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## THE STUDY OF TECHNOLOGICAL PECULIARITIES FOR IMPROVEMENT OF CHEMICAL AND PHYSICO-MECHANICAL PROPERTIES OF REACTION-SINTERED CERAMIC MATERIALS BASED ON MOLYBDENUM DISILICIDE

Vasiliy Kovbashyn<sup>1</sup>; Igor Bochar<sup>2</sup>

<sup>1</sup>Ternopil Ivan Puluj National Technical University, Ternopil, Ukraine

<sup>2</sup>Ternopil Volodymyr Gnatyuk National Pedagogical University, Ternopil,  
Ukraine

**Summary.** Recommended ways to improve the chemical and physico-mechanical properties of reaction-sintered ceramic materials based on molybdenum disilicide have been described. In order to significantly increase the operating temperatures and change to more stringent operating conditions for ceramic products, it is necessary to improve existing methods of processing ceramics and significantly change the development of new ones. Various means for processing of ceramic materials have been studied and analyzed, which include the introduction of activating additives, hardening with dispersed particles, filamentary crystals and fibers and application of a protective coating to prevent rapid oxidation at surface layers. Carrying out partial purification of the initial starting components from various impurities can significantly increase some characteristics of the disilicide of molybdenum ceramics. Disilicide of molybdenum ceramics has been researched to have significant influence on the physical and mechanical properties (thermal conductivity, electrical resistance, coefficient of thermal expansion and strength) of molybdenum ceramics disilicides, which are introduced into the base material both with the initial components and in the process of its technological production. It has been established that it is possible to increase operating temperatures and ensure the use of molybdenum disilicide-based ceramic materials in harsher working conditions can be achieved by introducing of aluminum, boron, beryllium, iron, yttrium, nickel and cobalt powders into the charge, strengthening titanium coating, which includes silicification and titanium technology. It has been researched that at high temperatures and in the presence of molten silicon the synthesis and crystallization of molybdenum disilicide occur on the grains in the surface layers, as well as dissolution and recrystallization of submicron particles of molybdenum disilicide take place at certain depth. Based on the published data analysis and conducted research, the complex of measures for improving the chemical and physico-mechanical properties of reaction-sintered ceramic materials based on molybdenum disilicide has been proposed.

**Key words:** molybdenum disilicide, heat resistance, ceramic materials, protective coating.

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**Statement of the problem.** Ceramic materials based on molybdenum disilicide ( $MoSi_2$ ) have been widely used in different engineering fields due to high temperature rigidity, low density, high Young's modulus, low coefficient of thermal expansion, high firmness, high wear resistance and erosion deficiency [1]. Among the used technical ceramic materials, the material obtained by reaction sintering of molybdenum disilicide has become widely used. It has a practically non-porous, two-phase composition, which consists of 80–90% of molybdenum disilicide and 5–20% of free silicon [2]. In order to significantly increase the operating temperatures and change to more stringent operating conditions of ceramic products, it is necessary to improve the existing methods of processing ceramics and significantly change the development of new ones. Therefore, solving such complex problems is currently being carried out in various engineering and technological ways.

**Evaluation of the latest researches and publications.** There are different engineering and technological ways for advancement of technologies and improvement of the chemical and physico-mechanical properties of ceramic materials based on molybdenum disilicide [3]. The most important operational disadvantage of products based on molybdenum disilicide is their low stability at high operating temperatures. In this case, high-temperature oxidation occurs, destruction of bonds between ceramic grains and loss of material strength [2]. In our opinion, one of the promising ways to solve this problem is to study the technological peculiarities for improving the chemical and physico-mechanical properties of ceramic materials based on molybdenum disilicide. It is possible to increase operating temperatures and ensure the use of ceramic materials based on silicon carbide in harsher operating conditions by introducing aluminum, boron, beryllium, iron, yttrium, nickel and cobalt powders into the charge; strengthening with dispersed titanium diboride particles and applying a protective coating.

