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PREPARATION OF TARGETS BASED ON ALUMINIDE SYSTEMS FOR MAGNETRON DEPOSITION OF HEAT-RESISTANT COATINGS

Abstract: The present work offers results of experimental work on obtaining targets based on aluminide systems which composition corresponds to nickel, titanium and cobalt monoaluminides for magnetron sputtering purposed at obtaining intermetallic aluminide coatings on steel substrates. In the course of the process the work on mixing the powders of each system and pressing them on a steel substrate has been conducted, which, being the basis of the target, ensures the tightness of the magnetron seals and the removal of heat at the required level. The blanks obtained in such a manner were sintered in a vacuum induction furnace, as well as on a constructed test installation through hot pressing method. It was demonstrated that sintering in an induction furnace leads to a large porosity of the blanks, that isn't admissible at production of targets. At sintering samples by hot pressing, simultaneous pressing and sintering of powders were achieved, which became the advantage of hot pressing, in which the material is rapidly compacted at relatively low specific pressures. The combination of pressing and sintering in one operation made it possible to obtain samples that were practically nonporous. At the same time sectional targets from aluminum and plates of cobalt, nickel and titanium were obtained. The preparation of such targets was carried out by pouring the aluminum melt into a mold on the bottom of which the plates were laid. It was revealed that sectional targets retain their shape during the sputtering process and are not destroyed. However, they do not allow obtaining coatings of precisely specified composition. The produced targets were sputtered on a magnetron sputtering installation to produce aluminide films on a steel substrate. As a result, high-quality AlNi, AlCo, and AlTi aluminide coatings of uniform thickness, characterized by high adhesion to the substrate were obtained. So the coatings having a uniform, defect-free surface, a homogeneous structure and tightly adhering to the substrate were obtained. The conducted experimental work enabled to determine the optimal methods for obtaining and configuration of targets for magnetron sputtering of coatings based on nickel, cobalt and titanium aluminides. In order to obtain the coatings having a minimum deviation in chemical composition from the set one, it is recommended to use targets sintered from powder mixtures by hot pressing method.

Key words: coating, nickel, titanium, cobalt aluminides, sintered target, magnetron, hot pressing, sputtering

Introduction. Regardless the sufficiently high strength of a number of metals, alloyed steels and alloys due to their low corrosion resistance cannot be used in manufacturing the parts, operating under extreme operating conditions at high temperatures and in corrosive environments. The application of protective coatings on the working surface of products is one of the effective technological ways of increasing their corrosion resistance [1]. Traditional alloyed structural austenitic steels (AISI 300 series) and ferritic grades (AISI 400 series) are characterized by high heat resistance at temperatures up to 500-600 °C, but at higher temperatures rapidly corrode [2].

One of the perspective ways of protecting the surface of steel products from destruction under the influence of elevated or high temperatures is the application of heat-resistant coatings. Due to the formation of a dense oxide film, such coatings are capable to resist chemical corrosion in a dehydrated gaseous medium at high temperatures [3].

Researches on the use of intermetallides of Al-Ni, Al-Ti, Al-Co, Al-Fe, etc. systems increasingly being carried out as materials of refractory coatings [4-11]. Well resistance to oxidation of aluminides of these systems was the reason for recommending them for use as protective coatings on high-tech products of aerospace engineering and power engineering. Continued advancement of the high-temperature coatings properties are particularly associated with the optimization of the chemical composition, the development and development of new technologies for their deposition, and with the use of new methods for improving the structure and properties of both known and newly created coatings.

Heat resistant coatings based on aluminides can be formed in various ways. The most suitable for the deposition of thin films of aluminides on steel substrates are methods of ion-plasma (<10 μm) and vacuum arc spraying (<100 μm). These methods of surface modification are based on the condensation of

a substance from the vapor phase in a vacuum, which ensures the production of coatings characterized by high uniformity and low contamination. For the formation of thick ($> 100 \mu\text{m}$) coatings based on aluminides, the electron-beam surfacing method is promising. At the same time, it is possible to use such methods as: gas-thermal spraying, gas-dynamic spraying, and many others [4-11].

