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MAGNETRON SPUTTERING OF PROTECTIVE COATINGS ON PARTS OF SURFACES OF PRODUCTS AND TOOLS USING MULTIELEMENT TARGETS MANUFACTURED USING A NEW TECHNOLOGY

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Recently, the use of magnetron sputtering for the deposition of protective coatings has been intensively developed. The great prospect of using this method is due to the possibility of applying high-entropy coatings, which are used to protect surfaces that are simultaneously exposed to elevated temperatures, aggressive media and various types of wear. However, the use of the method of creating high-entropy coatings greatly hinders the absence in many cases of the required alloys, which have not only a certain qualitative elemental composition, but also a quantitative one. We managed to solve this problem by creating multi-element targets, with the help of which it becomes possible to create not only almost any elemental composition, but also to regulate the quantitative composition of elements. In this paper, we have proposed a method for manufacturing new types of targets for magnetron sputtering, with a detailed description of the technological chain of their manufacture. The novelty lies in the possibility of applying multi-element coatings when using one target. This is due to the fact that the number of different elements in the target can be measured in tens. The paper also presents the obtained positive result of using a target, which included five different metals, during magnetron sputtering.

Keywords: magnetron sputtering, protective coating, multi-element targets, high entropy coatings.

Introduction

The number of the Earth's population is growing rapidly, the number of various mechanisms necessary for people's lives and their comfort is increasing at an even faster pace, while the alloys from which they are made are becoming more and more difficult to manufacture and, accordingly, more expensive.

One of the ways to significantly save expensive metals (titanium, tungsten, molybdenum, nickel, etc.) is to apply protective coatings to parts made from simpler, cheaper alloys. In this case, the thickness of the deposited films usually lies in the range of several microns. Such an insignificant film thickness is quite enough to, for example, significantly improve corrosion resistance, heat and heat resistance, wear resistance, and also reduce the coefficient of friction of parts. Thus, coatings meet many parameters of operational and technological requirements.

There are many ways to deposit protective coatings [1], but the most promising are magnetron sputtering, ion and vacuum arc deposition [2]. These methods have a high degree of ionization, flux density and particle energy. Their wide range of deposited film parameters can be controlled by changing the substrate temperature, working gas pressure, substrate potential, and a number of other technological parameters.

1. Experiment technique

We have chosen the method of magnetron sputtering, which makes it possible to obtain coatings from almost any metals, alloys and semiconductor materials, and also, depending on the composition of the working atmosphere, to obtain films of oxides, nitrides, carbides, etc.

The aim of this work was the simultaneous deposition of a film using a single target consisting of Cr-Ni-Ti-Fe-Cu elements, which makes it possible to obtain a high-entropy coating.

To solve the tasks set, the technological chain given below was used.

At the first stage, the targets for magnetron sputtering were prepared. The metals that make up the future target were purchased pre-ground to an average of 50 microns. They were dosed in equal at.% and placed in the grinding jars of a ball mill. The grinding jars and balls were made from tungsten carbide. The mixture of metals placed in the grinding jars was poured with Nefras C2 - 80/120 gasoline and the ball mill worked for six hours. After grinding, the powder was separated from gasoline and sent for further drying for 4 hours, being in a vacuum hood.

After drying, the powder was sent for examination using a MIRA-3 LMU scanning electron microscope. The studies of the resulting powder using an electron microscope made it possible to find that, firstly, the grain size decreased, from an average of 50 microns to 2 microns, and secondly, the elemental composition of an individual grain after manipulation with a ball mill contains all the elements , which were in the grinding jar, and a good preservation of the proportion of elements (at.%) is recorded.

At the next stage of manufacturing targets for magnetron sputtering, disks 12 mm in diameter and 3 mm thick were created from micropowders pressed using a hydraulic press (20 tons). The small size of the discs was due to insufficient pressure to press the larger discs. After pressing, the discs were sintered in a vacuum furnace at a temperature of 800^0C for two hours and then cooled in a residual vacuum to room temperature. Due to the small size of the discs, a steel matrix (steel 08) was made in the form of a disc 100 mm in diameter and 10 mm thick. Round 3 mm recesses were milled into them with a diameter to fit the dimensions of the manufactured targets, which were then inserted into the matrix on a “hot” one. Fig.1 shows a view of an already working target, which consists of a matrix into which disks are inserted. The composition of the disks consists of Cr-Ni-Ti-Fe-Cu.

