C. Moreover, the ratio of intensities of peaks is changed. The results obtained allow us to assume that solid-phase reactions take place in the annealing process, which can be presented in the form of scheme shown in Figure 4.

Annealing of the multilayer coating activates the diffusion mobility of atoms that leads to the appearance of equilibrium and metastable phases. Thus, at the first stage of heat treatment at 800 °C the formation of silicide Ti_5Si_3, Ti-based solid solution, and also silicon solid solution in intermetallic ε-"$TiCr_2$" (ε-"Ti(Cr, Si)$_2$") is possible. In accordance with a phase state diagram [13], at chromium adding to compound ε-"$TiCr_2$" to preserve its stability, a part of chromium should be removed from its composition. The released chromium can be used for formation of chromium silicide Cr_5Si_3, however, the absence of this phase on a diffraction pattern of lines makes it possible to assume that penetration of atoms of free chromium into titanium silicide Ti_5Si_3 is most probable. This leads to the formation of solid solution (Ti, Cr)$_5$Si$_3$ of an assumed composition $Ti_{42-37}Cr_{20-25}Si_{38}$. It should be noted that increase in annealing temperature causes a partial decomposition of silicide. Its volume fraction is decreased so much that diffraction peaks corresponding to this phase are disappeared. Annealing at 1100 °C leads to a phase recrystallization with the formation of the new phase with an approximate crystalline structure, similar to that which was observed in annealing of alloy $Ti_{60}Cr_{25}Si_{15}$ [12]. On photos of microstructures this structural constituent has a grey colour, its composition is $Ti_{63}Cr_{27}Si_{10}$. With increase in annealing temperature up to 1100 °C the volume fraction of given phase is increased. The compositions of remained phases are changed negligibly in this case.

Thus, the presented investigations make it possible to conclude that a quasi-crystalline structure (approximate) can be produced by annealing at temperatures above 800 °C of coatings with a microlayer structure consisting of alternating thin layers Ti–Cr and Si. Phase with a quasi-crystalline structure is formed as a result of proceeding of solid-phase reactions which are accompanied by the formation of intermediate metastable phases on the base of intermetallic ε-"$TiCr_2$" and silicide Ti_5Si_3.

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INTERACTION OF STEEL FIBERS WITH ALUMINIUM MATRIX IN PRODUCING COMPOSITE MATERIAL USING METHOD OF ELECTRON BEAM DISPERSION OF MELT

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Process of producing the composite material, aluminium matrix/steel fibers, using the method of melt dispersion from an intermediate crucible during the electron beam remelting is described. Effect of technological conditions on nature of interaction of fiber with matrix is investigated. Macro- and microstructures of samples are examined and the composition of forming phases is determined.

Key words: *composite material, melt dispersion, aluminium matrix, steel fibers, intermetallic compound*

Superhigh physical and mechanical properties of fibrous composite structural materials on the base of metallic fibers and matrix can be attained theoretically. It is not managed to realize this in practice due to technological difficulties connected with providing necessary conditions of interaction of metals at the fiber–matrix interface. The interface should satisfy two conditions: uniformity of mechanical properties and absence of a wide transition zone in chemical composition. The fulfillment of the first condition means the effective redistribution of load in deformation and is provided by the absence of surface defects and oxidation in fibers. To fulfill the second condition it is necessary to suppress the diffusion processes at the fiber–matrix interface [1].

The required technological conditions are provided by the new method of producing the composite material, namely an electron beam dispersion of melt (EBDM) developed at the E.O. Paton Electric Welding Institute [2, 3]. The process is proceeding under the conditions of high vacuum ($P < 10^{-1}$ Pa) that prevents the oxidation of the surface of metallic fibers during their heating. This technology makes it possible to provide a minimum thickness of zone of diffusion interaction of fiber and matrix materials. The method can be used for producing a fibrous composite material on the base of matrix from light metal alloys reinforced with strengthening fibers. The aim of the present investigation was to study the matrix structure and nature of its interaction with a fiber.

The following materials were used: for matrix — aluminium alloy of AMg1 type (composition, wt.%: Al — base; Cu — 0.17; Fe — 0.14; Mg — 0.52; Mn — 0.02; Si — 0.1), for fibers — steel wire of 0.18 mm diameter (composition, wt.%: Fe — 98.5; Al — 0.27; Cr — 0.12; Mn — 0.75)*. This pair of alloys without noticeable alloying was selected from the point of view of purity of experiment to observe more clearly the aluminium interaction with iron. In addition, the authors were oriented on estimated data for aluminium matrix and steel fibers when selecting the technological parameters [4].

Wire was tightened on a steelwork, which was fixed on 20 mm thick plate with holes for thermocouples. Matrix was made according to a technological scheme [3] both with preheating of substrate (plate) to 400 and 500 K and without preheating. Fibers were not preheated. The efficiency of melt dispersion was 150 kg/h. The temperature of the substrate was recorded during all the time of producing composite using tungsten-rhenium thermocouples connected to a recorder (Figure 1). After deposition of 30–40 mm thick matrix the process was interrupted and a workpiece was soaked under vacuum during 1 h.

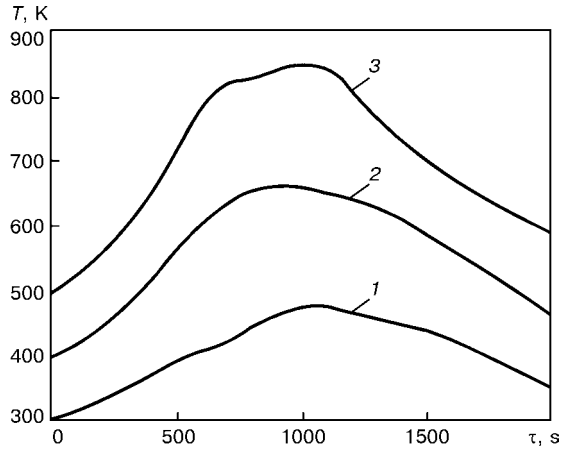

Figure 1. Temperature cycles of substrate preheated up to 300 (*1*), 400 (*2*) and 500 (*3*) K

*Data of spectral analysis.

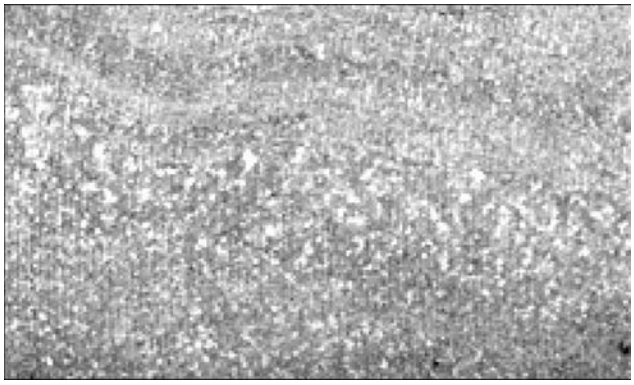

Figure 2. Macrostructure of transverse section of fibrous composite (×5)

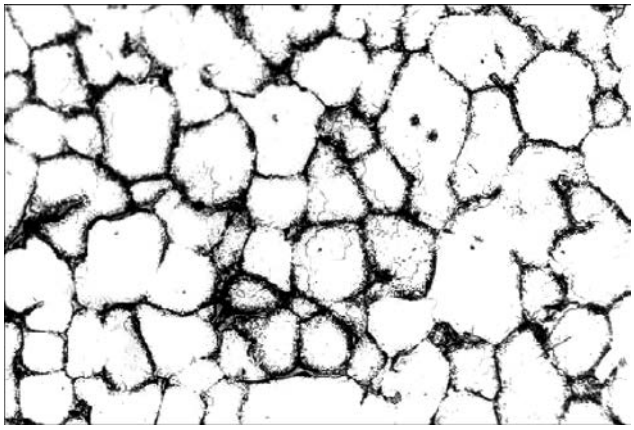

Figure 3. Microstructure of aluminium matrix (×100)

Samples were manufactured from the composite produced for macro- and microstructures examination. Macrostructure of a transverse section of composite on the base of aluminium matrix is banded with wavy boundaries which correspond to the crystallization layers (Figure 2). Defects in the form of pores and discontinuities which could be caused by a non-fusion of successively crystallizing layers were not observed. The layers of crystallization, observed on a macrosection of sample transverse section, are differed by a grain size and degree of etching. In parallel with 80–130 μm fine-grain regions, there are regions where the grain size reaches 400 μm.

Metallographic examinations of the microstructure of samples on the base of aluminium matrix were made in optical microscope «Neophot 32» after etching in 0.5 % water solution of hydrofluoric acid. Microstructure of sample of aluminium composite material is heterophase (Figure 3), that is due to a low solubility of some elements (including foreign impurities of silicon and iron) in aluminium. Grain boundaries of α-solid solution are wide, their more intensive etching is associated with intracrystalline liquation, there are precipitations of intermetallic phases at the grain boundaries.

The adhesion of matrix with fiber is not observed on samples of the composite produced without preheating of the substrate, a pore is formed around the fiber (Figure 4, *a*). When substrate is preheated the chemical interaction of metals occurs at the matrix–fiber interface and an interlayer is formed (Figure 4, *b, c*). The interlayer is homogeneous, grey (on non-etched sections). The interlayer thickness is varied within 10–50 μm depending on temperature of the substrate preheating.

Chemical composition of the interlayer was examined by the X-ray method in Camebax microanalyzer. 46 measurements (2 μm pitch) were made for each sample, starting from aluminium matrix through the

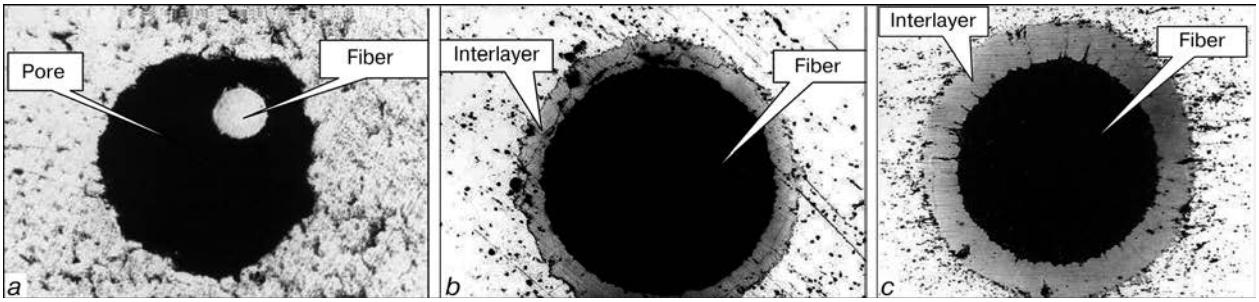

Figure 4. Nature of fiber interaction with matrix at substrate preheating to temperature 300 (*a*) (×50), 400 (*b*) (×250) and 500 (*c*) K (×250)

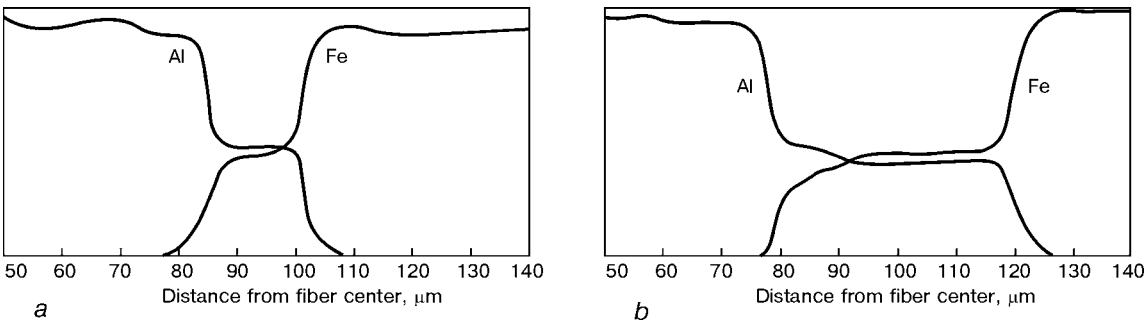

Figure 5. Nature of distribution of aluminium and iron in fiber region at substrate preheating up to temperature 400 (*a*) and 500 (*b*) K

layer into the region of a steel fiber. Graphs of distribution of aluminium, iron (Figure 5), and also carbon, silicon and magnesium were plotted. Nature of change in aluminium content is as follows: from maximum in matrix with a decrease to 50–60 % in interlayer and reduction to zero in steel wire. The iron content is changed from minimum in aluminium matrix to 40–50 % in interlayer and reaches maximum in wire. The carbon content in the interlayer is not changed that proves the absence of carbides (i.e. interlayer is not a carbide compound). Silicon and magnesium content is not also increased.