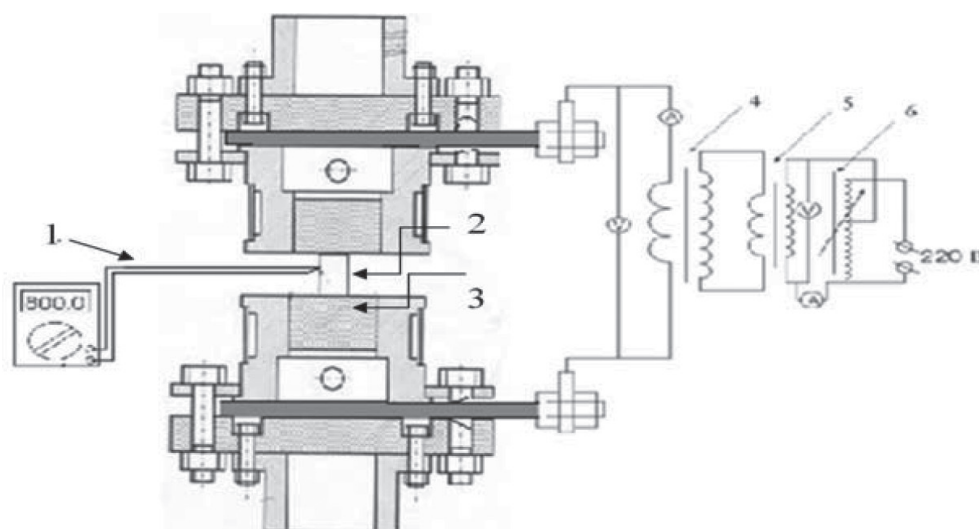
Films obtained by the method of magnetron sputtering have a higher adhesion compared to those deposited by vacuum arc spraying or using other methods of applying alloying exclusion. In magnetron sputtering, it is possible to simultaneously spray two or more different targets, use mosaic (composite or sectional) targets, and use composite targets containing all the necessary components in the desired ratio. For their production methods of powder metallurgy and, in particular, the formation and sintering of a mixture of powders are widely used.

Through the variation of the target composition and substrate the temperature, it is possible to obtain an aluminide films of different chemical and phase composition, and to increase their adhesion due to diffusion mixing at the contact boundary of the substrate or barrier layer and coating [11]. However, in view of the high brittleness of the aluminides, the fabrication from them of targets for magnetron sputtering by fusion is greatly complicated. The production of targets by sintering mixtures of powders of pure metals is associated with such problems as the development of an uncontrolled SHS reaction upon heating to $650 \text{ }^\circ\text{C}$ of Al-Ni and Al-Co powders, swelling of Al-Ti compacts, and the formation of porosity. This leads to the occurrence of a high level of internal stresses, under the influence of which the target is often destroyed at the stage of further processing. The developed porosity does not allow cooling of the target, which is an important requirement in industrial magnetron sputtering plants. In such a manner, in order to increase the use of coatings from aluminides, it

is required to improve the method of manufacturing targets.

Experimental part. Targets of $\varnothing 100$ and a height of 8-9 mm were obtained by two methods: sintering of aluminum powders with nickel, cobalt, titanium and the method of pouring the plates of these metals with aluminum melt.

For obtaining the sintered targets with compositions corresponding to monoaluminides of nickel, titanium, and cobalt, a mixture of powders (wt. %): Ni + 30 % Al, Ti + 41 % Al, Co + 25 % Al have been obtained. Nickel powders of grade PK-1u, nickel PNA-UT1, titanium PTM-1 and aluminum PA-0 were used. Suspended powders of each system were intensively mixed and pressed on a substrate of $\varnothing 100 \text{ mm}$ and a thickness of 5 mm. Substrate of non-magnetic material - steel AISI 304 [5] serves as the base of the target, provides tightness of magnetron sealants and heat removal at the required level. The thickness of the pressed layer from the mixture was 4-5 mm. To ensure adhesion between them, the surface of the substrate was pre-treated with sandblasting and made grooves with a width and depth of $\sim 1 \text{ mm}$. The resulting blanks were sintered in a vacuum induction furnace UIPV-0.001 and by hot pressing on a constructed experimental setup. The installation allows pressing at a pressure of up to 100 tons under conditions of resistive heating of the press matrix. The design is assembled on the basis of the hydraulic press DMY-100 and is equipped with a system for supplying electric current, adjustable voltage to the press mat, has a water-cooled insulated rigging (Figure 1). In order to carry out the pressing in



