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DEVELOPMENT FOR PLASMA SPRAYING OF COMPOSITE MATERIAL BASED ON OXIDE CERAMICS

The results of a study of powders of the following composition are presented: $Al_2O_3+30\%TiO_2+12.5\%MoS_2$; $Al_2O_3+30\%TiO_2+12.5\%CaF_2$. To obtain these compositions, the following dispersed materials were used: titanium PTM grade, aluminum oxide EBM-40, molybdenum disulfide MoS_2 or calcium fluoride CaF_2 . The synthesis of the compositions was carried out in a reactor in the self-propagation mode without supplying energy from an external source, in a nitrogen-oxygen environment with an oxygen content of 10 to 25 wt.% and a pressure of 0.1-0.9 MPa, which is necessary for the powder oxidation reaction titanium. For spheroidization, particles of the resulting composite powder were introduced into a plasma jet and sprayed into a steel cylinder 1 m long filled with argon. The coatings were applied using an APS air plasma spraying installation from Plasma-Tekhnik AG. The disadvantage of carbide ceramics with solid lubricant inclusions is high thermal dissociation during plasma spraying. During flight in a plasma jet, carbide ceramic particles are prone to loss of stability with a change in chemical composition, which leads to a high coefficient of friction for coatings and negatively affects their performance properties. A composite ceramic material based on oxide ceramics with the addition of a solid lubricant, obtained by the method of self-propagating high-temperature synthesis, has good technological characteristics, is resistant to maintaining the chemical composition during plasma spraying and is capable of forming coatings with high wear resistance and low coefficient friction.

Keywords: composite material, use of SHS powders, oxide ceramics, plasma spraying, coatings, durability and reliability, heating rate of powder parts.

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РАЗРАБОТКА ДЛЯ ПЛАЗМЕННОГО НАПЫЛЕНИЯ КОМПОЗИЦИОННОГО МАТЕРИАЛА НА ОСНОВЕ ОКСИДНОЙ КЕРАМИКИ

Приведены результаты исследования порошков следующего состава: $Al_2O_3+30\%TiO_2+12,5\%MoS_2$; $Al_2O_3+30\%TiO_2+12,5\%CaF_2$. Для получения указанных композиций использовались следующие дисперсные материалы: титан марки ПТМ, оксид алюминия марки ЭБМ-40, дисульфид молибдена MoS_2 или фтористый кальций CaF_2 .

Синтез композиций осуществлялся в реакторе в режиме самораспространения без подвода энергии от внешнего источника, в азотно-кислородной среде при содержании кислорода от 10 до 25 мас.% и давлении 0,1-0,9 МПа, которая необходима для проведения реакции окисления порошка титана. Для сфероидизации частицы полученного композиционного порошка вводили в плазменную струю и производили их распыление в стальной цилиндр, длиной 1 м, заполненный аргоном. Покрытия наносили на установке плазменного напыления на воздухе APS фирмы "Плазма-Техник АГ". Недостатком карбидной керамики с включениями твердой смазки, является высокая термическая диссоциация в процессе плазменного напыления. В период полета в плазменной струе частицы карбидной керамики склонны к потере устойчивости с изменением химического состава, что приводит к получению высокого коэффициента трения покрытий и негативно отражается на их эксплуатационных свойствах. Композиционный керамический материал на основе оксидной керамики с добавлением твердой смазки, полученный методом самораспространяющегося высокотемпературного синтеза, обладает хорошими технологическими характери-

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стиками, устойчив к поддержанию химического состава в процессе плазменного напыления и способен формировать покрытия с высокой износостойкостью и низким коэффициентом трения.

Ключевые слова: композиционный материал, применение СВС – порошков, оксидная керамика, плазменное напыления, покрытия, долговечность и надежность, скорость нагрева порошковых части.