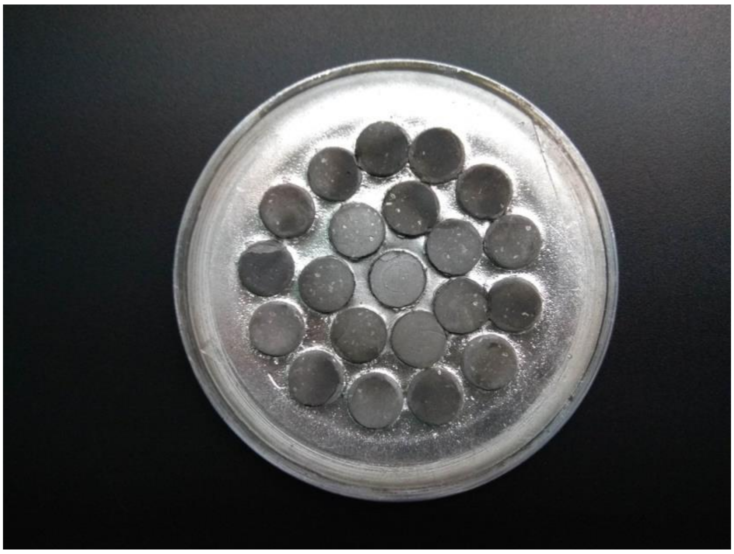

Fig.1. View of the target, consisting of a matrix and 21 disks.

We assume that the technology used to create targets by this method has the advantage of deposition of high-entropy coatings consisting of a large number of elements.

In order to check the possibility of creating disks for a target with a large number of elements, we conducted an experiment to obtain a mixture of 8 elements - Ni-Co-Fe-Ti-Cr-Zr-Mo-W. The choice of refractory metals was necessary to test the possibility of their sintering in vacuum at a relatively low temperature for them - 1000^0C. The manufacture of the disks was successful, they had quite sufficient strength, enough to be inserted into the mounting holes of the matrix.

Thus, it can be expected that the targets may contain several times more elements than five elements, which makes it possible to expand the range of physical and mechanical parameters of the deposited films.

At the final stage, in order to carry out magnetron sputtering of films using a prepared target, substrates were made, on which coatings are applied. In the manufacture of substrates, AISI 321 steel was used. The substrate is a hexagon with edges of 15 mm and a thickness of 5 mm. Both planes of the hexagon were ground on a surface grinder, one, which was subsequently coated, was polished on a polishing machine to a mirror finish, and final polishing took place in an electrolytic-plasma polishing bath. After unloading from

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the bath, the part was washed in an ultrasonic bath and treated with steam using a steam jet device, wiped with coarse calico soaked in ethanol and placed in a drying cabinet and kept in it for two hours at a temperature of 150^0C.

Fig.2. Information - measuring device for determining the coefficient of sliding friction.

Fig.3. Tribometer.

To determine the coefficients of friction, the experimental setup "Information - measuring device for determining the coefficient of sliding friction" was used, the appearance of which is shown in Fig.2, and the tribometer shown in Fig.3 was used to measure wear resistance. Both devices were created by employees Scientific Research Center "Ion-plasma technologies and modern instrumentation" at E.A. Buketov KarU.

2. Results and discussion

The films were deposited in a chamber ion-plasma vacuum unit. NNV-6.6-I1, which in the basic configuration is designed for applying various single-layer and multilayer coatings on a wide range of metal products with a diameter of up to 200 mm and a length of up to 250 mm by the method of substance condensation with ion bombardment.

Instead of one of the three electric arc evaporators, we installed a magnetron, and instead of the second, a plasma source with an incandescent cathode, which makes it possible to clean their surfaces with a stream of ions (argon) by bombarding the substrates placed in the chamber before coating [3].

In the case of coating only one surface, the substrates are fixed and the coating is applied frontally. If it is necessary to apply a coating on the entire surface of the substrate, for example, during a further study of the heat and heat resistance of the sample, the entire surface of the substrates is polished, and the substrates in the installation chamber rotate both around their axis and rotate in a circle, with adjustable speeds of both rotations. The use of sample rotation makes it possible to deposit coatings on complex-shaped parts [4].

Thin films of Cr-Ni-Ti-Fe-Cu + Fe (~5 μm) were deposited on AISI 321 steel substrates by magnetron sputtering in an argon atmosphere in an NNV-6.6I1 vacuum setup. Additional contribution (Fe) of the target to the elemental composition of the coating, in addition to disks, a steel (steel 08) matrix contributed.

Fig.4 shows the spectrum taken from the central part of the disk obtained by the above method using a MIRA-3 LMU scanning electron microscope.