Consequently, the interlayer observed at the aluminium matrix–steel fiber interface is intermetallic Fe_xAl_y. Coming from the average content of aluminium ~ 73 at.% and iron ~ 25.8 at.%, it can be stated that this is $FeAl_3$.

CONCLUSIONS

1. Samples of composite material on the base of aluminium matrix and steel fibres were produced using the EBDM method.

2. There is no adhesion bond between fibres and matrix without the substrate preheating.

3. At substrate preheating up to 400 K $FeAl_3$ interlayer is formed between the fiber and matrix, whose thickness is growing with increase in heating temperature.

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*In this article there are mistakes in Figure 3 (p. 20) and in text (p. 20). It should be read «... thickness of intermetallic layer for aluminium and titanium matrices is 23–38 and 15–30 μm» instead of 0.23–0.38 and 0.15–0.30 μm, respectively.

THERMAL FIELD OF A SINGLE CRYSTAL AT A COMBINED HEATING*

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The thermal fields of tungsten single crystals were studied in the conditions of plasma-induction zone melting (PIZM) using a mathematical model. Relationships of change in pool depth and curvature of solid–liquid phase interface were obtained indicating the efficiency of effect of a combined heating on the formation of structure of single crystals. It is shown that it is necessary to use a two-section inductor for thermal field control in growing single crystals of refractory metals.

Key words: *tungsten, single crystal, plasma-induction zone melting, thermal field, mathematical model*

The method of PIZM [1] is based on use of two independent heat sources that makes it possible to provide the more flexible control of the temperature field of crystal and metal pool. Unlike the electron beam zonal and plasma-arc droplet melting, there appears a feasibility in PIZM to change the curvature of the phase interface and direction of the crystals growth. The phase interface should become plane in a perfect case, and in each definite case it can be concave, convex or have the intricate shape.

Investigation of the thermal field of the single crystal in the PIZM process is necessary to predict the quality of crystals being grown, and it is possible also to define optimum ratios of power parameters of heat sources by study of gradients of temperatures, shape and curvature of interface of solid and liquid phases.

The pattern of thermal fields was studied on the example of tungsten single crystals using a mathematical model which is described in detail in [2]. Perfection of heat sources, initial and boundary conditions of heat conductivity equation are taken the same as in the above-mentioned work.

During the plasma-induction growing of single crystals the maximum current value was limited to 2 kA. It is not rational to use the plasma sources of current above 2 kA, because the electrodynamic effect of current on the pool and its local overheating will lead to the intensification of molten metal stirring and its spattering.

Mathematical model allows us to preset any values of current (power) of a plasma arc, but the practical value of the results obtained for the range of more than 2 kA will be reduced, as the electrodynamic effect of current on metal pool and convective heat and mass transfer in its volume are not taken into account in the model.

Geometrical sizes of an induction heaters can be changed depending on the single crystal diameter, but the power density or specific surface power should be kept constant.

The important moment in the investigations of the thermal field is the selection of linear sizes of a single crystal. By varying the single crystal diameter its length l should be more than critical. The longer the model single crystal the closer the temperature values in the zone adjacent to the phase interface which are observed in the stationary conditions. However, the increase in power of heat source at different methods of growing single crystal (electron beam zonal melting, plasma-arc zonal melting and PIZM) does not influence greatly the temperature at the single crystal length exceeding its three diameters [2]. Taking into account this circumstance, and also the expenses for experiment connected with a duration of the process, limited capacity of the computer, it is rational to select the single crystal length equal to its 3–5 diameters.

Before studying the thermal fields, forming in the single crystal under the action of two heat sources and changing depending on geometric parameters of induction heating source, let us consider the effect of specific power of the induction heating W_{ind} on the distribution of temperatures in the single crystal (Figure 1).

It is seen from the Figure that the highest gradient of temperatures is preserved in the upper part of the single crystal. The gradient is higher when the specific power of the induction preheating is lower. At the single crystal length equal to approximately its diameter, the value of the temperature gradient is tended to zero. With 4 times increase in power of the induction preheating the temperature gradient is 6–7 times decreased. Therefore, it is necessary to increase the specific power of the induction preheating when the problems of temperature gradient minimizing in the single crystal body are solved.

*The work was fulfilled under the finance support of Science and Technology Center in Ukraine.

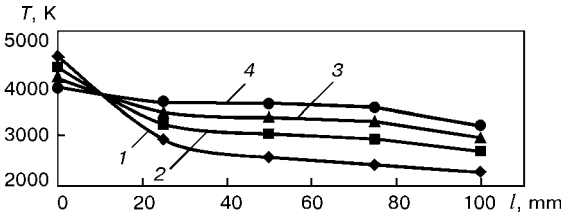

Figure 1. Distribution of temperature along the axis of tungsten single crystal of 30 mm diameter at different values of specific power of the inductor: *1* — 100; *2* — 200; *3* — 300; *4* — 400 W/cm^2 (pool height at crystal lateral surface is 2.33 mm)

The temperature gradient in the middle part of the single crystal is minimum and does not depend on specific power of preheating. Moreover, the absolute value of temperatures is rising with increase in the preheating power.

The temperature gradient is higher in the lower part of the single crystal, where a priming crystal is placed, than that in the middle part, not depending on a specific preheating power. This is explained by the so-called edge effect, which is associated with an effect of inductor and change in condition of heat removal at the single crystal edge. With 4 times increase in a specific power of preheating the temperature gradient is 3 times decreased. The change in temperature gradient depending on the power of the induction preheating, at other conditions being equal, in the lower part of the single crystal is 2 times lower as compared with the upper its part where the heat is entered from two sources.

When the inductor height h_{ind} and length of the single crystal l are equal, it is rational to estimate the change in power of the plasma heating source W_{pl} depending on the specific power of the induction heat source. Figure 2 reflects this relationship. It follows from the Figure that the power of the plasma-arc source is changed in inverse proportion to the induction source power. With 4 times increase in power of the induction heating the power of the plasma-arc heating is 3 times decreased. The total power is not remained constant, it is rising. This influences negatively on technical-economical characteristics of the process, but makes it possible to control the curvature of interface of solid and liquid phases and, thus, to affect the formation of the single crystal structure.

The effect of inductor height at a fixed power of the plasma-arc heating source on the change of depth H of metal pool is shown in Figure 3. As is seen, the higher the specific power of the induction preheating,

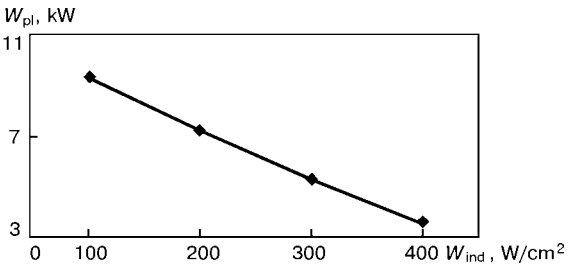

Figure 2. Dependence of plasma source power on specific power of induction source in growing single crystal of 30 mm diameter

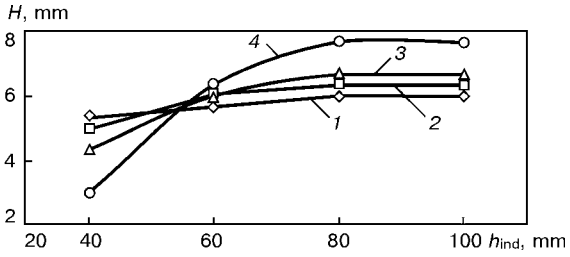

Figure 3. Dependence of pool depth in crystal axis on inductor height at different values of specific power of inductor: *1* — 100; *2* — 200; *3* — 300; *4* — 400 W/cm^2 (tungsten single crystal of 30 mm diameter and 100 mm height)

the more sensitive the pool depth to the change in the inductor height. Thus, with 4 times increase in the specific power the pool depth in crystal axis is 7.5–8 times increased. The pool depth will remain constant in that case when the inductor height 2.7 times exceeds it. The further increase in height of inductor does not influence the temperature field in the region of the interface of liquid and solid phases and does not influence the curvature of its surface and, consequently, the formation of a single crystal structure (Figure 4).

With increase in the inductor height the pool depth is changed not only along the crystal axis, but also at its lateral surface, here the pool depth along the crystal axis is growing faster. It follows that it is rational to optimize the inductor height, possibly by changing its design, to perform the process in the condition close to optimum.

Coming from the results of preliminary investigations and technological considerations, the use of a two-section inductor was suggested. Sections may consist of several single-type coils connected in parallel. The expediency of use of this inductor was confirmed by the results of examination of patterns of thermal field of the single crystals forming under the action of single- and two-section inductors. In the experiment the total height of the inductor was not changed and was equal to the crystal length. However, the ratio of height of the upper and lower sections was changed. As is seen from Figure 5, the two-section inductor provides the larger depth of the pool. However, unlike the single-section inductor, where its effect on temperature of the lower part of the single

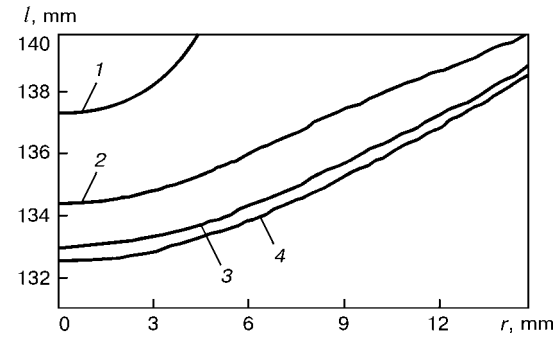

Figure 4. Change in pool depth and curvature of solid and liquid phases at different values of inductor height: *1* — 40; *2* — 60; *3* — 80; *4* — 100 mm (tungsten single crystal is 30 mm; specific power of inductor is 400 W/cm^2, power of plasma-arc source is 3.57 kW)

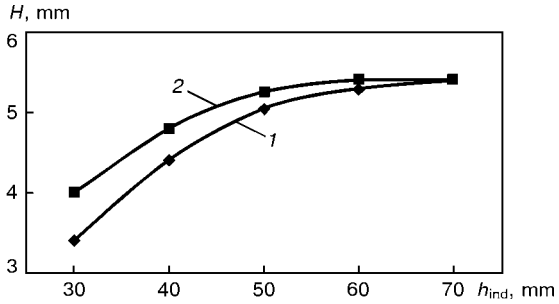

Figure 5. Effect of inductor height on pool depth: *1* — single-section inductor; *2* — two-section inductor (tungsten single crystal of 20 mm diameter and 100 mm height; specific power of inductor is 300 W/cm^2, power of plasma-arc source is 2.66 kW)

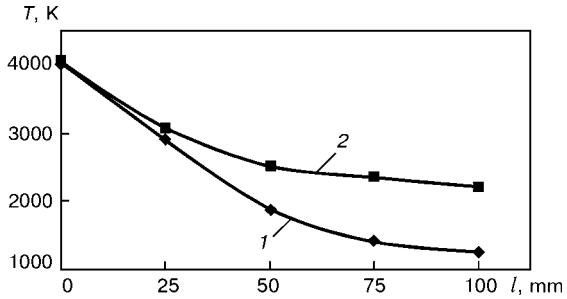

Figure 6. Distribution of temperature along the tungsten single crystal axis of 20 mm diameter and 100 mm height in use of single- (*1*) and two-section (*2*) inductors (specific power of single-section inductor is 300 W/cm^2; two-section inductor: upper section is 300 and lower section is 84 W/cm^2, height of upper section is 30 mm)

crystal is reduced with decrease in its height, in use of the two-section inductor the temperature of the lower part of the single crystal is remained unchanged after decrease in height of the upper section below critical value (Figure 6).

The processes of formation of the single crystal structure are not completed in the region of the phase interface, but they are continued in a solid phase. Therefore, it is extremely necessary to control the temperature in this part of the crystal. The structure of the single crystal is subjected to changes in the solid phase because of thermal stresses occurring under the action of the gradient of temperatures. If the temperature of the single crystal exceeds the half of value of an absolute temperature of melting [3], the stresses are relaxed and do not influence the processes of structure transformation in the solid phase. During growing the tungsten single crystal the temperature of induction preheating of its lower part should be higher than 1880 K, otherwise it does not provide the complete annealing of the growing crystal in the process of the zone melting. But the mentioned temperature is typical of the condition of a thermal deformation, and this is a state in which the dislocations are partially annihilated. Therefore, the lower section of the inductor should provide this temperature. As is seen from Figure 6, the two-section inductor creates the necessary temperature of the crystal preheating in its lower part.