1. Introduction

Containing solid lubricants for application to wear surfaces of parts by plasma spraying and subsequent treatment with highly concentrated energy flows, should provide regulation of structure formation and the production of wear-resistant surface layers of coatings in combination with increased cohesive and adhesive strength of the sawn material. To do this, when developing the scientific foundations and technological principles of applying such coatings, the processes and mechanism of synthesis of initial composite powders, the processes and mechanism of formation of coatings during plasma spraying of compositions and subsequent exposure to highly concentrated energy flows should be taken into account [1-3]. The mechanism for strengthening coatings sawn and treated with highly concentrated energy flows should take into account the possibility of regulating the fine structure of the applied compositions, including for the production of amorphous phases. Based on the results of the research, it is necessary to optimize the technological parameters for applying the coatings being developed. When studying the processes of friction and wear of coatings obtained using optimal technology, their antifriction properties and wear resistance under friction conditions with marginal (imperfect) lubrication or in the absence of a lubricant, at increased contact loads and temperatures, should be studied. The mentioned friction conditions correspond to the operating modes of a wide range of tribocouplings (heavy-loaded friction pairs of internal combustion engines, pumps, metallurgical and other equipment). Successful preliminary experiments on the synthesis of composite powders with solid lubricant inclusions show that there is a fundamental possibility of obtaining such powders with appropriate development of SHS technology [4-6]. As noted, this opens up prospects for the effective modification of friction surfaces with solid lubricant components during gas-thermal spraying of the mentioned powder materials. Pulsed plasma effects on self-lubricating sprayed coatings contribute to increasing the efficiency of modification and additionally improving the tribological properties of friction surfaces. This statement is based on the existing prerequisites for the strengthening of coatings when exposed to highly concentrated energy flows.

2. Technology for producing a composite material based on oxide ceramics with solid lubricant inclusions

To test the possibility of obtaining SHS powders with solid lubricant components, the synthesis of compositions containing MoS₂ and CaF₂ was carried out. Research was carried out on powders of the following composition: Al₂O₃+30%TiO₂+12.5%MoS₂; Al₂O₃+30%TiO₂+12.5%CaF₂. To obtain these compositions, the following dispersed materials were used: titanium grade PTM, aluminum oxide grade EBM-40, molybdenum disulfide MoS₂ or calcium fluoride CaF₂, taken in a ratio of 57.5:30:12.5. Mixing of the starting components was carried out in a ball mill with a ball to charge ratio of 25:1 and a mechanical activation time of 2-4 hours. The synthesis of the compositions was carried out in a reactor in self-propagation mode without supplying energy from an external source, in a nitrogen-oxygen environment with an oxygen content of 10 to 25 wt.% and a pressure of 0.1-0.9 MPa, which is necessary for the powder oxidation reaction titanium. The reactor is equipped with current leads with a tungsten helix to initiate the process. The use of a nitrogen-oxygen environment during synthesis with an oxygen content of 10 to 25 wt.% is necessary to carry out the oxidation reaction of titanium powder. When the oxygen content is less than 10 wt.% and

the pressure is less than 0.1 MPa, complete oxidation of the powder and sufficient penetration of the reagent gas into the charge layer do not occur. When the oxygen content is more than 25 wt.% and the pressure is more than 0.9 MPa, the combustion temperature rises so much that the particles melt and form a barrier layer that prevents the penetration of the reagent gas into the charge layer. The synthesis products were crushed in a jaw crusher to obtain a fraction of 0.050-0.063 mm, which ensures the spraying of oxide ceramic coatings with a maximum utilization rate of the material. The operation of thermochemical treatment in an air environment at a temperature of 500-800° C for 4-6 hours is carried out in order to oxidize the particles of the composite powder. At a temperature of thermochemical treatment of less than 500° C and a time of less than 4 hours, complete oxidation of particles does not occur, and carrying out the operation at a temperature of more than 800° C and a time of more than 5 hours leads to an increase in energy consumption and partial sintering of particles among themselves [7]. For spheroidization, particles of the resulting composite powder were introduced into a plasma jet and sprayed into a 1 m long steel cylinder filled with argon. The degree of spheroidization was determined by the form factor of the particles (degree of non-sphericity, value 1 - corresponds to a sphere) by the method of optical metallography. The power of the plasma jet was varied from 30 to 40 kW. With a plasma jet power of less than 30 kW, more than 50% of the particles had a form factor of less than 0.7; with a plasma jet power of 40 kW, more than 90% of the particles had a form factor of 0.9 - 1. When the plasma jet power increased above 40 kW, the increase The particle form factor is insignificant. Applying a thin-film metal shell to Al₂O₃+30%TiO₂+12.5%MoS₂ powder particles; Al₂O₃+30%TiO₂+12.5%CaF₂. carried out by chemical nickel plating. When performing the main operation, a solution of the following composition was used: nickel chloride – 28 g/l; sodium hypophosphite - 30 g/l; sodium citrate - 10 g/l; acetic acid – 10 ml/l. The solution temperature was maintained within 363 – 368 K, pH was 9.0 – 9.5. To obtain a uniform coating of particles, the solution with the powder in the bath was subjected to forced mixing. the optimal duration of nickel plating is 10-12 hours. During this time, a thin-film shell of Ni with a thickness of 6-7 microns is formed on the particles, which is necessary and sufficient according to the accepted criterion for optimizing the cladding of powders for plasma spraying of coatings. Analysis of particle sizes, shape and surface topography of the synthesized composite powders was carried out using scanning electron microscopy (SEM). The study of the structure of particles of composite powders was carried out by metallographic analysis of thin sections of their cross section. For this purpose, Unimet (Japan) and MeF-3 (Austria) microscopes were used.