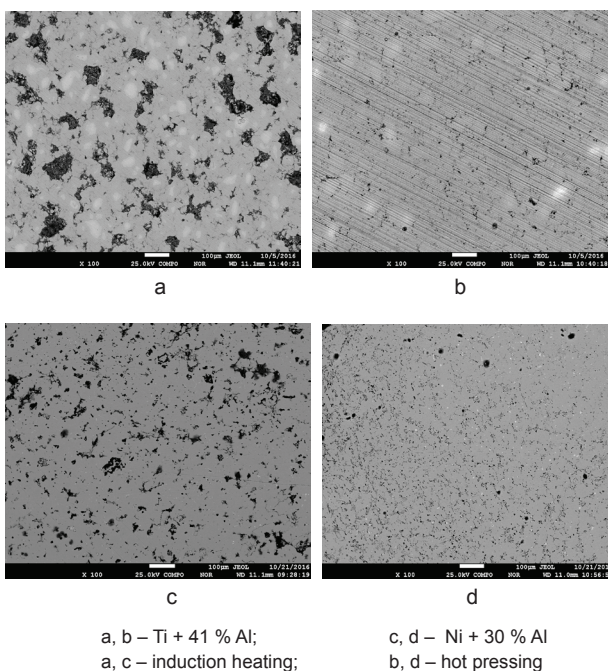
1 – electronic thermometer with thermocouple; 2 – sample; 3 – graphite tab; 4, 5 – power transformer; 6 – autotransformer.

Figure 1 – Attachment for hot pressing on a hydraulic press DMY-100 (under conditions of resistive electrical heating)

these conditions, a press matrix corresponding to the size of the target obtained.

The sintering of the samples in an induction furnace was performed under taper heating conditions necessary for the prevention of SHS reactions development. At the time of the start of the self-heating of the briquettes, we reduced the current supplied to the furnace inductor until the SHS process dissipation. Heating was carried out up to ~ 1000 °C, holding at this temperature for 20 minutes. As a result of the metallographic analysis, it was revealed that the samples obtained under these sintering conditions have a high porosity, which is not permissible in the production of targets.

At the sintering of samples through hot pressing method, the elements of the press matrix were preheated in the furnace to 650 °C. The base of the press matrix was heated to the bottom plate of the press, on top of which graphite disks 10 mm thick were placed, between which a sample of a disc with pressed powder was placed. Then they were pressed with a hot press punch. Pressing was carried out at a constant pressure at a level of 1.5 MPa, with a continuous current transmission of ~ 4000 A. Due to the electrical resistance of the elements of the system, both the matrix press and the sintered disc were heated. In this way, simultaneous pressing and sintering of powders were achieved. Figure 2 shows the microstructures of samples sintered in an induction furnace and hot pressing method.

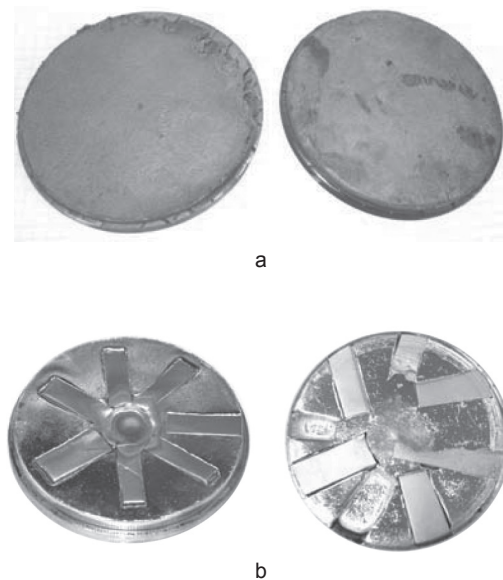


a, b – Ti + 41 % Al;
a, c – induction heating; c, d – Ni + 30 % Al
b, d – hot pressing

Figure 2 – Microstructure of samples of aluminides of titanium and nickel obtained by sintering under various conditions

The porosity of samples sintered in an induction furnace is 10-18 %. The combination of pressing and sintering in one operation made it possible to obtain samples practically non-porous. This circumstance was the merit of hot pressing, in which the material is rapidly compacted at comparatively low specific pressures. The obtained targets layout are demonstrated in Figure 3a.