Thus, the investigations demonstrate the feasibility of control of the pool depth and curvature of the interface of solid and liquid phases by changing the power of plasma and induction heat sources. The use of the two-section inductor makes it possible not only to control the temperature field in the pool region, but also to create conditions for relaxation of thermal stresses in the zone of a priming crystal.

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MICROCOMPUTER PANEL OF OPERATOR-TECHNOLOGIST OF ACS TP OF GROWING SINGLE CRYSTALS*

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It is shown that the most rational way of producing large oriented single crystals of refractory metals with stable properties using technology of plasma-induction zone melting is associated with the development of ACS TP. The comprehensive description of one of the functional elements of a partially automated control system of the process of growing crystals, i.e. microcomputer panel of the operator-technologist, is presented. The use of the developed panel enables the operator to make the monitoring and control of the process easier, and also to decrease the effect of a human factor on the reproducibility of properties of the crystals produced.

Key words: *single crystal, plasma-induction zone melting, ACS TP, object-oriented approach, microcomputer panel*

The probability nature of results, obtained in the process of development and improvement of the technological process, is typical of the most industrial technologies. The history of evolution of science and technology shows that at the stages of their formation a human factor plays one of the key roles in the stability of results obtained. In this aspect, the role of an operator in the development of unique precision technological processes, such as method of growing large oriented single crystals of refractory metals using a plasma-induction zone melting (PIZM), developed at the E.O. Paton Electric Welding Institute of the NAS of Ukraine, is especially actual [1].

The peculiar feature of this method is the application of two independent heat sources (plasma-arc and induction). The growing of single crystals is performed by a layer-by-layer deposition of consumable rods by a plasma arc on a single crystal primer, located in the zone of action of a high-frequency field of the inductor. The degree of structural perfection, stability of geometry, quality of a lateral surface of PIZM single crystals depends greatly on the operator's skill.

The producing of single crystals of such refractory metals as tungsten and molybdenum with stable properties is a necessary condition for the development of technological processes of their further processing, for example, wide-section single crystal rolled metal. This will make it possible to widen greatly the existing fields of application of single crystals and to find, possibly, the new applications. The continuous control of a plasma arc power and high-frequency heating, control of consumption and composition of plasma-forming mixture of gases (argon + helium), as well as control of molten metal pool in the formation of single crystal are realized, at least, with three operators. If to take into account that the duration of the technological process of growing crystals, using PIZM technology, exceeds 20 h, then the role of human factor in reproducibility of properties of the crystals produced becomes very important, and the process performance can be referred to the category of art. The solution of this problem is associated with the development of the automatic control system of technological process (ACS TP) of growing single crystals.

Within the scope of the fulfilled complex of research and design works on the study and automation of the process of plasma-induction growing of single crystals of refractory metals a partially automated system of technological process has been developed and designed. In the development of the architecture of ACS TP of growing single crystals an object-oriented approach was used [2]. Therefore, ACS TP represents a network distributed system of automated control (Figure 1), whose elements are linked by network concentrator (HUB Ethernet 10/100 Base-T) and interacted with each other with the help of the reliable network protocols (TCP/IP).

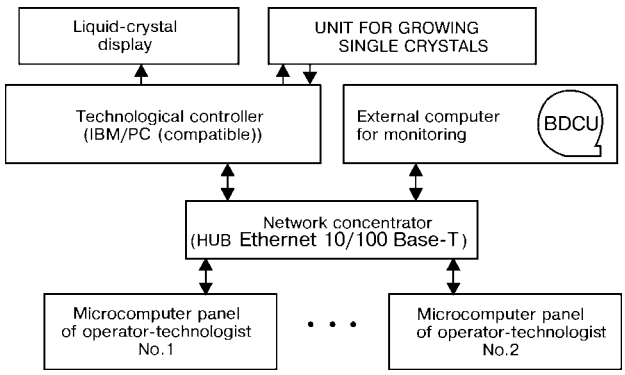

Figure 1. Integrated structure of ACS TP of growing single crystals

* The work was fulfilled under the finance support of Science and Technology Center in Ukraine.

Figure 2. Appearance of microcomputer panel

One of the functional units of ACS TP is a microcomputer panel (Figure 2) of the operator-technologist (further panel), which is designed for a fast monitoring and control of the technological process of the crystal growing. The panel is used for realizing works of ACS TP both in the automatic and manual conditions. The use of the panel releases operator from manipulating with different buttons and switches and constant observing the arrows of instruments, which are arranged on different panels of the technological equipment. In addition, the panel realizes the checkout of operations being fulfilled and can block the erroneous actions of the operator.

The panel represents a microcomputer, by its design, of IBM PC/AT-386-40MHz type (Figure 3), assembled on the base of microcomputer ICOP-6015 (Figure 4) [3]. The panel includes an integral membrane keyboard with abridged set of buttons (20 pcs), LC-indicator is connected to output of microcomputer printer port, network map of microcomputer of NE-2000 type and port of a successive exchange (RS-232) are used. The working program of the panel is realized in language C++ and operates in medium OC MS DOS. Interaction through the interface Ethernet 10 Base-T is performed using a package TCP/IP-protocol (OC BSD type). Program-driver for a controlling computer, providing interaction with the panel has been designed and delivered.

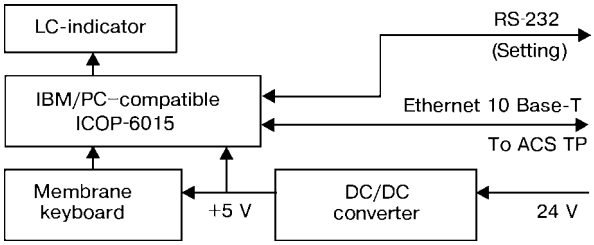

Figure 3. Schematic diagram of microcomputer panel

The panel has a dust and spatter-protective casing. It is convenient in use by keeping in hands or arranging in any place, necessary for the operator. The panel allows operator to fulfill the following operations: to monitor the real values of current and voltage of the plasmatron; to change the current set-up of the plasmatron; to change the set-up of inductor power; to control the feed of left and right rods («FORWARD», «BACKWARD» and «STOP»); to control the ingot withdrawal; to control the plasmatron displacement; to change quickly the technological process parameters in interactive condition; to reprogram the technological process macrooperations («MELTING RUNNING», «START OF GROWING SINGLE CRYSTAL» and «STOP OF GROWING SINGLE CRYSTAL»).

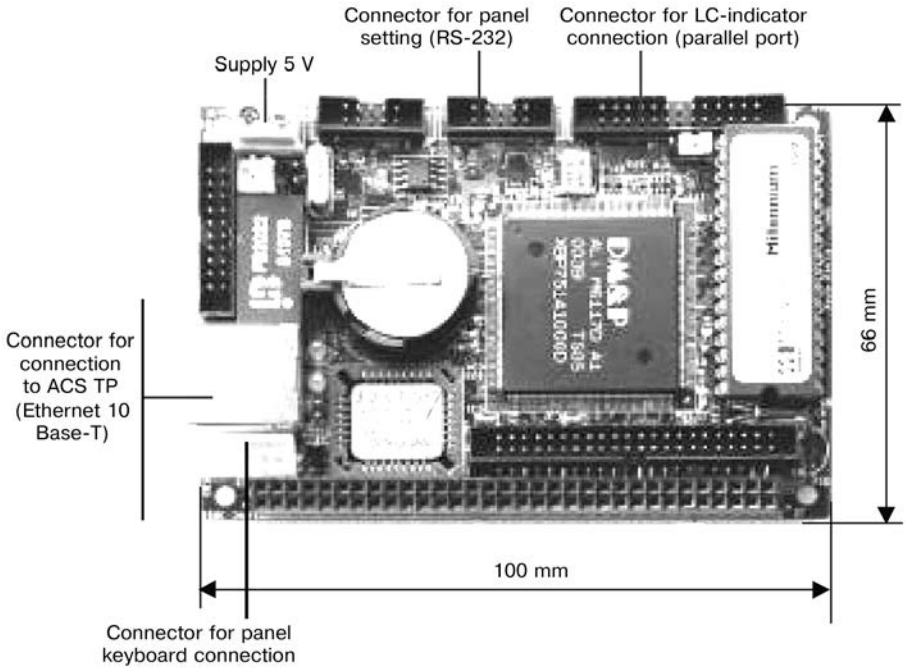

Figure 4. Scheme of microcomputer ICOP-6015

In the development of the controlling program the state graphs were used to realize the real time condition. Example of a controlling graph of states which realizes the condition of correction of set-ups of plasmatron and inductor power is shown in Figure 5. Here, in state «0» with about 1 ms time period the port for input data of network map Ethernet is interrogated and in case of pressing buttons «I» or «W» on the panel the transition occurs into states «1»–«2» or «3»–«4», respectively, where the pressing of buttons «+» or «–» is processed. In addition, the value of set-up is either increased by value «delta», or decreased. When graph is functioning for realization of operation condition with an operator the appropriate messages (procedure «mes») are supplied to the panel. To come out from condition of set-up correction a button «C/R» should be pressed on the panel (states «5»–«0»). When pressing button «Enter» the current value of setting is given to the value of set-up and it is processed by the circuits of automatic stabilizing of the melting condition.

The panel has wide opportunities when interacting the operator. To reflect the information on LC-indicator the following conditions are realized: output of static text; output of text with a flickering; output of text in the form of running line.

There is a special condition of output of indications of timers which can be used to control the technological operation duration. For the operator's convenience a condition of indication of current time and date is introduced. Condition of the panel blocking is realized which can be used in situations when it is necessary to limit the access to the control of technological process. The condition of panel blocking is provided by hardware without use of a mechanical lock.

The panel is supplied from the DC power source. The channels of communication are decoupled galvanically relative to supply circuits and each other.

An additional advantage of the offered solution is the fact that it is possible to use several panels (up to 32) simultaneously in the composition of ACS TP, which can have identical purpose or differ in their functions. The main characteristics of the developed panel are as follows:

Processor	M6177D 386SX-40
Capacitance of FLASH disc, Megabyte	0.512–288

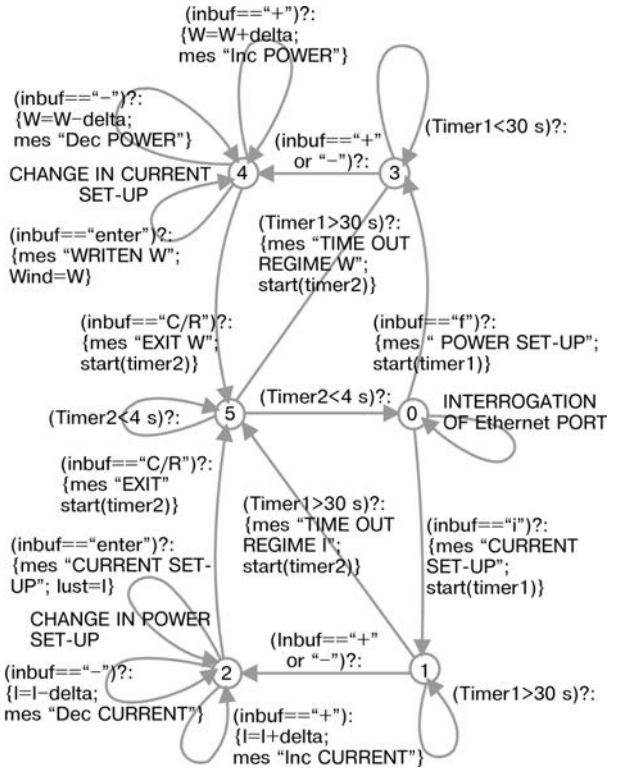

Figure 5. Controlling graph of states for conditions of correction of set-up of plasmatron current and inductor power

Sign-synthesizing LC-indicator	2 lines by 16 symbols
Keyboard	membrane, 20 buttons
Range of supply DC voltage, V	10–36
Connector	type 2RM22 KUN10Sh1V1
Consumed power, W	≤ 4.0
Dimensions, mm	197×184×84
Degree of protection	IP54
Operating temperature, °C	0–50

The panel can be also used in the composition of the automatic system of control of welding and assembly processes where a continuous control of technological parameters is required and the operator has a feasibility to interfere in the technological process.