3. Application of wear-resistant coatings

The coatings were applied using an APS air plasma spraying installation from Plasma-Tekhnik AG under the conditions given in Table 1 and figure 1-4. Friction and wear tests were carried out on an SMTs-2 friction machine according to the scheme: coated pads - cast iron roller (SCh24-44), pads coated with nitrided steel (38XVA). Tribological tests were carried out under conditions of friction with lubricant and in the absence of lubricant at a load in a friction pair of 5 MPa for 10 hours. Data on the parameters of friction and wear of coatings made from powders obtained according to the prototype and the claimed invention are given in Table 2. The results of metallographic analysis were also taken into account when studying the size and shape of particles.

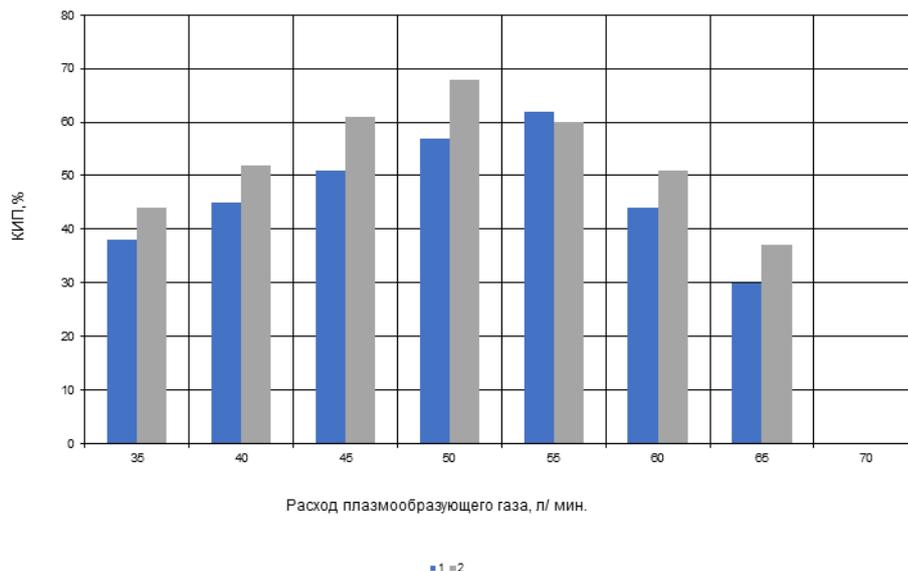


Figure 1. Dependence of the KIP, % on the spraying distance L, mm for powders Al₂O₃- 30% TiO₂-12.5% MoS₂ (1 - with a fraction of 63...100 μm; 2 - with a fraction of 40...63 μm; I=450 A, RN=45 l/min, R por.=3.5 kg/hour)

