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Sintered powder high-entropy target cathodes for wear-resistant coatings

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Abstract. Modern machine-building production equipped with high-performance mechatronic systems and numerically-controlled and adaptive control machines for blade cutting of heat-resistant chromium-nickel and titanium alloys requires increasing the operating properties of cutting tools working at high temperature-force loads in the contact zone, respectively with a significant stress-strain state of the cutting wedge. It is possible to solve the problem of increasing wear resistance and serviceability by developing and introducing new tooling material, as well as by applying wear-resistant coatings. The paper presents the results on development of technology for obtaining high-entropy target cathodes by spark plasma sintering with subsequent application of wear-resistant coatings on metal-cutting tools by both magnetron and ion-plasma methods. Samples of sintered high-entropy target cathodes of different compositions (more than fourteen) and at different modes of their sintering (depending on temperature in five modes) with their subsequent optimization and two standard sizes (20 and 80 mm) were obtained for further application of wear-resistant coatings on the magnetron unit. The authors carried out structural and phase analysis and studied physicomechanical properties of the obtained high-entropy target cathodes: density, hardness, electrical conductivity, emissivity. The possibility of obtaining high-entropy target cathodes by spark plasma sintering was confirmed experimentally, and the effect of sintering temperature on structure and properties of the sintered samples of high-entropy target cathodes was established. Dependence of physicomechanical and electrophysical parameters of target cathodes on technological modes of spark plasma sintering is shown.

Keywords: sintered powder high-entropy target cathodes, spark plasma sintering, composition, hardness, electrical conductivity, density, structural-phase composition

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Спеченные порошковые высокоэнтропийные катоды-мишени для износостойких покрытий

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Аннотация. Современное машиностроительное производство, оснащенное высокопроизводительными мехатронными системами и станками с числовым программным и адаптивным управлением для лезвийной обработки резанием жаропрочных хромоникелевых и титановых сплавов, требует повышения эксплуатационных свойств режущего инструмента, работающего при высоких температурно-силовых нагрузках в контактной зоне и, соответственно, при существенном напряженно-деформированном состоянии режущего клина. Решение вопроса повышения износостойкости и работоспособности возможно как путем разработки и внедрения нового инструментального материала, так и применения износостойких покрытий. В работе представлены результаты по разработке технологии получения высокоэнтропийных катодов-мишеней путем искрового плазменного спекания с последующим нанесением износостойких покрытий на металлорежущий инструмент магнетронным и ионно-плазменными методами. Получены образцы спеченных высокоэнтропийных катодов-мишеней различных по составу композиций (более четырнадцати) при разных режимах спекания (в зависимости от температуры в пяти режимах) с последующей их оптимизацией, а также двух типоразмеров (20 и 80 мм) для дальнейшего использования для нанесения износостойких покрытий на магнетронной установке. Проведены структурный и фазовый анализы, а также исследование физико-механических свойств полученных высокоэнтропийных катодов-мишеней: плотности, твердости, электропроводности, эмиссионной способности. Экспериментально подтверждена возможность получения высокоэнтропийных катодов-мишеней методом искрового плазменного спекания, при этом показано влияние температуры спекания на структуру и свойства спеченных образцов высокоэнтропийных катодов-мишеней. Установлены зависимости физико-механических и электрофизических параметров катодов-мишеней от технологических режимов процесса искрового плазменного спекания.

Ключевые слова: спеченные порошковые высокоэнтропийные катоды-мишени, искровое плазменное спекание, композиция, твердость, электропроводность, плотность, структурно-фазовый состав

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Introduction

Today, manufacturers are dedicated to enhancing the durability and reliability of their products because it directly impacts efficiency and market competitiveness. One effective approach to achieve this is by improving the metal-cutting process. Even in the case of advanced high-performance NC machine tools, the cutting tool often becomes the bottleneck. It constrains both the machine tool's capability and the quality of the machined surface. Conventional wear-resistant coatings for cutting tools, deposited using one to four solid cathodes, no longer meet current requirements. Such coatings are indispensable for high-speed machining, tools operating under elevated temperatures and loads in the contact zone, and the machining of high-strength, heat-resistant Cr–Ni and Ti alloys.