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INVESTIGATION OF INTERPHASE INTERACTION OF FERROTITANIUM WITH BORON CARBIDE IN POWDER MIXTURES FOR DEPOSITION OF THERMAL COATINGS

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Interaction of ferrotitanium with boron carbide, which is accompanied by a significant exothermal effect, is investigated. It was established that the maximum exothermal effect is attained at ferrotitanium interaction with boron carbide in powder mixture of the following composition: 85 wt.% FeTi + 15 wt.% B_4C with the formation of reaction products TiB_2, TiC, Fe_2B, Fe_3B, Fe_3C. This powder mixture is the promising material for coating deposition using the methods of thermal spraying.

Key words: *exothermal interaction, differential thermal analysis, microstructure, phases of Fe–Ti–B–C system*

Boron carbide, owing to its unique properties, such as high hardness, wear resistance, chemical resistance and others, finds a wide application in the modern engineering both in a pure form and also in the form of cermets. Alloys on the base of diborides of transition metals with additions of iron and boron carbide (5–15 wt.%) are used as surfacing materials [1]. Alloy of boron carbide with 5 wt.% of titanium, possessing, from the data of [2], the highest hardness among the materials on the base of boron carbides and metals of IV–V groups (0.5–10 wt.% of metal) is used as an indentor for measuring microhardness at high temperatures [3]. As was shown earlier [4], the boron carbide in combination with refractory metals, in particular with titanium, is the promising material also for deposition of wear-resistant coatings using the methods of thermal spraying. In addition, in the process of thermal spraying the interaction between components of the composition leads to the formation of carboboride alloys of titanium which have the higher level of mechanical properties than carbide and boride of titanium. From the data of [5] the minimum values of coefficients of friction and wear for Ti–B–C system are observed in alloys of eutectic composition.

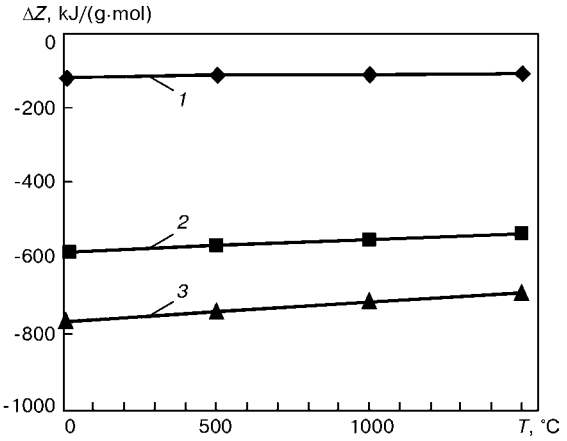

Figure 1. Temperature relationship of Gibbs energy change for reactions of boron carbides with titanium (numbers of curves correspond to numbers of reactions in Table 1)

Carbide TiC and titanium borides Ti_2B_5, TiB_2, Ti_3B_4, TiB belong to those compounds, whose formation in the condition of self-spreading high-temperature synthesis (SHS) is accompanied by a high exothermal effect and adiabatic rise of temperature T_{ad} both in case of a direct synthesis from elements [6, 7] and also as a result of titanium reaction with boron carbide [4, 8]. Thermodynamic estimation of titanium interaction with boron carbide (Table 1, Figure 1) shows that at concentration of initial substances, corresponding to stoichiometric coefficients of equation

$$B_4C + 3Ti = TiC + 2TiB_2,$$

the adiabatic rise in temperature reaches 2869 °C.

As to the system Fe–B_4C, then it does not belong to highly-exothermic. For thermodynamically most probable reaction of formation of iron boride in mixture with carbon (reaction 3, Table 2, Figure 2), the adiabatic rise of temperature is 651 °C. As a result of SHS the porous products of interaction are, as a rule, formed. One of causes of their porosity is large volume changes of components during the synthesis. Therefore, as shown in [9], the producing of porous-free SHS-composite of Ti–B–C is possible by combination of the process of combustion with a next rolling of the synthesis products. The material produced is similar to carbide-tungsten materials by its properties.

To select the composite material composition for coating deposition using the methods of thermal spraying the interaction of ferrotitanium with boron carbide was investigated in the present work. As initial materials, the powders of ferrotitanium (44.8 wt.% Ti) with particle size of 40–100 μm and boron carbide with particle size of 5–40 μm (content

Table 1. Thermal activity of reactions of boron carbide with titanium

No.	Reactions	B_4C content		Value of heat effect			ΔT_{ad}, °C
		wt.%	vol.%	kJ/(g·mol) of B_4C	kJ/(g·mol) of mixture	kJ/cm^3 of mixture	
1	$B_4C + Ti = TiC + 4B$	54	67	121.68	1180	3.72	605
2	$B_4C + 2Ti = 2TiB_2 + C$	37	51	585.26	3874	13.52	2483
3	$B_4C + 3Ti = TiC + 2TiB_2$	28	41	768.94	3860	14.26	2869

Table 2. Thermal activity of reactions of boron carbide with iron

No.	Reactions	B_4C content		Value of heat effect			ΔT_{ad}, °C
		wt.%	vol.%	kJ/(g·mol) of B_4C	kJ/(g·mol) of mixture	kJ/cm^3 of mixture	
1	$B_4C + 8Fe = 4Fe_2B + C$	11	28	51.8	103	6.58	119
2	$B_4C + 11Fe = 4Fe_2B + Fe_3C$	8	22	26.7	40	2.67	50
3	$B_4C + 4Fe = 4FeB + C$	20	44	196.4	705	3.90	651
4	$B_4C + 7Fe = 4FeB + Fe_3C$	12	30	171.3	384	2.40	426
5	$B_4C + 3Fe = Fe_3C + 4B$	25	50	−87.1	−39	−2.0	−3.25

of particles of size up to 5 μm was about 70 vol.%) were used.

The interaction was studied using the method of a differential thermal analysis (DTA) on samples manufactured from mixture of powders of ferrotitanium with 10, 15 and 30 wt.% of boron carbide. The mentioned compositions cover the regions of thermodynamically most possible reactions: Ti–28 wt.% B_4C and Fe–20 wt.% B_4C. Heating and cooling in DTA were performed at a constant rate 80 K/min in medium of high-purity helium at 50 Pa pressure. Cylindrical samples of 0.5 g, preliminary pressed at 3 t/cm^2 pressure, were placed into crucibles made from zirconium dioxide. The produced ingots were examined using the methods of metallographic, microdurometric and X-ray diffraction phase analysis (XDPA). Results of DTA are given in Figure 3 and Table 3. Nature of DTA curves proves that mechanism of interaction is similar for all three compositions examined. The ratio of intensity of exothermal peaks

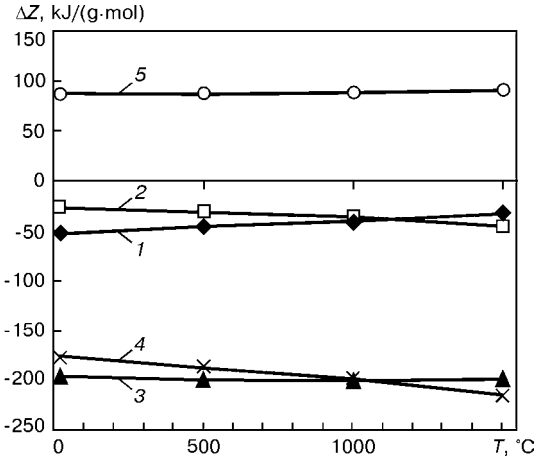

Figure 2. Temperature relationship of Gibbs energy change for reactions of boron carbide with iron (numbers of curves correspond to numbers of reactions in Table 2)

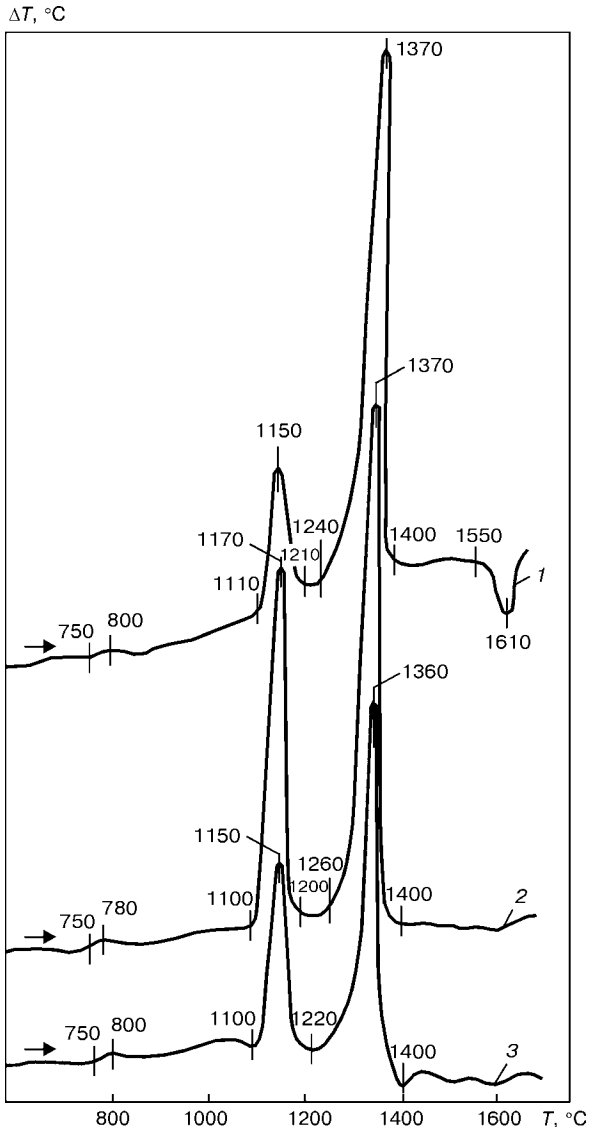

Figure 3. Thermograms of powder mixtures of ferrotitanium with 30 (*1*), 15 (*2*) and 10.5 (*3*) wt.% B_4C

Table 3. Results of investigations of products of interaction FeTi with B_4C

No.	Mixture composition, wt.%	Parameters of exothermal peaks		Total peaks area, mm^2	Ratio of peaks areas	Phase composition* of DTA ingots
		Maximum temperature, °C	Area, mm^2			
1	70FeTi + 30B_4C	1150 1370	470 1710	2180	0.3	TiB_2, TiC, Fe_2Ti, B_4C, $B_{13}C_2$, FeTi
2	85FeTi + 15B_4C	1170 1370	1000 1750	2750	0.6	TiB_2, TiC, Fe_3B, Fe_2B, Fe_3C, B_4C, $B_{13}C_2$, Fe_2Ti
3	89.5FeTi + 10.5B_4C	1150 1360	570 1243	1813	0.5	TiB_2, TiC, Fe_2Ti, $B_{13}C_2$, B_4C, Fe_3C, Fe_2B, Fe_3B

*Phases of initial mixtures: FeTi (basic), Fe_2 Ti (impurity), B_4 C (basic), $B_{13}C_2$ (impurity).

is only changed. Maximum total area under the curves corresponding to the value of heat effect of the reaction is referred to composition FeTi + 15 wt.% B_4C. In all three compositions, in accordance with data of XDPA, titanium diboride TiB_2 and titanium carbide TiC_{1-x} are the main phase components. Except them, the residual boron carbide (B_4C with $B_{13}C_2$ impurity) and ferrotitanium of Fe_2Ti composition (in mixtures with 10 and 15 wt.% B_4C) were revealed.

Samples after DTA were porous (Figure 4). As the reaction is accompanied by significant volume changes (volume reduction in formation of mixture TiC + TiB_2 is about 10 %), reagents occurred to be soon separated not only by the products of interaction, but also by a continuous zone of porosity [8]. The rate of reaction proceeding is abruptly decreased in this case, and non-reacted initial substances are preserved in the sample. As a result of interaction of initial components a fine-dispersed multi-phase structure, whose microstructure is 11.0–19.3 GPa, is formed.

To clarify the sequence of formation of new phases in DTA the powder mixture was heated to the temperature of completion of the first stage of exothermal reaction (1170 °C) with a subsequent intensive cooling (at about 200 K/min rate).

Method XDPA revealed phases TiC (basic), TiB_2, Fe_3C and initial FeTi and B_4C in the sample after DTA. This indicates that the first exothermal peak is due to SHS proceeding with the formation of the titanium carbide, while the second peak (higher in value) is associated with the formation of titanium diboride, as TiB_2 dominates in the reaction products after heating above 1370 °C. As to the synthesis of borides and carbides of iron, then the contribution of exothermicity of processes of their formation to total heat of reaction is negligible, that is proved also by the results of thermodynamic calculations.