Sectional targets were obtained from aluminum grade A5N and plates of cobalt grade K0, nickel grade H-0 and titanium grade VT1-0. Plates 3-4 mm thick were cut with a size of 15x30 mm. The preparation of the targets was carried out by pouring the aluminum melt into a mold on the bottom of which the plates were laid, so that in the area of sputtering the targets their area was 50-55 % of the area of aluminum. The sprayed area is a ring of \varnothing 60-90 mm. Samples of targets produced by this method are shown in Figure 3b.



a – targets obtained by hot pressing of powders mixture
Ti + 41 % Al and Ni + 30 % Al on a steel substrate
b – sectional targets obtained by aluminum pouring of Ni and Co plates

Figure 3 – Targets with \varnothing 100 for magnetron sputtering

Sputtering experiments were performed on a magnetron sputtering unit equipped with an ion source APEL-IS-21CELL with an APEL-IS-3500 power source and two APEL-MRE100 magnetrons with an APEL-M-3PDC power source of 3 kW, designed to spray targets \varnothing 100 mm. The substrates for the deposition of aluminide films were cut from AISI304 sheet steel with a size of 20x20x0.5 mm. One side of the substrates turned out to be a mirror, the second was a ground and etched. The steel plates were previously degreased with hexane and weighed.

Microstructures and microanalysis of the coatings were carried out on a microprobe analyzer JXA-8230 (JEOL). The surface structure was studied with magnifications up to 5000 times at an accelerating voltage of 20 kV and an electron beam current of up to 7 nA, microanalysis was carried out at a current of - 20 nA. The phase composition was examined using a D8 Advance diffractometer (BRUKER): CuK α radiation ($\lambda \approx 1.54 \text{ \AA}$); $U = 40 \text{ kV}$, $I = 40 \text{ mA}$; shooting speed 0.1-1 deg/min; angle interval 2θ 4-90° with scanning step 0.01°. X-ray photography was carried out with Bragg-Brentano focusing using a semiconductor detector.

Results and discussion. The obtained experimental materials enables us to conclude that, at the usual cold pressing, and subsequent sintering in the furnace, the target samples had a high porosity and poorly connected with the steel base due to warping caused by the development of the SHS process. This makes it impossible to use such a method in preparing the targets.

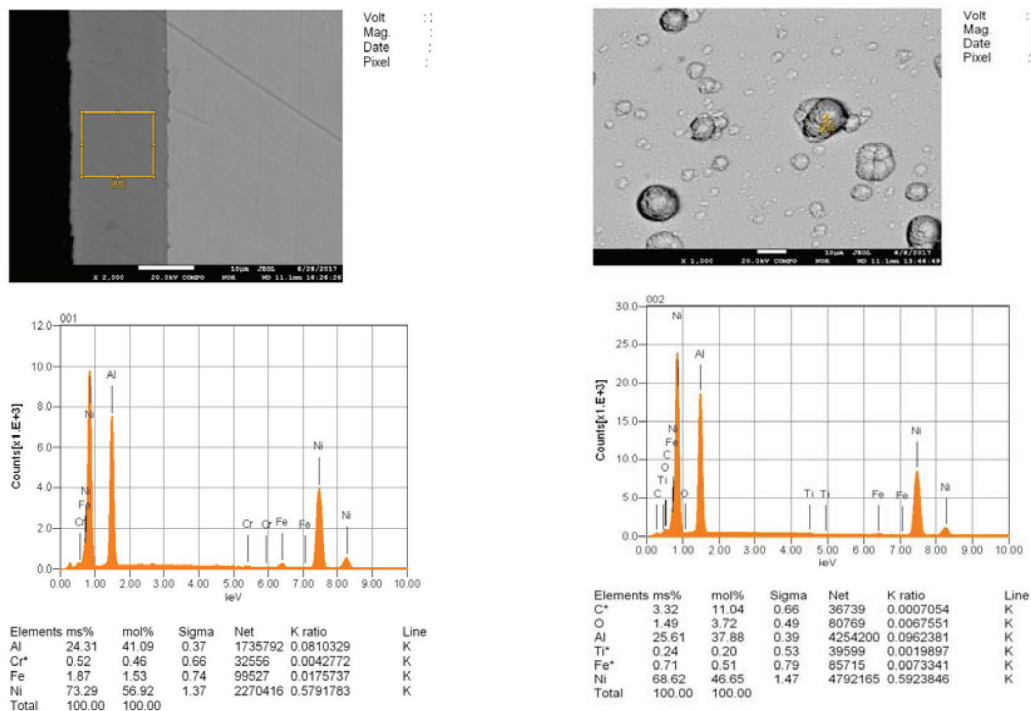
The samples of the targets obtained by the hot pressing method had a flat surface without significant defects. The sintered material was characterized by high adhesion to the steel substrate. Thus, due to the short-term high-temperature action on the powder mixture, the optimum ratio of the heating and deformation modes of the material, and also the peculiarities of the passage