It should be noted that the difference in the elemental composition of the disc and the deposited film may be due to the difference in the "sticking" coefficients of the elements used. As for the “tail” of foreign elements observed in Fig.2, this is due to both the oxidation of the disks and the “dirt” that appeared during their pressing. Finishing cleaning with a filament cathode plasma source cleans both the substrates and the target, which allows you to get rid of the “tail” of foreign elements during coating deposition. In the first series of experiments, the coefficients of friction of disks and applied coatings were measured, since this parameter is important for increasing the service life of the rubbing parts of mechanisms [5]. Fig.5 shows the spectrum taken from the coating on the substrate using a MIRA-3 LMU scanning electron microscope.

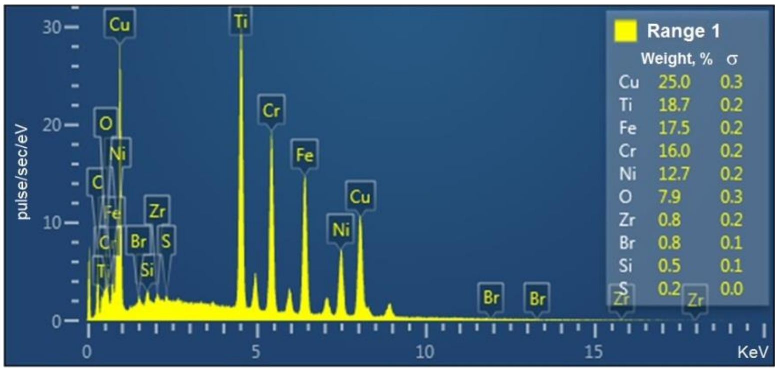

Fig.4. Spectrum taken from the central part of the target disk using a scanning electron microscope.

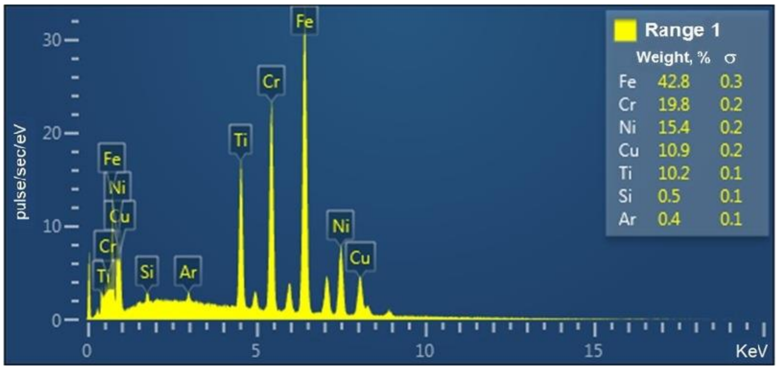

Fig.5. Spectrum taken from a coating on a substrate using a scanning electron microscope

For a more accurate result, measurements were taken 25 times on each metal plate. Copper, AISI 201 steel and aluminum plates were used. The results obtained are shown in table 1. In the second series of experiments, the Vickers microhardness of disks and deposited coatings was measured on an HVS-1000A device [6]. One sample was measured 10 times. The results obtained are shown in table 1. In the final series of experiments, the wear resistance of discs and applied coatings was measured [7].

During the rotation of a ball made of steel with high hardness, the coating wears out, and a spherical hole is formed in the film deposited on the substrate. The number of wells on one sample (20 pcs) was chosen so that the accuracy of the RADWAG AS60/220R2 electronic balance - 0.06 mg - was enough to obtain reliable information about the sample weight loss, and, at the same time, that the depth of the well did not exceed the thickness of the coating layer – 5 μm. In addition, similar measurements were carried out with substrates, the results of which for the measured coefficients of friction, microhardness and wear of AISI 321 steel are also shown in Table 1.

Table 1. The table shows the values of the coefficients of sliding friction, microhardness and abrasion rate of the multi-element disks, coatings and substrates that we have manufactured.

Object	Friction coefficient			Microhardness HV	Wear, μg/s
	on Al	on steel AISI 201	on Cu		
Disk Cr-Ni-Ti-Fe-Cu	0.09±0.006	0.07±0.006	0.11±0.007	580±0.4	0.3±0.07
Coating Cr-Ni-Ti-Fe-Cu+ Fe, on the substrate	0.07±0.003	0.06±0.003	0.08±0.004	790±0.2	0.1±0.06
Substrate AISI 321	0.10±0.003	0.25±0.002	0.39±0.003	363±0.3	0.4 ±0.03

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Conclusion

The results obtained during testing the use of the above technological chain indicate that the applied composite multi-element coating Cr-Ni-Ti-Fe-Cu + Fe has a high microhardness and wear resistance, with relatively low values of friction coefficients. Thus, microhardness and wear resistance are several times higher than similar characteristics of the substrate material, and the coefficient of friction is at least 30% lower, which can increase the service life of machine parts and mechanisms.

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