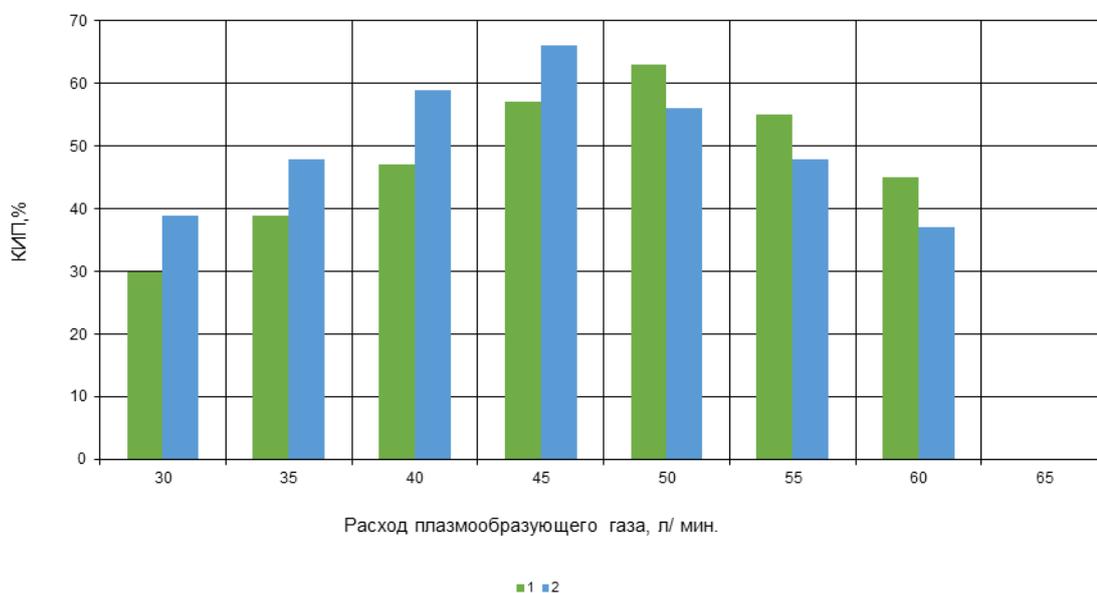


Figure 2. Dependence of the instrumentation and control, % on the consumption of plasma-forming gas N₂ for powders Al₂O₃-30% TiO₂-12.5% MoS₂ (1 - with a fraction of 63...100 μm; 2 - with a fraction of 40...63 μm; L=110 mm; I=500 A; R por.=3.0 kg/hour)

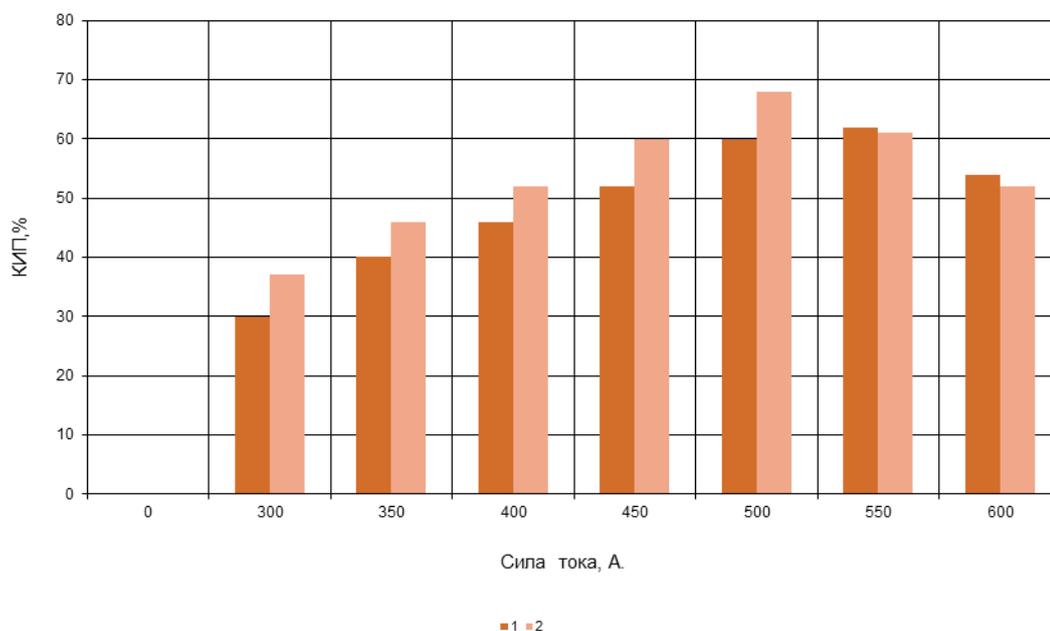


Figure 3. Dependence of the KIP, % on the electric arc current I, A for powders Al₂O₃-30%TiO₂-12.5%MoS₂ (1 - with a fraction of 63...100 μm; 2 - with a fraction of 40...63 μm; L=90 mm; RN=50 l/min, R por.=4.5 kg/hour)