of electric current through the sample, it is possible to obtain materials with low values of porosity and residual mechanical stresses by hot pressing under conditions of resistive heating.

The target samples obtained by casting nickel, cobalt and titanium plates with aluminum melt turned out to be sufficiently dense to provide tightness when directly mounted on a magnetron.

The produced targets by the pouring method and the method of hot pressing were set alternately on magnetrons. Substrates for deposition were mounted in a magnetron sputtering chamber, ion-purification was carried out and then sputtering was carried out. Ionic purification was carried out with argon at an operating voltage of 3.00 kV, a current of 60-80 mA, a pressure of $3 \cdot 10^{-3}$ Torr, and a duration of 20 minutes. It was found that for a coating thickness of $\pm 4, 5 \text{ \mu m}$, the sputtering time should be 1 hour, with a current of 2 A, a voltage of 550 V, and an argon pressure of $1 \cdot 10^{-2}$ mm Hg. Accordingly, to obtain thicker coatings, the spraying time should be proportionally increased. As a result, uniform thicknesses of AlNi, AlCo, and AlTi aluminide coatings were obtained. Externally obtained coatings had a uniform, defect-free surface. Spontaneous peeling of these films was not detected.

The coatings have a homogeneous structure and adhere closely to the substrate (Figures 4a, 5a, 6a).



a – coating cross-section

b – coating surface

Figure 4 – Results of microprobe and electron microscopic analysis of AlNi coating