It is possible to assume the following mechanism of FeTi interaction with B_4C on the basis of data of DTA and XDPA. Boron carbide comes into reaction

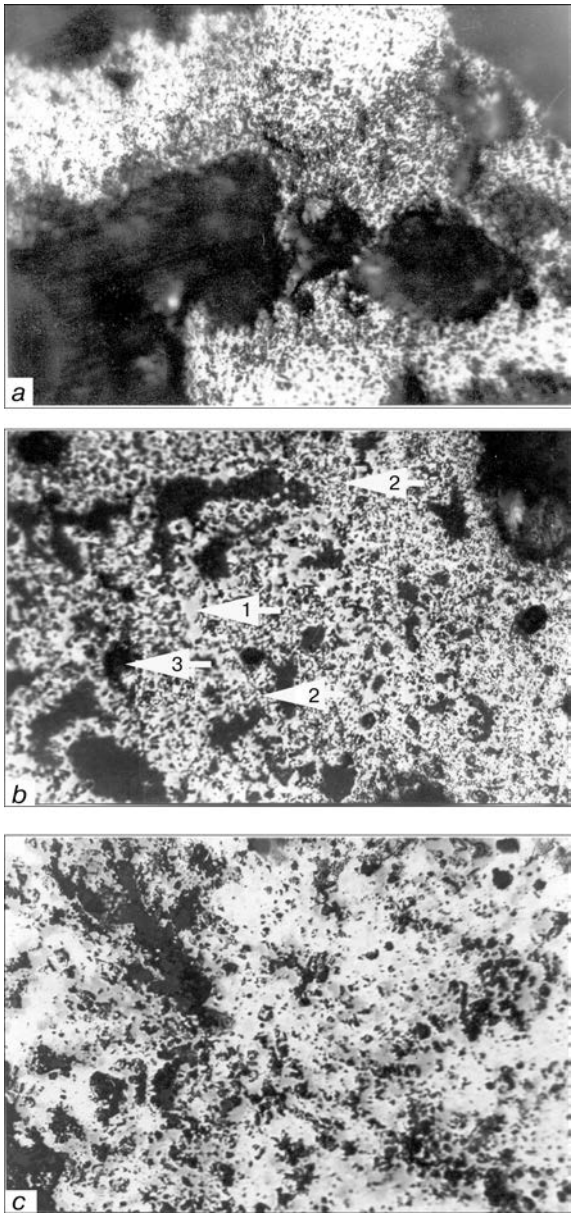

Figure 4. Microstructures of products of interaction of ferrotitanium with 30 (*a*), 15 (*b*) and 10.5 (*c*) wt.% B_4C: *1* — titanium carbide; *2* — titanium diboride; *3* — boron carbide (×400)

with titanium, included into ferrotitanium composition, with the formation, first of all, of TiC, as the coefficient of carbon diffusion in titanium is higher than the coefficient of boron diffusion [8]. Titanium diboride is formed at higher temperature (second stage). Decrease of titanium content in ferrotitanium leads to the transition of FeTi to Fe_2Ti, that is proved by redistribution of intensity of X-ray lines of the mentioned phases in DTA-samples as compared with initial mixture of powders. If the main phase in initial mixture is FeTi, and Fe_2Ti is only impurity, then in DTA ingots it is vice versa, FeTi is revealed only in ingot with 30 wt.% B_4C in the amount which is smaller as compared with Fe_2Ti.

As to the newly formed phases on iron base, then their content in DTA products is small, and in the sample from initial mixture with 30 wt.% B_4C they were not revealed by the XDPA method.

Comparison of the results obtained with data of earlier investigations of interaction in Ti–B_4C system [4] shows that replacement titanium by FeTi leads to the shifting of temperature of beginning of the active interaction with B_4C into the region of lower temperatures (from 1300 to 1110 °C). In addition, in case of interaction in Ti–B_4C system both exothermal peaks are coalesced into one, whose width is, respectively, increased. It is characteristic that products of interaction both in Ti–B_4C system and in FeTi–B_4C system are porous, reaction is not interrupted even after heating up to 2000 °C. However, as was shown earlier [4], a special preparation of initial materials (for example, rolling and preliminary sintering of initial mixture of the mentioned powders with a subsequent crushing and selection of a necessary fraction) makes it possible to produce dense coatings with a high strength of adhesion with the base using the method of a plasma spraying.

Thus, the interaction of ferrotitanium with boron carbide is accompanied by a significant exothermal effect due to synthesis of TiB_2- and TiC-phases characterized by the highest values of formation heat. Maximum exothermal effect is attained in use of 85 wt.% FeTi + 15 wt.% B_4C mixture. Here, a fine-dispersed multiphase structure, consisting of boride and carbide phases TiB_2, TiC, Fe_2B, Fe_3B, Fe_3C with 13.6 GPa mean microhardness, is formed. The powder mixture with this combination of initial components is the challenging material for deposition of protective coatings using the methods of thermal spraying. To decrease porosity, a preliminary treatment of powder mixture before spraying or the rolling of as-sprayed coating are necessary.

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FUNCTIONAL GRADIENT MATERIALS: NEW MATERIALS SCIENCE SOLUTIONS

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Problems of development and realization of new materials science solutions based on the technology of functional gradient materials (FGM) are considered. Tendencies in the development of theory and technology of FGM are analyzed, examples of solutions for producing metallic composites, hard alloys, protective coatings, and also more complex components for power engineering are given.

Key words: *functional gradient materials, composites, hard alloys, protective coatings, modelling*

Tendencies of the development of advanced materials science were concentrated over many years on producing homogeneous quality alloys and other materials having constant preset properties and service characteristics [1–5]. Conventional composite materials, produced by integrating metal or ceramic matrix and a dispersed phase, use the synergetic characteristics of each of these constituents. These composites have a uniformly distributed strengthening phase and their resulting properties are homogeneous.

In many fields of engineering a problem has been appeared in the creation of functional structures of materials with a high degree of relaxation of thermal stresses, resistance to oxidation and thermal shock, that required the producing of combination of different properties in a definite anisotropy of material structure. To solve this problem the works in many countries are carried out for the creation these materials [6–11], named «functional gradient materials» (FGM).

FGM represent in a general case a material characterized by a preset distribution of composition, structure or properties in volume. FGM are differed from isotropic materials by the presence of a gradient of structure and properties (hardness, density, heat conductivity). These gradients are created by special processes and controlled quantitatively to improve significantly the properties of a final product [6, 8, 10].

The first task was to develop materials and structures for space vehicles, capable to withstand thermal stresses. However, this conception was greatly widened for the development of combinations of different materials without a clearly-expressed interface to produce structures with new numerous functions [9–11].

In spite of undoubted progress in this field over the recent years and large growth of research works, the theoretical investigation and development of principles of FGM remain insufficient. In the major part of works, a definite method, used for FGM production, is mainly investigated [11]. There are no almost works associated with the development of an adequate description of FGM properties and their dependence on concentration gradient that should provide the main result of «feedback» used for direct designing of FGM-components. The most experimental trends of investigations in the field of FGM have led to the accumulation of comprehensive experimental material which cannot be used properly due to the absence of a sufficient theoretical base. It should be noted that situation in this direction is improved, as during recently the theoretical bases and «design» of these materials are being developed. In spite of producing definite samples and components, the application of the obtained materials science solutions in industry requires, nevertheless, additional investigations.

To produce FGM-component it is necessary to develop firstly the preliminary design of this material which should possess the desirable characteristics. Theoretical statements, used for designing composite materials, are not, as a rule, suitable for the calculation of properties and determination of optimum structure of FGM [6, 12].

Similar gradient materials are characterized also by more complex behaviuor as compared with conventional (homogeneous) materials. Diffusion processes and chemical reactions can lead to the degradation of FGM or deterioration of their properties. Therefore, the second task in the development of FGM technology is the creation of components with gradients, required by the customer, taking here into account the possible side effects in use of these materials.

Critical analysis, made by the authors, made it possible to classify the approaches to obtain the gradient of concentration in materials. It is possible to produce the gradient of composition in volume FGM by different methods, for example, chemical and electrochemical, physical methods (deposition from vapour), plasma spraying and synthesis, self-spreading high-temperature synthesis, however, a powder metallurgy (PM) is the most economical technology.

The simplest method of producing FGM using the powder metallurgy consists in pressing of different powder mixtures in a preset sequence and subsequent sintering or hot pressing. The works [6–14] give more than hundred combinations of FGM, produced by the

PM. Numerous applications of FGM (above 200) were identified in Japanese national program (for example, tools, barrier coatings, heat-resistant and anticorrosion components, piesoelectric heads, electron and optical devices, etc.) [9, 10]. At present the investigations are carried out in the field of application of FGM in electronics, optics, nuclear engineering, medicine, where the gradient of other properties (refraction characteristic, radiation resistance, dielectric permeability, etc.) is more important [11, 14].

Structure of gradient material in the simplest case is characterized by anisotropic distribution of one component in the volume of another component (for two-phase system). FGM may have chemical (metal–metal, ceramics) or physical (solid material–pores, coarse–fine particles) anisotropic nature. Distribution of phase and its anisotropy can be described by a combination of functions in three-dimensional Euclidean space. In some cases there are difficulties in measuring the function of concentration itself, that requires numerous experiments and numerous calculations, especially for the materials, produced by the PM. Modelling is the real alternative for description and calculation of FGM [10–14].

For example, the developed micromechanical model [6, 12] was used for description and prediction of properties of some FGM with an arbitrary three-dimensional gradient of concentration of the component and their behaviour under the test conditions. Calculations made using the developed models and theoretical statements showed the sensitivity of the model offered to a shape of function of the concentration gradient as compared with relationships used usually in the theory of composite materials [15, 16].

The results of calculations according to the developed software were compared with other approaches, used usually in the theory of composite compounds, of type of rules of mixture and other models for a coefficient of thermal expansion, heat conductivity, modulus of elasticity and shear. Figure 1 shows the calculated properties of FGM tungsten–copper with a different type of function of distribution, that confirms the sensitivity of the offered model to the kind and «shape» of function of concentration, while other relationships take into account only the volume concentration of components [15–17].

The advantage of these models is that the preliminary evaluation of stress level can be made even before its experimental measurement without use of complicated numerical methods. Thus, there appears a feasibility to define the ways of «optimum» gradient of FGM even at the initial stages of the design [10, 14].

Modelling of processes of producing FGM is especially effective at more complicated process of PM, for example, in sintering materials with a strong interaction of phases. When developing the model of sintering of gradient hard alloys the above-mentioned micromechanical model was used for calculation of mechanical and thermal properties of FGM with an arbitrary three-dimensional gradient of composition

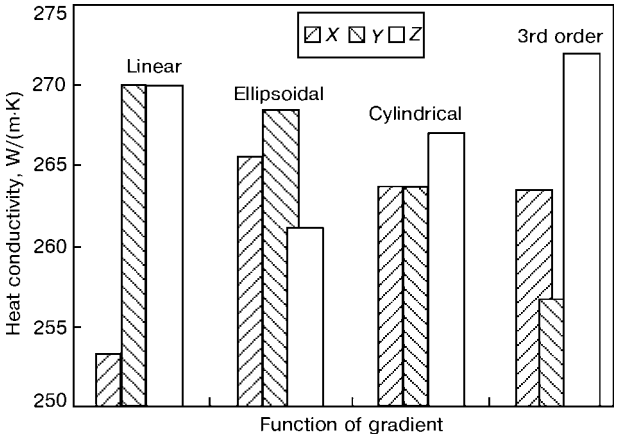

Figure 1. Heat conductivity in axes *X*, *Y* and *Z* for different functions of distribution of copper concentration in FGM tungsten–copper (total volume share of copper is 0.5)

within the preset boundary conditions at a proper heat flow before calculation of the sintering process proper [18, 19]. The computer program has been created on the basis of experimental measurements and above-developed theoretical statements. This program makes it possible to evaluate the necessary parameters of the process of producing gradient hard alloys. To make experimental evaluation of the model, an optical dilatometer was developed, with the help of which it is possible to determine macroscopic values of displacement of sample continuously during all the sintering process. Figure 2 shows the distribution of stresses occurring due to non-uniform sintering of FGM WC–Co. The quantitative results showed that the model developed for hard alloys predicts exactly the density of material and change in shape of the sample during sintering. The use of adequate modelling can reduce greatly the need in the experiment conductance.