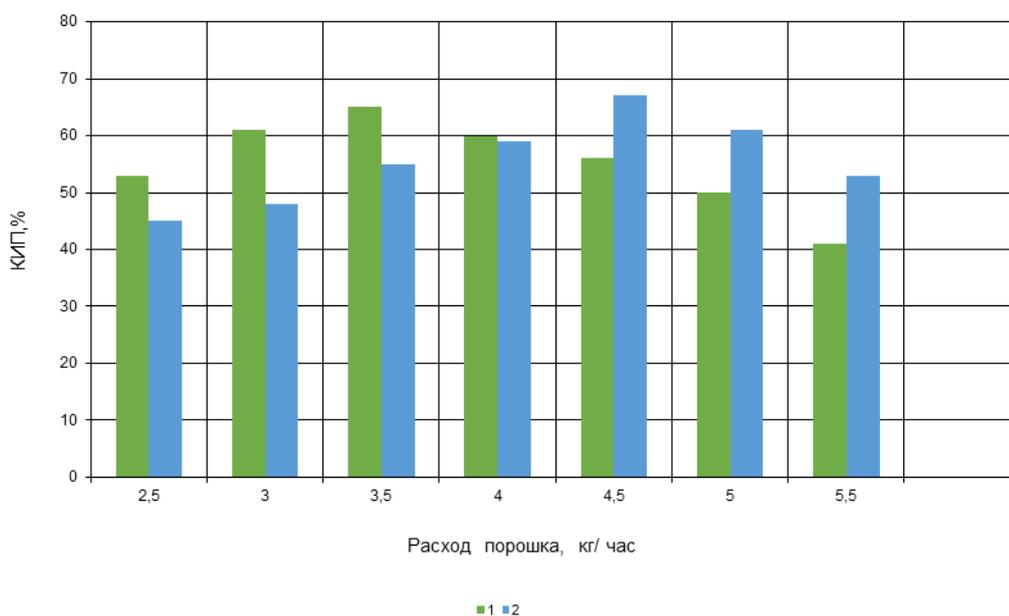


Figure 4. Dependence of the KIP, % on the powder consumption Rп, kg/hour for oxide powders (1 - Al₂O₃-30% Cr₂O₃-12.5%MoS₂; 2 - Al₂O₃-25%TiO₂-25%Cr₂O₃-12.5%MoS₂; L=100 mm; I=500 A; RN=50 l/min)

Table 1. Plasma spraying modes

Sprayed material	Spray modes				
	Plasma-tron arc current, A	Arc voltage, V	Sputtering distance, mm	Consumption of plasma-forming gas-hydrogen, l/min	Consumption of sprayed powder, kg/hour
Ni80Cr20+12,5%MoS ₂ +40%TiC	400	75	120	10	3,0
Ni80Cr20+12,5%CaF ₂ +40%TiC	400	75	120	10	3,0
Al ₂ O ₃ +30%TiO ₂ +12,5%MoS ₂	450	80	110	12	3,5
Al ₂ O ₃ +30%TiO ₂ +12,5%CaF ₂	450	80	110	12	3,5

Consumption of plasma-forming gas - argon 50 l/min

According to metallographic analysis data, the main components of composite particles are oxide phases and solid lubricant inclusions in the form of molybdenum disulfide or calcium fluoride (Figure 5a). The presence of the mentioned constituent powder particles creates the prerequisites for obtaining wear-resistant coatings from them, which are effective against molecular-mechanical and abrasive wear under unfavorable friction conditions (boundary lubrication or lack of lubricant, increased temperature effects) [8, 9]. The powders under consideration are characterized by a complex geometric shape and a developed surface relief of the particles. The tendency to form lumps reduces the flowability of powder materials and their manufacturability during plasma spraying of coatings.