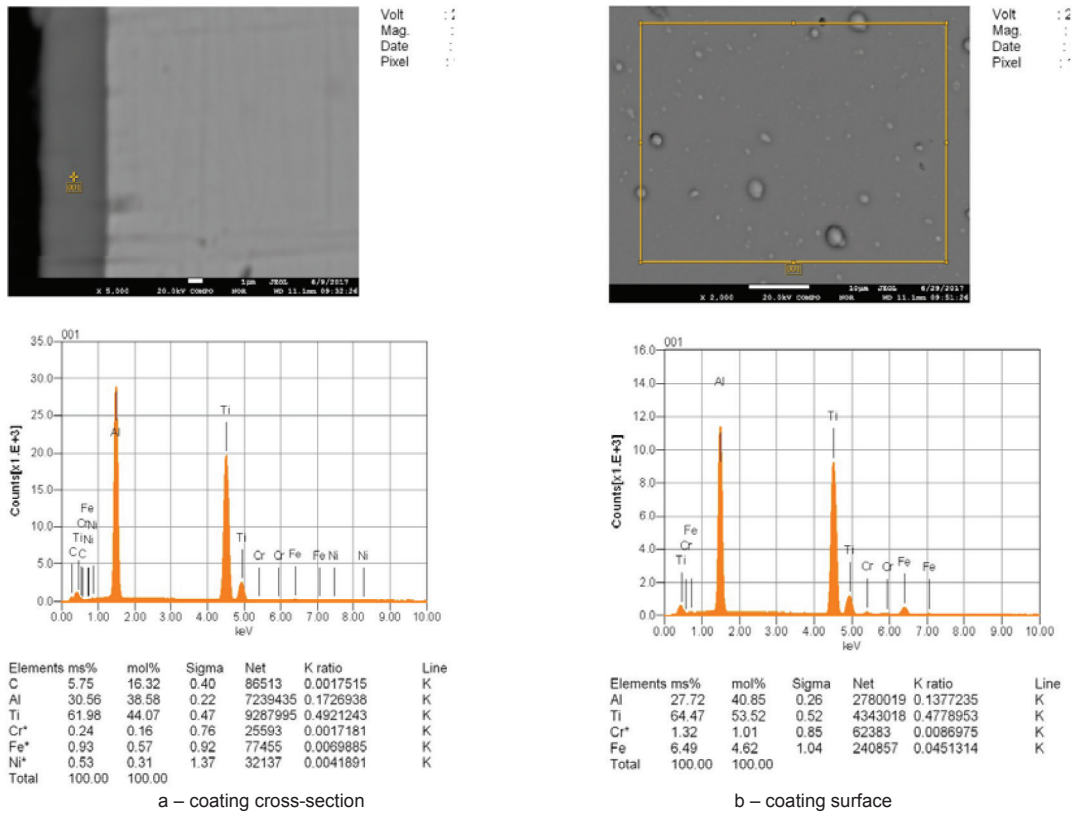


Figure 5 – Results of microprobe and electron microscopic analysis of AlTi coating

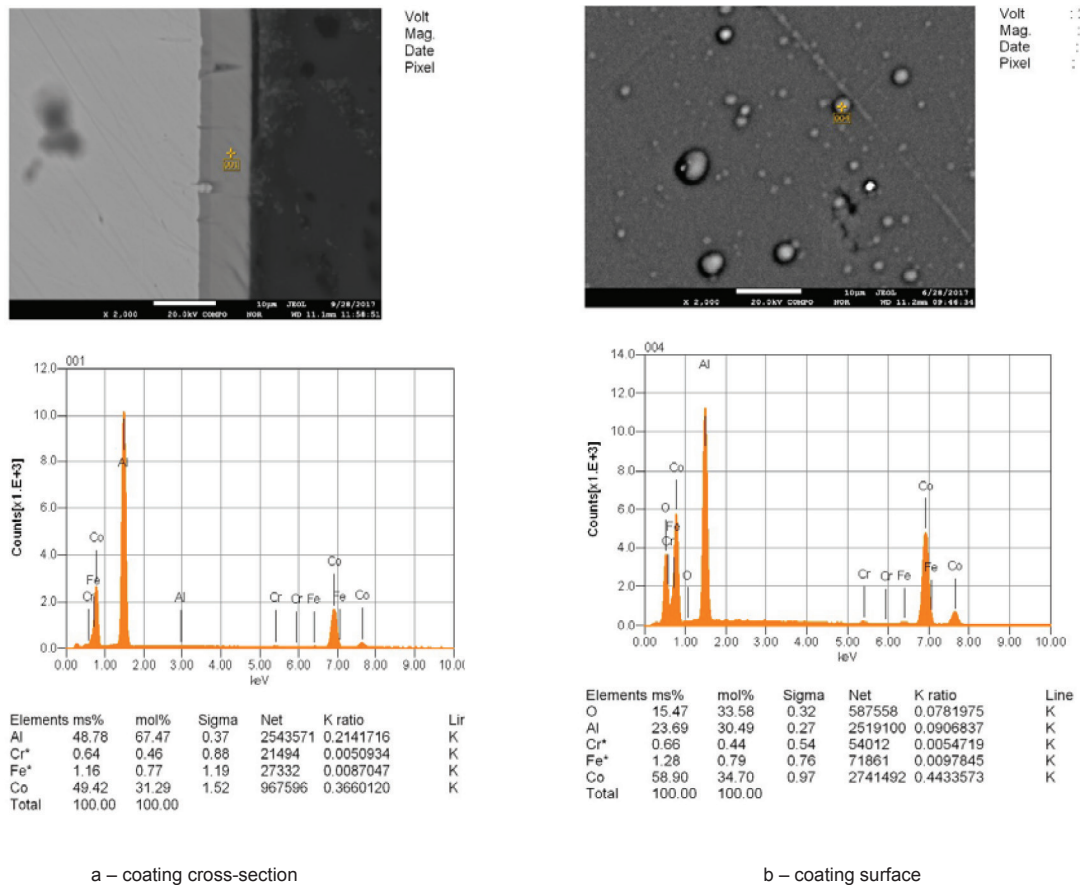


Figure 6 – Results of microprobe and electron microscopic analysis of AlCo coating