As another example of practical application of FGM it is possible to illustrate the so-called matching complaint pads which are designed for reduction of the level of stresses in devices for conversion of heat energy into electrical energy. In these systems a thermoelectrical effect is used, which is based on the property of semiconductors to generate the difference of potentials at an appropriate temperature gradient.

Practical problem of realization of this device is the difference of coefficients of thermal expansion of semiconductors and metallic materials that cause high thermal stresses. The use of matching FGM-pads makes it possible not only to reduce the level of stresses by several times, but also to create the preset anisotropy of electrical conductivity at a sufficiently isotropic heat conductivity [21, 22]. Figure 3 shows the pilot sample of this FGM-pad produced in Helsinki University of Technology using the method of PM and consolidated in Japan using the method of electric spark (plasma) sintering under pressure [23].

These pads are designed for conversion of nuclear energy into electrical energy. Conception [21] of nuclear power station on the Moon, developed in Japan, predicts its failure-free operation during 20–30 years.

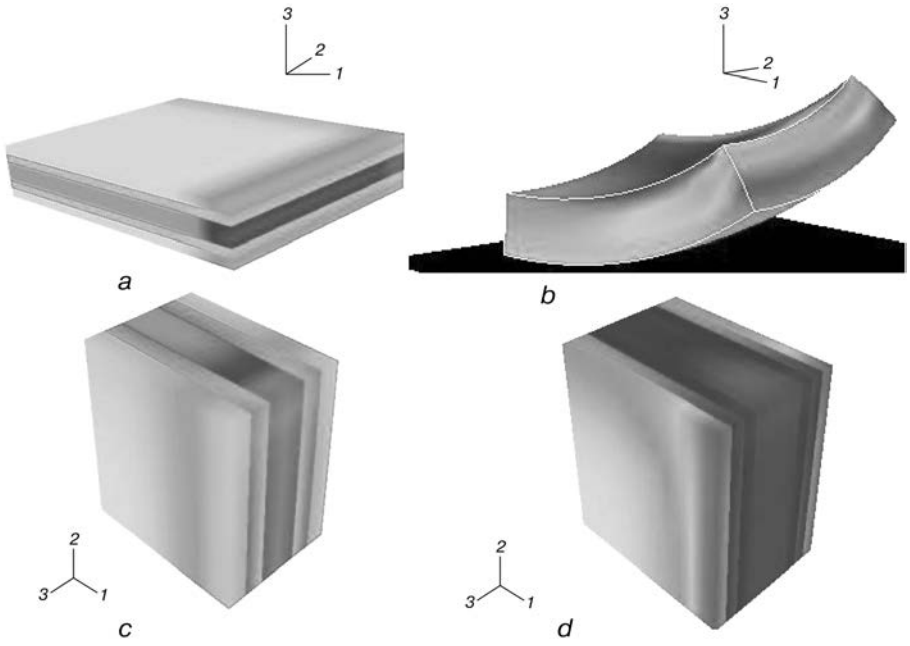

Figure 2. Distribution of stresses in symmetrical (*a*) and non-symmetrical (*b*) FGM-pads after sintering, and also in a symmetrical pad with a small (*c*) and large (*d*) number of gradient layers [19, 20]

In this case the maintenance and replacement of units should be reduced to minimum. Similar reactor RAPID-L of 200 kW capacity occupies only 24 m^3 and, therefore, can be also used on Earth. The reactor operation is fully automatic, as it has no controlling rods, instead of them a principle of mercury thermometer is used where the liquid lithium-6 plays a role of mercury. With a temperature rise, lithium is expanded and adsorbs neutrons more actively, thus delaying reaction. The liquid heat-carrier is circulating around the contour with the help of an electromagnetic pump. Thermoelectrical modules convert a part of heat into electrical energy supplied to the consumer [24]. Thus, the whole process takes place without use of any moving mechanisms, but only on the basis of physical laws. It was shown [21, 22] that the optimized design of FGM-pad can accept a heat flow of about 300 kW/m^2, that even at the 10 % present level of efficiency factor of conversion, allows consumers to be supplied both with heat and also electricity. Simpler structure of FGM and method of its producing makes it possible to reduce the cost of system and to increase its efficiency.

One of the most quickly-spreading application of FGM is their use in different coatings (power engineering, gas turbines, chemical industry, etc.). In these fields the coatings should be resistant to high temperatures, aggressive media and thermomechanical cyclic actions. From the point of view of cost of production, the simple methods of plasma and thermal spraying in air are most attractive. Composition, structure and design of FGM-coating were first optimized using models [25], accounting for elastic and plastic deformation, geometry of component and operation conditions. The samples produced were subjected to thermocycling in a special stand under the conditions of changing heat flow from 0 to 0.85 MW/m^2.

FGM-coatings demonstrated much higher service life than conventional coatings (number of cycles before fracture was increased by hundred times) and absence of long horizontal cracks (Figure 4).

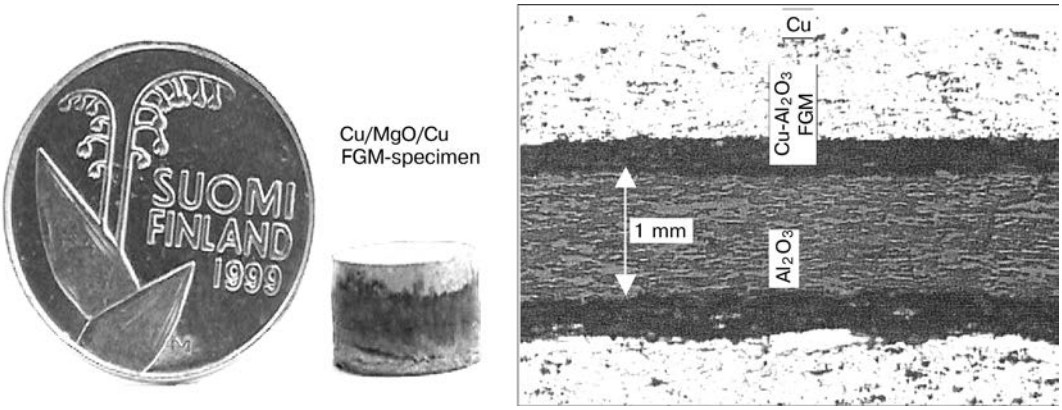

Figure 3. General view (*on the left*) and structure of copper–aluminium oxide–copper FGM-pad [21, 22]

Microcracks in the area of coating with a larger fraction of a ceramic phase are favourable as relaxators of stresses (3–8 times reduction of stresses as compared with a reference sample), decreasing simultaneously the heat conductivity of the entire coating. FGM-coatings protect reliably the metal surface from oxidation, that was confirmed by long-term tests. Air annealing at 1000 °C for 330 h showed that there were no noticeable chromium diffusion and formation of spinel under the FGM-coating, while the reference sample (duplex coating without FGM) had 2–3-fold depletion of metal coating with chromium.

Similar approach was used in the development of coatings for the chemical industry (processing of materials of a cellulose production in so-called soda boilers). These coatings should operate several years in melts and gas atmospheres with a high content of chlorine, sulphur, soda and salts of alkali metals.

The composition and structure for this coating (Figure 5) were selected so that to provide compressive stresses in a ceramic layer, synergetic passivity of a cermet layer and effect of delay of corrosion reaction due to creation of deposits of inert products of corrosion in the coating micropores. It was revealed after investigations that there are no almost sulphur, chlorine and alkali metals in a metal-containing layer of FGM-coating [25]. Under these conditions the duplex ceramic coatings do not withstand occurring stresses, and a special stainless steel begins to fracture noticeably even after 10–20 h.

FGM-coatings showed the great advantages in protection of carbon/carbon fibrous composites. Potential of use of these composites is limited usually by a low resistance of carbon fibres and matrix to oxidation at high temperatures. Homogeneous or multilayer coatings have a limited term of service and do not allow product to be under service without a sufficient level of reliability. It was suggested to use the conception of FGM to produce the multilayer structure with different levels of a functional gradient [26]. Unlike the other methods, there are no abrupt interface between the phases in the structure of as-produced material. Behaviour of material at cyclic oxidation in the range of mean temperatures (500–700 °C), where conventional protective mechanisms (formation of SiO_2 layers or silicates) have not yet operated, was investigated using a method thermogravimetry (Table). A noticeable oxidation of carbon fibres under FGM-layer was not observed, and the FGM-coating itself was not laminated during tests and after them (in determination of sample strength). The investigations show the advantages of FGM for the protection of carbon composites from oxidation as compared with conventional materials and homogeneous coatings [6, 26].

Properties of FGM-coatings on carbon composites [26]

Parameters	*Serial material*	*Experimental FGM*
Tensile strength at 20 °C, MPa	200–350	370–400
Time of thermocycling in air, h (20 — 1500 — 20 °C, cycle time 20 min)	27	200
Reduction in tensile strength, %	20	0–2
Number of thermal cycles in air:		
20 — 1350 — 20 °C for 2 min	20–35	50
20 — 1100 — 20 °C for 5 h	5*	4
20 — 600 — 20 °C for 1 h	1–5*	20

*Material fractured.

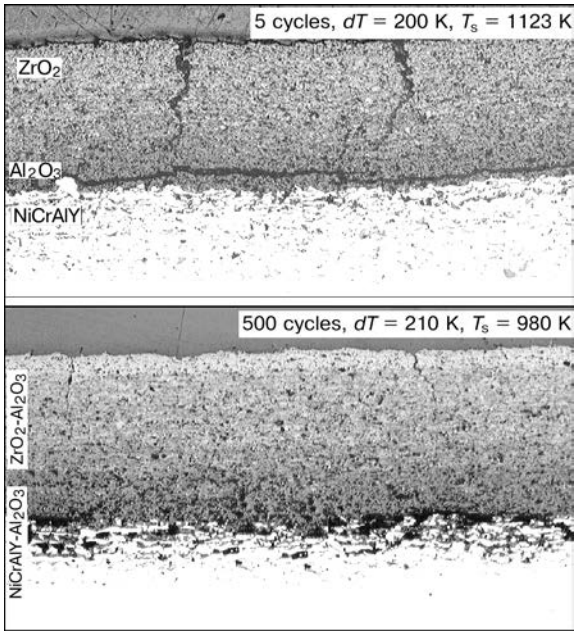

Figure 4. Conventional (*at the top*) and FGM thermoprotective coatings after tests

Similar to FGM-coatings, the solutions were developed for joining ceramics with metals, designed

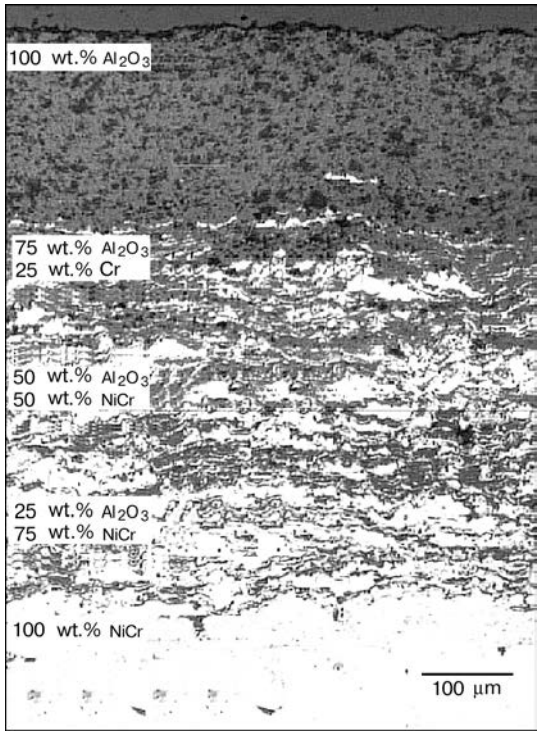

Figure 5. Protective gradient coating on carbon steel [25]

for operation under the conditions of high temperatures and mechanical stresses (power equipment). The use of FGM in these joints makes it possible to reduce the level of thermal stresses of dissimilar materials, for example, heat-resistant alloy IN-738 and ceramics on Al_2O_3 base. It is shown [6, 8, 10, 11] that the change in anisotropy characteristic (function of phase distribution in FGM) leads to different kinetics of generation of thermal stresses, that stipulates the longer term of service of these joints. Optimum parameters of this FGM-layer were determined and dynamics of change in stresses in case of a thermal shock was calculated.