Additionally, advanced power metallurgy processes have garnered significant attention [1 – 3] because they facilitate the manufacturing of highly intricate components with improved performance [4; 5].

The objective of this study is to develop a powder spark plasma sintering process and assess the properties of multi-component wear-resistant coatings applied to metal cutting tools, along with evaluating the service life of the target cathode.

High-entropy alloys, known for their high-temperature resistance, wear resistance, hardness, and strength [6 – 9], are of considerable interest. The properties of such coatings are contingent on the elemental composition and deposition process, which necessitate optimization. Another crucial factor is the characteristics of high-entropy target cathodes [10; 11] employed in depositing wear-resistant coatings on cutting tools.

High-entropy alloys have been the subject of intensive research [12 – 15]. They possess distinctive features, including:

– elevated entropy of mixing (S_{mix}) compared to conventional multi-component materials [16];

– unique mechanical properties resulting from specific thermal processes, atom diffusion under certain structural and phase [17];

– significant influence on the crystal lattice due to the content of iron, nickel, molybdenum, aluminum, among other elements;

– binary or ternary phase diagrams are used to estimate the phase composition [18];

– while alloying elements initiate solid solution hardening and discrete phase separation [19];

– as a result, these alloys are classified as a special category [20].

As mentioned in [5; 9; 15], certain high-entropy alloys exhibit exceptional properties, including hardness, heat resistance, thermal stability, corrosion resistance, and wear resistance.

Materials and Methods

Casting and melting are the primary processes employed in the production of high-entropy alloys [20]. However, powder sintering, particularly spark plasma sintering (SPS), holds significant promise as well [6; 7; 12]. The testing process was divided into two phases, conducted at the Spark Plasma Sintering Lab, Center for New Materials and Technologies, National Engineering Center, STANKIN University. In Phase 1, our focus was on the production of high-entropy target cathodes, while in Phase 2, we deposited the coatings and conducted tests to assess their properties. The results of Phase 1, which involved the manufacturing of powder high-entropy cathodes, are summarized below.

In order to determine the optimal powder composition for spark plasma sintering of the target cathodes, we conducted an analysis of a wear-resistant mixture comprising more than 12 commercially available metal powders. After establishing the quantitative and qualitative composition based on the Yum-Roseri rule, we followed these steps:

– preparation of the powder mixture with a specified particle size distribution;

– pre-compaction of the powder mixture in a mold using a 3851 Manual BenchTOP 12 manual hydraulic lab press (Carver, USA);

– utilization of a A KCE-FCT-H-HP-D25-SD furnace (FCT, Germany) for spark plasma sintering, with variations ranging from 500 to 1600 °C in 50 °C increments;

– application of compaction forces at 25, 50, 80, and 100 kN;

– adjustment of the heating rate to either 50 or 100 °C/min;

– implementation of holding times at 1, 2, 3, 4, and 5 min;

– usage of sample sizes measuring 20 and 80 mm.

Following this, we conducted an examination of the properties exhibited by the resulting target cathodes, employing the following methodologies:

– measurement of density using the hydrostatic technique;

– utilization of a Wilson Rockwell 574 hardness tester (Germany) to measure hardness;

– estimation of electrical conductivity through the phase-sensitive eddy current method using a portable Fischer SIGMASCOPE instrument (Helmut Fischer GmbH+Co.KG, Germany);

– application of scanning electron microscopy (SEM) to determine the elemental, qualitative, and quantitative compositions of the samples. For this purpose, we utilized a Phenom ProX microscope (Netherlands) and a Difray 401K desktop *X*-ray diffractometer (Russia).