Figures 3-5 show the elemental composition, surface structure and cross sections of the coatings obtained on the surface of steel substrates. Their deposition was carried out using targets made by hot pressing. Similar coatings with structure and characteristics with minor deviations in composition ± 4 at % were obtained using sectional targets. The resulting coatings had a nonporous fine-grained structure, which is typical for coatings formed under conditions of a cold substrate (Figures 4b, 5b, 6b).

During the work performance the coatings X-ray phase studies were carried out. The results demonstrated the formation of intermetallide phases AlNi, AlCo and AlTi, formed in accordance with the phase diagrams. The main phases on samples coated with nickel aluminide are NiAl and significant amounts of Al_3Ni_2 . AlTi phases as well as AlTi_2 were detected on samples with Al-Ti coatings. Al (Co), Al_3Co phases were detected on samples with Al-Co coatings.

In the course of the 5-hours sputtering experiment, it was established that the Ni + 30 % Al and Ti + 41 % Al targets obtained by the hot pressing method retain their shape, a uniform groove formed in the spraying region. the Co + 25 % Al target was melted and destroyed (Figure 7) during the sputtering process, which was a consequence of the development of the SHS reaction between the phases in the structure of the sintered material due to its heating. Sectional targets were characterized by stable sputtering throughout the entire experiment.



a – initial

b – after sputtering

Figure 7 – Cobalt aluminide target destruction

Conclusions. The conducted experimental work gave an opportunity to determine the optimal methods of obtaining and configuring targets for magnetron sputtering of coatings based on nickel, cobalt and titanium aluminides. The sectional targets obtained by casting plates from a more refractory alloying material with an aluminum melt are easier to fabricate. Such targets hold their shape and do not destruct during the sputtering process. However, due to the difference in the rates of evaporation of elements and their magnetic properties, for the required composition assurance, it is necessary to select the ratio of the areas of the

sputtered elements on the surface of the target.

For the preparation of coatings having a minimum deviation in chemical composition from the specified, it is advisable to use the targets sintered from powder mixtures. The appropriate method of producing them consist in pressing the powders onto the steel substrate followed by hot pressing. The use of direct resistive heating of compacts to maintain the temperature at a given level makes it possible to substantially simplify the sintering equipment. The sintering should have sufficient duration and temperature in order to enable the mutual diffusion between the components of the powder mixtures to prevent the development of the SHS reaction during magnetron sputtering. Powder technologies gives an opportunity to obtain a diversified compositional variety of multi-component and multiphase materials that are considered to be labor-intensive or impossible to obtain by other methods.

As can be seen from the above, the present work demonstrates the use perspectiveness of powder materials targets prepared by hot pressing method for the precipitation of nickel, cobalt and titanium aluminides coatings by magnetron sputtering.

The present work has been performed within the framework of the grant No. 1196/GF4 “Creation of the fundamentals of the technology for the production of high-strength and heat-resistant intermetallic superalloys on the basis of aluminides by the powder metallurgy method” of the Ministry of Education and Science of the Republic of Kazakhstan.

During the course of the work the equipment of the “National Scientific Laboratory of Collective Use” under the priority direction “Technologies for the hydrocarbon and mining-metallurgical sectors and related service industries” were used.