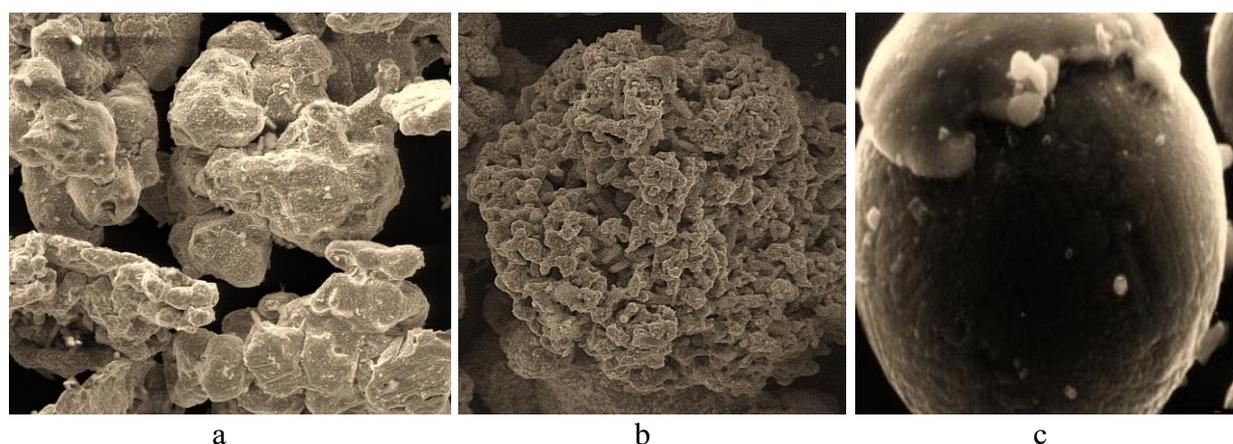


Figure 5. Microstructure of Al₂O₃+30%TiO₂+12.5%MoS₂ composite powder obtained by SHS method: a -after crushing (x 200); b - after spheroidization (x 400); c - after cladding (x 400).

Therefore, to improve the technological parameters of the powders, their spheroidization was carried out [10, 11] by introducing powder particles into a plasma jet with a power of 30-40 kW and spraying in an argon environment (Figure 5b) followed by cladding (Fig-

ure 5c). According to Figure 3, a continuous coating is formed on the surfaces of particles when cladding powders (individual micro-sections of it crumbled during the preparation of polished sections). The mass content of Ni - P in the form of a cladding shell is 30 – 40%. Plasma coatings from composite powders $Al_2O_3+30\%TiO_2+12.5\%MoS_2$; $Al_2O_3+30\%TiO_2+12.5\%CaF_2$. are promising for restoring and strengthening parts operating under unfavorable friction conditions, which is confirmed by the results of Table 2.

Table 2. Friction and wear of coatings on cast iron and steel

Method of obtaining the material	Friction on cast iron				Friction on 38XIOA Ст45			
	Friction with lubricant		Dry friction		Friction with lubricant		Dry friction	
	Coating wear, μm	Coefficient friction	Wear of coating, μm	Load bully, kg	Coating wear, μm	Coefficient friction	Wear of coating, μm	Load bully, kg
Ni80Cr20+ 12,5%MoS ₂ +40%TiC	1,2	0,011	10,2	5,8	1,9	0,03	6,1	6,9
$Al_2O_3+30\%$ $TiO_2+12,5\%$ MoS_2	0,9	0,008	7,9	6,9	1,6	0,02	4,9	8,1

4. Conclusion

The disadvantage of carbide ceramics with solid lubricant inclusions is high thermal dissociation during plasma spraying. During flight in a plasma jet, carbide ceramic particles are prone to loss of stability with a change in chemical composition, which leads to a high coefficient of friction for coatings and negatively affects their performance properties. A composite ceramic material based on oxide ceramics with the addition of a solid lubricant, obtained by self-propagating high-temperature synthesis, has good technological characteristics, is resistant to maintaining the chemical composition during plasma spraying and is capable of forming coatings with high wear resistance and low coefficient friction. Coatings obtained from $Al_2O_3+30\%TiO_2+12.5\%MoS_2$ powder; manufactured by the method of self-propagating high-temperature synthesis, have 1.2 times greater wear resistance during friction with lubricant and 1.3 times greater wear resistance during dry friction on cast iron and steel than a coating obtained from Ni80Cr20 + 12.5% MoS₂ powder +55% TiC. Thus, the proposed method makes it possible to increase the wear resistance of coatings.

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