Results and Discussion

The manufactured samples of the high-entropy target cathodes, with compositions including:

– $Al_{20}-Ti_{20}-Zr_{15}-V_{15}-Cr_{15}-Nb_{15}$;
– $Al_{20}-Hf_{15}-Mo_{15}-Co_{15}-Ta_{10}-W_{10}-Zr_{15}$;
– $Al_{20}-Hf_{15}-V_{15}-Cr_{15}-Ti_{15}-Ta_{10}-W_{10}$;
– $Al_{20}-Hf_{10}-Ni_{15}-Ti_{25}-W_{10}-Zr_{20}$;
– $Mo_{20}-Nb_{20}-Ni_{20}-Ta_{20}-W_{20}$;
– $Nb_{20}-Hf_{20}-Ti_{20}-Zr_{20}-Ta_{20}$

and others, subjected to testing for their suitability in depositing nano-coatings on cutting tools. The key properties of these target cathodes, aside from the particle size distribution of the powder mixture, encompass density, hardness, electrical conductivity, as well as elemental, qualitative, and quantitative compositions (refer to the table). The test results reveal that higher sintering temperatures lead to increased relative density, hardness, and electrical conductivity.

Conclusion

Based on the test results, we successfully developed a spark plasma sintering process for the production of high-entropy target cathodes, essential for depositing wear-resistant coatings on cutting tools. Through our research, we pinpointed the optimal compositions and sintering conditions necessary to achieve the desired coating structure, as well as the desired physical and

Results under sintering conditions: sintering temperature 1200 °C; pressing pressure 25 kN; holding time 1 min; heating rate 100 °C/min; sample diameter 20 mm

Результаты инсытаний при режимах спекания: температура спекания 1200 °С; давление прессования 25 кН; время выдержки 1 мин; скорость нагрева 100 °С/мин; диаметр образца 20 мм

Density, g/cm^3	Hardness, HB	Electrical conductivity, MSm/m^2	SEM			
			element	atomic number	content, %	
					at.	wt.
$Al_{20}-Ti_{20}-Zr_{15}-V_{15}-Cr_{15}-Nb_{15}$						
8.60	104.6	0.67 – 0.74	Ti	22	29.71	32.28
			Al	13	43.64	26.73
			Zr	40	10.71	22.18
			Cr	24	15.93	18.80
$Al_{20}-Hf_{15}-Mo_{15}-Co_{15}-Ta_{10}-W_{10}-Zr_{15}$						
9.99	110.2	0.64 – 1.20	Mo	42	26.50	26.07
			Co	27	26.12	15.78
			Zr	40	14.36	13.43
			Hf	72	10.38	19.00
			W	74	9.53	17.96
			Br	35	8.84	7.24
			C	6	4.27	0.53

mechanical properties. Our findings confirm the feasibility of employing spark plasma sintering for the creation high-entropy target cathodes. Furthermore, we identified the influence of sintering temperature on the structure and properties of high-entropy target cathode samples, as well as the interrelationships between the physical and mechanical properties of these samples and the variables of the SPS process. The introduction of target cathodes crafted through this process promises to enhance high-speed machining efficiency when utilizing cutting tools with wear-resistant coatings. Such tools have the potential to form secondary and diamond-like structures within the contact zone, leading to the adaptation and self-organization of friction and wear processes.

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Contribution of the Authors

S. N. Grigor'yev – statement and solution of the problem of increasing the wear resistance of metal-cutting tools by developing the technology of obtaining high-entropy target cathodes for application of innovative wear-resistant coatings.

M. Sh. Migranov – theoretical and experimental analysis of methods for obtaining high-entropy target cathodes, justification of choice and modes of spark plasma sintering of high-entropy target cathodes, formulation of conclusions.

M. A. Volosova – development of experimental research methods, selection of necessary equipment for full-scale tests.

A. S. Gusev – performance of experimental tests, processing of the results.

Вклад авторов

С. Н. Григорьев – постановка и решение задачи по повышению износоустойчивости металлорежущего инструмента путем разработки технологии получения высокоэнтропийных катодов-мишеней для нанесения инновационных износостойких покрытий.

М. Ш. Мигранов – теоретико-экспериментальный анализ методов получения высокоэнтропийных катодов-мишеней, обоснование выбора и режимов искрового плазменного спекания высокоэнтропийных катодов-мишеней, формулирование выводов по результатам исследований.

М. А. Волосова – разработка методик экспериментальных исследований, подбор необходимого оборудования для натурных испытаний.

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