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ТҮЙІНДЕМЕ

Бұл мақалада, болатты төсенділерінде интерметаллидті алюминидті жабындыларын алу мақсатымен магнетронды тозаңдату арқылы алюминид жүйелердің негізіндегі құрамы никель, титан, кобальт монноалюминидтеріне сәйкес келетіні нысаналарды алуға бағытталған эксперименттік жұмыстар өткізілгені көрсетілді. Әр жүйенің ұнтақтарын араластыру және болат төсендісіне пресстеу жұмыстары өткізілді. Бұл төсенді нысананың негізі болып табылады және жылуды керекті денгейде шығаратындай магнетронның бітеулігін қамтамасыз етуге бағытталған. Осылайша алынған дайындамалар индукциялы пеште және құрастырылған эксперименттік қондырғысында ыстықпен пресстеу арқылы пісіріп-біріктірілді. Нысана үлгілерін индукциялы пеште пісіріп-біріктіру сатылы кезеңмен өткізілгеніне қарамастан үлгілердің кеуектері көп пайда болғаны көрсетілген. Ал ыстықпен пресстеу әдісі өте тиімді екені анықталды, өйткені пресстеу мен пісіру операциялары бір кезеңде өту арқылы үлгілердің кеуектері пайда болмағандықтан ыстықпен пресстеу әдісі тиімді болып табылды. Сонымен қатар секциялы нысаналар алынды. Олар, қалыптағы кобальт, никель, титан тілімдері орналасқан алюминий балқымасымен құйылып алынды. Бұндай нысаналар тозаңдату кезінде өз пішіндерін сақтап қиратылмайтыны көрсетілді. Бірақ олар керекті құрамы болатын жабындыларды алуға мүмкіндік бермейді. Болат төсендісінде алюминид жабындыларын алу үшін жасалған нысаналар магнетронды қондырғысында тозаңдатылды. Нәтижесінде жоғары адгезиясы бар, сапалы, бірқалыпты Al-Ni, Al-Ti, Al-Co алюминидтер жабындалары алынды. Интерметалды фазалары бар болатын алынған жабындылар бір қалыпты құрылымға ие және болат төсендісіне тығыз орналасқан. Өткізілген эксперименттік жұмыстардың арқасында кобальт, никель, титан алюминидтер негізінде жабындыларды алу үшін магнетронды тозаңдатуға нысаналардың оңтайлы алу жолдары көрсетілді. Жабындылардың берілген белгілі құрамы болу үшін ұнтақ қоспасынан ыстықпен пресстеу арқылы алынған нысаналарды қолдану ұсынылады.

Түйінді сөздер: жабындылар, никель, титан, кобальт алюминидтер, нысаналар, магнетрон, ыстықпен пресстеу

РЕЗЮМЕ

Представлены результаты экспериментальных работ по получению мишеней на основе алюминидных систем, состав которых соответствует моноалюминидам никеля, титана и кобальта, для магнетронного напыления с целью получения интерметаллидных алюминидных покрытий на стальных подложках. Проведены работы по смешиванию порошков каждой системы и напрессовки к стальной подложке, которая являясь основой мишени, обеспечивает герметичность уплотнителей магнетрона и отвод тепла на необходимом уровне. Полученные таким образом заготовки спекались в вакуумной индукционной печи, а также путем горячего прессования на сконструированной экспериментальной установке. Показано, что спекание в индукционной печи приводит к большой пористости заготовок, что не допустимо при изготовлении мишеней. При спекании образцов методом горячего прессования достигалось одновременное прессование и спекание порошков, что и стало достоинством горячего прессования, при котором быстро уплотняется материал при сравнительно малых удельных давлениях прессования. Совмещение прессования и спекания в одной операции позволило получить образцы практически беспористыми. Вместе с тем были получены секционные мишени из алюминия и пластин кобальта, никеля и титана. Получение таких мишеней проводили заливкой алюминиевого расплава в форму, на дно которой были выложены пластины. Выявлено, что секционные мишени сохраняют свою форму в процессе напыления и не разрушаются. Однако они не позволяют получать покрытия точно заданного состава. Изготовленные мишени расплывались на установке магнетронного напыления для получения пленок алюминидов на стальной подложке. В результате были получены качественные, равномерные по толщине покрытия алюминидов $AlNi$, $AlCo$ и $AlTi$, характеризующиеся высокой адгезией к подложке. Получены покрытия, имеющие однородную, бездефектную поверхность, однородную структуру и плотно прилегающие к подложке. Проведенные экспериментальные работы позволили определить оптимальные способы получения и конфигурацию мишеней для магнетронного напыления покрытий на основе алюминидов никеля, кобальта и титана. Для получения покрытий, имеющих минимальное отклонение по химическому составу от заданного, рекомендуется использовать мишени, спеченные из порошковых смесей методом горячего прессования.

Ключевые слова: покрытия, алюминиды никеля, титана, кобальта, мишень, магнетрон, горячее прессование, напыление

Received 24.10.2017.