FGM demonstrated also effectiveness in the more «exotic» applications, for example, in the conditions of a thermonuclear reactor. Structural materials which can be used in the reactor, should meet many requirements, such as high resistance to heat shock, thermal fatigue, high mechanical strength, resistance to neutron radiation and others. An important characteristic of materials is their effect on plasma parameters. Plasma contaminations deteriorate its characteristics and their removal is made by a discharge on diverters. During discharge the plasma along the lines of the magnetic field is damped on the plates of diverters for very short time, thus causing instantaneous evaporation of a thin layer of the material [6, 16, 17].

Most part of this evaporated material is deposited back on the surface, thus trapping the undesirable impurities from plasma. Relaxation of thermal stresses was determined for FGM tungsten–copper. In [27] the model of FGM-diverter was studied and thermal stresses in its different parts and also effect of gradient on their change were determined. The use of theoretical and model statements developed [6, 9–12, 14–17] made it possible to calculate FGM tungsten–copper for plates of diverters and to offer an optimum design of the material allowing 3–6 times decrease in the level of thermal stresses. The analysis shows that the use of FGM in reactors of other designs, power equipment of related branches of machine-building can increase significantly the service life of different components.

Thus, the general situation with the progress of materials science solutions on FGM base represents a great interest. However, at present the practical application of FGM is difficult due to the lack of data about their use in various operation conditions. The initial design of FGM gives calculations of optimum gradient of some properties and composition. This gradient cannot be determined without knowledge in precise requirements for the conditions of application of this material. If these requirements are known, then the second important problem is the reduction in cost of FGM production as compared with homogeneous materials having, however, the lower level of service properties. Taking into account the tendencies of increasing the requirements for the materials, it is expected that the use of the FGM-solutions will be one of the widely used trends in the materials science [28–30].

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INTERNATIONAL SEMINAR «MODERN TECHNOLOGIES AND NEW STRUCTURAL MATERIALS IN CHEMICAL ENGINEERING AND INDUSTRY»

The International Seminar «Modern Technologies and New Structural Materials in Chemical Engineering and Industry» was held in Kyiv at the E.O. Paton Electric Welding Institute from 25 till 27 November, 2002. The Seminar was organised by the National Academy of Sciences of Ukraine, Ministry for the Industrial Policy of Ukraine, E.O. Paton Electric Welding Institute, Open Joint Stock Company «UkrNIIkhimmash» and International Association «INTERM» of the PWI. Chairman of the Seminar Organising Committee was Prof. K.A. Yushchenko, Deputy Director of the PWI and Corresponding Member of the NAS of Ukraine, and Deputy Chairman was Dr. Yu.B. Danilov, Deputy Director of the Company «UkrNIIkhimmash».

The Seminar was attended by more than 100 scientists and engineers from research and development institutes and higher education establishments, representatives of chemical engineering and oil-and-gas industry, chemical and oil-and-gas factories, specialists in production and realisation of steels, alloys and parts for chemical and petrochemical engineering and oil-and-gas industry from Ukraine, Germany, Austria and Portugal.

Prof. B.E. Paton, President of the NAS of Ukraine, noted in his welcome address and opening remarks to the Seminar participants that a prolonged depression in economics of Ukraine had a negative effect on the state of chemical, petrochemical and oil-and-gas industries. The major part of basic equipment of chemical engineering facilities exhausted their designed life long ago and need replacement and repair.

The sustained growth of economics of Ukraine observed since 2001 allows the problems associated with renovation and further development of these industries to be solved at a qualitatively new level.

The most important tasks for the immediate future are as follows:

- building of advanced apparatuses and equipment for chemical, petrochemical and oil-and-gas enterprises, making of new materials (steels, alloys etc.), and development of corrosion protection methods;
- development of reliable methods and instruments for evaluation of state of active equipment without interruption of its operation and estimation of residual life of the equipment, as well as new approaches to its repair;
- catching up with the backlog in application of new certified technologies, materials and equipment for preparation and welding production;

Prof. K.A. Yushchenko is making presentation at the Seminar

- development and realisation of new quality assurance systems for manufacture and repair of equipment at a level of international standards;
- increase in number of different levels of welding specialists certified in compliance with international standards;
- reduction of manual labour in manufacture of equipment.

It was gratifying that the Seminar was attended by specialists from all regions of Ukraine, as well as from such known companies as «ThyssenKrupp VDM Austria GmbH» and «Voest Alpine», which are the major manufacturers of high-quality structural materials.

Prof. B.E. Paton thanked all the attendees on behalf of the Organising Committee and wished the Seminar to be a success.

Mr. I.D. Ruchko, Head of Department for Chemical Engineering and Accessories at the Ministry of Industrial Policy of Ukraine, Prof. K.A. Yushchenko and Dr. Yu.B. Danilov in their presentations emphasised an importance of chemical and oil-and-gas industries for further development of industrial potential of Ukraine, its agriculture, social policy of the country to handle the needs of the population and export of products. Basic equipment of the majority of chemical, petrochemical and oil-and-gas enterprises of Ukraine is worn out to a considerable degree. Therefore, the scientific substantiation for application of new materials, building of new advanced equipment, upgrading and repair of existing equipment is an important and pressing problem.

Given below is the list of presentations with their short summaries.

During the Seminar

Dr. Yu.B. Danilov *(Open Joint Stock Company «UkrNIIkhimmash», Kharkiv) «Main trends in development of new structures for chemical and oil-and-gas industry enterprises».* Chemical engineering is the industry characterised by a rapid change of ranges of products and processes. Therefore, its most important task is to make a fundamentally new equipment, upgrade and repair existing facilities, automate processes and equipment, and develop methods to increase corrosion resistance. Ukraine has a sufficient scientific and industrial potential to cope with the above tasks.

Eng. Yu.Ya. Nekhaenko *(Severodonetsk State R&D Institute for Chemical Engineering) «Research and development activities of the Severodonetsk NIIkhimmash in manufacture of apparatuses for chemical enterprises».* The Institute is involved in research and development aimed at elaboration of integrated methods for diagnostics and repair of equipment in operation at chemical and petrochemical enterprises, design and manufacture of vessels and apparatuses with a working pressure of up to 16 MPa.

Dr. G.D. Samojlenko *(Limited-Liability Company «Topol», subsidiary of the Nikopol Yuzhnotrubny Works) «Ranges of the «Topol» Company Products».* The Company produces high-quality seamless welded and centrifugally cast pipes, as well as wire and branch pipes from carbon and low-alloy steels, medium- and high-alloy steels and alloys for chemical and petrochemical industries. The Company has a research base and specialists for development of technologies for manufacture of the above products.

Dr. H. Portisch *(ThyssenKrupp VDM Austria GmbH) «Nickel in a nutshell — the role of nickel in engineering, its production, supply and typical applications».* The Nickel Development Institute, where Dr. Portisch is a staff member, is active in promotion of nickel and nickel-base alloys to industry, publishes «Nickel Magazine», collects and disseminates information on the world production of nickel and nickel-containing alloys, as well as products of these materials. The Institute can provide free publications on this subject matter.

Mr. I. Sajer, G.D. Surguchev *(ThyssenKrupp VDM Austria GmbH) «ThyssenKrupp VDM Austria GmbH — Organisation and Products».* The Company incorporates 300 enterprises and makes about 1 million tons of steel. The Company has melting furnaces of differing capacities, including a 30 ton furnace with $Ar–O_2$ purging and continuously operating ESR furnace. The Company produces carbon steels, stainless steels with a nickel content of more than 25 %, nickel-base alloys, titanium and various parts of the above materials for chemical engineering etc.

Dr. H. Alves *(ThyssenKrupp VDM, Portugal) «High-quality materials of ThyssenKrupp VDM Austria GmbH for chemical engineering».* This division of the Company produces stainless steels of the 304L and 316L grades, alloys 926, 31, 32, C278, C4, C22, C50, B2 and B4 resistant to wet corrosion in chemical production environments. Alloy 31 is resistant to corrosion in sulphuric acid at concentrations of 98–99 and 78–80 % and at different temperatures.

Mr. B. de Bör *(ThyssenKrupp VDM, Germany) «High-temperature materials of Thyssen Krupp VDM Austria GmbH for gas-and-oil, aircraft and power engineering».* Melting, pouring and casting of alloys for application at temperatures of up to 1200 °C are performed in open arc furnaces by the VAR and ESR methods using 20 ton furnace. Alloy 602CA has been recognised the best one for operation in carburisation environments at 800–1200 °C.

Dr. J. Lettner *(Voest Alpine and ThyssenKrupp VDM Austria GmbH) «Hot-rolled bimetal plates for chemical, gas-and-oil and aircraft engineering».* Bimetal billets are produced by the Company using a traditional stack method. The billets are rolled to plates of different thickness at a customer's request. Bimetal is tested to bending and shear. The degree of diffusion of elements in the zone of adhesion of the layers is also checked.

Prof. I.K. Pokhodnya *(E.O. Paton Electric Welding Institute) «Welding consumables. State-of-the-art and development trends».* Arc welding will be dominant among numerous welding processes during the next decades. This area will require development of methods to control structure and properties of welds and joints in order to provide their high strength, ductility and operational reliability. These properties will be achieved through optimisation of systems of alloying of the welds and thermal welding cycles, through finding new methods to control melting and electrode metal transfer processes, as well as processes of penetration of the base metal, solidification of the weld pool and cooling of the welds. New types of efficient equipment will be made, reliable technologies for production of welding consumables will be developed, systems for computer-aided analytical control of production of welding consumables will be built on the basis of the latest physical and chemical analysis methods and instruments. The advanced standards, meeting the European requirements, should be worked out to establish quality assurance systems for production of parts, materials and consumables, as well as for certification of products.

Prof. K.A. Yushchenko *(E.O. Paton Electric Welding Institute) «Current trends in application of new technologies for welding of different grades of steels and bimetals».* In addition to traditional welding processes, there are also new technologies and equipment for arc welding of high-alloy steels

and alloys that found wide application. They include arc welding methods using microplasma, hybrid plasma + laser methods, welding using activators (PA-TIG) and self-shielding flux-cored wires, solid-state joining (friction stir welding, manufacture of structural members by explosion welding). Repair technologies using welding, cladding and spraying show high promise for maintaining performance of chemical and oil-and-gas industry equipment. The PWI is prepared to assist enterprises in mastering both new and traditional technologies.

Prof. K.A. Yushchenko, Dr. Yu.N. Kakhovsky, G.V. Fadeeva, V.I. Samojlenko, *and* ***Dr. A.V. Bulat*** *(E.O. Paton Electric Welding Institute) «New electrodes of the ANB series for welding high-alloy steels and alloys».* The E.O. Paton Electric Welding Institute developed covered electrodes of the ANB-29, ANB-35, ANB-17 and ANB-17U, and ANB-42 (E-07Kh20N9, E-08Kh20N9G2B, E-02Kh19N18G5AM3 and E-04Kh23N24G4M3D3, respectively) grades for welding metal structures for chemical engineering. These electrodes are superior in a combination of their welding-operational properties, technological strength of weld metal and corrosion resistance to the known electrodes — analogues of the OZL-8 (E-07Kh20N9), TsL-11 (E-08Kh20N9G2B), EA-400/10U (E-07Kh19N11M3G2F) and OZL-17U (E-03Kh23N27M3D3G2B) grades.

Dr. V.F. Topolsky *(E.O. Paton Electric Welding Institute) «New processes for welding titanium and its alloys».* The alloy on the base of titanium of the T-110 grade, having strength of 1100 MPa in the annealed state, as well as technology for welding this alloy and a number of other alloys were developed. Proposals for the manufacture of drill pipes from titanium-base alloys for drilling of wells to a depth of 5000 m were substantiated. The PWI offers cooperation in mastering these advanced developments.

Dr. V.A. Anoshin *and* ***Dr. V.M. Ilyushenko*** *(E.O. Paton Electric Welding Institute) «Advanced technologies for welding copper and steel + monel bimetal».* The PWI developed and is applying advanced technologies for welding of copper using paste-like fluxes, in a nitrogen atmosphere, by plasma and ESW methods. Available are the technology and filler metals (wire, electrodes of the ANTs-3M grade) for welding of copper and steel + monel bimetal. Specialists of the PWI are prepared to assist industry in mastering these technologies.

Dr. I.V. Dovbishchenko *(E.O. Paton Electric Welding Institute) «Improvement of corrosion resistance of welded joints on aluminium equipment used to produce concentrated nitric acid».* The PWI developed technologies for improvement of corrosion resistance of welded joints on reaction vessels (autoclaves, bleaching columns, refrigerators, pipelines etc.) and parts made from commercial aluminium AD00 and AD000 of an increased purity by heat treatment. The technology recommended will allow service life of the equipment to be extended.

Dr. E.F. Pereplyotchikov *(E.O. Paton Electric Welding Institute) «Modern technologies and equipment for surfacing and hardening of pipeline valves».* the PWI developed technologies and substantiated the efficiency of using plasma-powder cladding for pipeline valve components. Provided was the information on new powders and equipment for plasma-powder cladding of valves. Examples of their efficient application for production and repair of valves were given.

Prof. A.Ya. Nedoseka, Dr. S.A. Nedoseka, M.A. Yaremenko *(E.O. Paton Electric Welding Institute),* ***A.A. Yolkin, Yu.F. Kurbatov, A.S. Vasiliev*** *(Odessa Port Factory) «Monitoring of technical state of equipment in operation».* Available are the advanced technologies for monitoring of quality of welded joints and estimation of specified service life of equipment. The technologies are based on analysis of the material condition vector (MCV technology). The system for technical diagnostics of equipment of the EMA-3 family was made and introduced into industry.

Prof. S.G. Polyakov *(E.O. Paton Electric Welding Institute) «Scientific principles and hardware for utilisation of electrochemical methods for corrosion monitoring at petrochemical enterprises».* Theory and practice of measurement of corrosion rate by the kinetic and polarisation resistance methods, as well as estimation of the effect of distribution of local corrosion centres on corrosion fracture susceptibility of a workpiece were worked out. Elements of the corrosion-measurement hardware (electrochemical cells, sensors etc.) were made and introduced into production. The developments are recommended for wide application at industrial enterprises.

Dr. V.A. Kachanov *(Open Joint Stock Company «UkrNIIkhimmash») «Investigation of corrosion behaviour of welded joints in new structural materials in aggressive environments».* Investigations were conducted to study corrosion resistance of the ThyssenKrupp VDM alloys 31, 33, 59 and B4, Russian alloys 06KhN28MDT and KhN65MBU and zirconium E-110 in environments containing sulphuric acid, alloys 33 and 06KhN28MDT in titania production processes, as well as alloys 201, 33 and 06KhN28DDT in concentrated caustic environments. Recommendations for their applications were given.

Dr. V.A. Kachanov *(Open Joint Stock Company «UkrNIIkhimmash») «Increase in corrosion cracking resistance of metals in a structure».* Recommendations for heat and hydraulic treatment of welded joints and equipment of steels 09G2S and St.3sp5 were developed and introduced into production in order to increase resistance to corrosion cracking.

Eng. S.V. Nesterenko *and* ***N.G. Yefimenko*** *(Kharkiv State Municipal Academy),* ***Dr. V.A. Kachanov*** *(Open Joint Stock Company «UkrNIIkhimmash») «Effect of REM and their master alloys on corrosion resistance of welds in austenitic steels».* Microalloying of weld metal of the 12Kh18N10T, 07Kh19N11M3, 04Kh18N9, 10Kh20N9G6T types with of 0.0019 and 1.0035 % of yttrium and cerium, respectively, leads to inhibition of corrosion in aggressive environments containing H_2SO_4, HNO_3 and NaOH.

Eng. G.E. Shepil *(Open Joint Stock Company «UkrNIIkhimmash») «Metallography of corrosion*

fractures of high alloys». Metallography of the ThyssenKrupp VDM alloys 31, 33, 59, B4 and 201 and welded joints in environments characteristic of titania and caustic soda production revealed both advantages and disadvantages of each of the above alloys. Certain heat treatment conditions were recommended to improve corrosion properties of welded joints of alloys 31 and 59.

Dr. B.A. Gru *(State R&D Institute «Khimtekhnologiya», Severodonetsk) «Corrosion of steels and alloys in some chemical engineering environments»*. The Institute is active in research on corrosion resistance of steels, alloys and metals in specific aggressive environments characteristic of chemical engineering. It was established, in particular, that in production of potassium saltpetre steel 06KhN28MDT exhibited an acceptable corrosion resistance in an intermediate product of NOCl at a temperature of 20 °C, alloys KhN78T and KhN65MV exhibited an absolute resistance in a mixture of $NOCl + Cl_2 + N_2O_4$ at 20 °C, and the ThyssenKrupp VDM alloy 59 (03Kh22N60M15) was the best in organic chlorine at 600 °C.

Eng. A.I. Kabashny *(Open Joint Stock Company «UkrNIIkhimmash») «Peculiarities of the technology for manufacture of heat exchangers from plates»*. The Company «UkrNIIkhimmash» developed designs and technologies for manufacture of heat exchanging elements of the plate and panel types from plates. Thermal efficiency of such elements is 1.5 times as high as that of tubular elements.

Eng. V.M. Dolinsky, D.G. Ryauzov, V.I. Cheremskaya *(Open Joint Stock Company «UkrNIIkhimmash») «Operation and monitoring of technical equipment with defects in welded joints»*. The Company «UkrNIIkhimmash» developed and successfully verified under industrial conditions the model test method allowing estimation of the residual safe life of chemical engineering structures. To apply this method in industry, it is necessary to revise standards on admissibility and inadmissibility of operation of equipment.

Eng. I.I. Kovalev *(Rubezhansk State Chemical Factory) «Experience in operation, manufacture and repair of ferrosilicide equipment»*.

Eng. A.S. Gerashchenko *(Joint Stock Company «UkrTATnafta», Kremenchug) «Problems of repair, operation, assurance of quality and reliability of petrochemical equipment supplied to Joint Stock Company «UkrTATnafta»*. The Kremenchug Oil Refinery, which is capable of refining 18 million tons of oil per year, has in operation 2977 vessels and apparatuses, 1750 pumps, 124 compressors, 492 tanks, 6600 pipings and 1700 pieces of other service equipment. A wish was expressed that a centralised production of special electrodes with a guaranteed quality for chemical and oil-and-gas enterprises be arranged in Ukraine.

Dr. L.I. Aslamova, Dr. I.M. Kadenko *(T. Shevchenko Kyiv National University) «Training of personnel working with the equipment comprising ionisation radiation sources»*. In Ukraine for the first time the «State Sanitary-Hygienic Rules and Regulations for Radiation Safety while Working with Metal Scrap» were developed. They have been introduced into effect since March 15, 2002. The Kyiv National University arranged courses on the above subject matter for legal and natural entities.

Dr. P.P. Protsenko, Prof. K.A. Yushchenko *(E.O. Paton Electric Welding Institute) «International system for training and certification of specialists involved in welding and related processes»*. The International Training and Certification Centre for training of international-qualification specialists, such as welding engineers, welding technologists, welding specialists, welding practitioners and welding inspectors is functioning on a regular basis at the E.O. Paton Electric Welding Institute.

Eng. V.V. Progolaev *(Open Joint Stock Company «UkrNIIkhimmash»),* ***Dr. G.G. Monko*** *and* ***Dr. L.V. Chekotilo*** *(E.O. Paton Electric Welding Institute) «Development of new standards and specifications for chemical and oil engineering»*. The Company «UkrNIIkhimmash» developed and introduced into effect basic branch standards for chemical, petrochemical and oil-and-gas engineering: GSTU 3-17-191–2000 and GSTU 3-020–2001 (developed with participation of PWI), national standards: DSTU 4003–2000 and DSTU 4026–2001, as well as standards harmonised with EN: DSTU EN 286.1–2002 and DSTU EN 286.2–2002. The standards are available at the Company «UkrNIIkhimmash».

Participating in the round-table discussion were representatives of industrial enterprises, R&D institutes and other organisations: P.P. Elagin, B.P. Kisly, B.Yu. Zhukovsky, L.E. Marchenko, A. Salnikov, A.P. Korop, S.V. Firsov, A.E. Noravsky, I.D. Groisman, I.V. Yavorsky, V.A. Bas, V.A. Barilyuk, T.V. Suprun, V.G. Fartushny and others. The participants expressed an interest in having such seminars held on a regular basis.

In the closing speech K.A. Yushchenko and Yu.D. Danilov thanked representatives of Ukrainian organisations, research and industrial enterprises, as well as specialists and scientists from Austria, Germany and Portugal, the «ThyssenKrupp VDM Austria GmbH», «Voest Alpine» companies and the Nickel Development Institute for participation in the Seminar and support rendered to its holding.

The Seminar demonstrated a high potential of science and industry for further improvement of chemical and oil-and-gas industries of Ukraine.

Dr. L.V. Chekotilo

75th BIRTHDAY ANNIVERSARY OF PROFESSOR Boris A. MOVCHAN

Boris A. Movchan, Professor, academician of the National Academy of Sciences of Ukraine, the outstanding scientist in the field of metals science and electron beam technology is 75.

B.A. Movchan was born on January 9, 1928 in Makeevka village (now Nosovsky region) of Chernigov district. After finishing secondary school in 1946, he entered Taras Shevchenko Kiev State University and graduated from it in 1951 on specialty: physics of metals.

Since 1951 and until now Prof. B. Movchan is working at the E.O. Paton Electric Welding Institute: staff scientist (1951–1960), chief of department of electron beam technology (1960–1994). Since 1994 Prof. Movchan is Director of International Center of Electron Beam Technologies of the E.O. Paton Electric Welding Institute of the NAS of Ukraine.

In 1954 B.A. Movchan defended his thesis for Cand. of Sci. (Eng.), and in 1961 — for Dr. of Sci. (Eng.). In 1978 he was elected Academician of the NAS of Ukraine. At present he is a member of New York Academy of Sciences, Honorable Professor of Pekin University of Aeronautics and Cosmonautics.

The scope of scientific interest is the structure and properties of inorganic materials, electron beam technology and new materials. Results of investigations of structure and properties are generalized in manuscripts: «Radioactive Isotopes in Investigation of Metals» (co-author, 1956), «Microscopic heterogeneity in cast alloys» (author, 1961), «Boundaries of Crystallites in Cast Metals and Alloys» (author, 1969).

Results of investigations of physical-chemical processes of refining, structure and properties of metallic materials are presented in manuscript «Electron Beam Melting and Refining of Metals and Alloys» (co-author, 1973).

Research and practical activity of Prof. Movchan was devoted over the recent decades to the development of theoretical and practical fundamentals of electron beam evaporation and condensation of different materials in vacuum and to creation of new inorganic materials and coatings having unique physical-chemical and service properties which cannot be produced by other known technologies. These were grounds for the creation of a large group of new technological processes and appropriate equipment having no analogues in the world by many characteristics.

On the basis of fundamental investigations under supervision of Prof. Movchan, the industrial technologies of deposition of high-temperature and heat-protective coatings on the gas turbine blades have been developed and implemented in aircraft and ship-building, and also in power machine-building, that make it possible to increase the reliability and efficiency in use of power units. These developments were generalized in manuscript «Heat-Resistant Coatings Deposited in Vacuum» (co-author, 1983).

Prof. Movchan can be named by right the founder of the new scientific school of producing materials and multifunctional coatings using the method of electron beam technology of evaporation and condensation in vacuum (Movchan, B.A. Inorganic materials deposited from vapour phase in vacuum. In: Advanced Materials Science of the XXI Century, 1998).

Prof. Movchan published more than 300 scientific works, 7 manuscripts (co-author in 3 of them), received more than 100 patents. He was a supervisor of 50 works for Cand. of Sci. and 6 works for Dr. of Sci. Prof. Movchan is the member of Committee of State Prizes of Ukraine in the field of science and technology, member of editorial boards of many scientific journals, several scientific councils.

Scientific activity of Prof. Movchan was marked by State Prize of Ukr. SSR in the field of science and technology (1974), Lenin Prize for the work in the field of electron beam technology (1984), Prize of Evgeny Paton of the NAS of Ukraine (1989). He was awarded two Orders of the Red Banner of Labour (1976, 1981), Order of Lenin (1988). He was awarded the order « For services» of III degree by the resolution of President of Ukraine of May 13, 1998 for the personal contribution to the development of national science, consolidation of the scientific-technical potential of Ukraine. He was also awarded with Honorable Diploma of American Vacuum Society (1983, 1988) and Honorable Diploma of Ministry of Aircraft Industry of China (1998).

Professor Boris A. Movchan is full of creative forces in his 75. We sincerely congratulate the hero of an anniversary and wish him strong health, every happiness and further success for the welfare of our Ukraine.

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