**Research objective** is to study the technological peculiarities for improving the chemical and physico-mechanical properties of ceramic materials based on molybdenum disilicide.

**Research findings.** According to research findings, one of the promising ways is the use of high-quality primary components for obtaining the base material, which contain a minimum amount of technological impurities [4]. The bending strength of such a reaction-sintered material in the operating range of 1000 °C is 650–790 MPa, which makes it possible to recommend it for manufacturing of important parts of gas turbines, diesel engines, as well as products that operate in hard conditions under the simultaneous action of various aggressive environments and high temperatures. Molybdenum disilicide materials most often have a covalent type of chemical bond, and differ from other engineering materials in their low strength. At the same time, a ceramic material with increased purity of grain boundaries has a high value of durable strength. When even a small amount of boron, aluminum, or carbon is added at high temperature, grain boundaries are destroyed. A significant influence on the physical and mechanical properties (thermal conductivity, specific electrical resistance, coefficient of thermal expansion and strength) of molybdenum disilicide ceramics has impurities introduced into the base material both with the original components and in the process of its technological production [5]. Impurities are located mainly along the grain boundaries and at the same time isolate heat and reduce the coefficient of effective thermal conductivity of ceramics. Simultaneously, there is a significant change in specific electrical resistance, because the grains of molybdenum disilicide have relatively low electrical conductivity, and the existing impurities formed by the phases can create an additional conductive network in the volume of the material at the boundaries of the grains. Carrying out partial purification of the initial output components from various impurities can significantly increase some characteristics of molybdenum disilicide ceramics.

Contemporary research pays considerable attention to the study of the influence of the granulometric composition and structure of the primary components on the physical and mechanical properties of disilicide molybdenum ceramics. It has been established [5] that the strength of reaction-sintered molybdenum disilicide materials is structurally sensitive. The most affordable and effective way to control the strength of the material is to change the size and fraction ratio of the primary molybdenum disilicide powders. There are two options for the formation of strengthened structures: monolithization of molybdenum disilicide framework simultaneously with a gradual decrease of free silicon content and homogenization of this structure [5]. Operation at high temperature in the presence of molten silicon demonstrated the synthesis and crystallization of molybdenum disilicide, as well as the dissolution and recrystallization of submicron particles of molybdenum

disilicide. This process ensures the reduction in contacts, and in high density compositions, – to the formation of local clusters of grains of strong adhesions with a significant violation of the original granulometry. Simultaneously, the structural-transforming role of physico-chemical processes increases at transition to the compositions of fine-grained fractions. There is an increase in the strength of the material with an increase in the level of homogeneity of the ceramic structure in the direction of a decrease in the gap between large and small MoSi<sub>2</sub> grains, dispersion and closer mutual distribution of molybdenum and silicon disilicide phases, a decrease in the weighted average grain size in polyfraction compositions, such processes contribute to the formation of strong structures. If multi-fraction compositions are used, the influence of homogeneity factors is very clearly manifested, the strength of ceramics increases from large to medium and small fractions.

At the same time as the quality and particle size composition of molybdenum disilicide ceramics, the technology of obtaining the base material has a significant impact on improving its properties, which in some cases can ensure the formation of single-phase ceramics that are similar in properties to monocrystalline molybdenum disilicide. To improve the technological and operational characteristics of products made of reaction-sintered disilicide molybdenum materials, pre-pressed porous specimen were impregnated with silicon [5]. The technological operation carried out significantly improves their machinability, wear and corrosion resistance, and heat resistance, however, with increased operating temperature regimes, the strength of such a composition begins to decrease. We propose to consider the study of some stages of the technology for improving the chemical and physico-mechanical properties of molybdenum disilicide by introducing additives, strengthening and coating.

**Introduction of additives.** In order to increase sintering activation process of molybdenum disilicide ceramics, a small amount (1–2 wt. %) of additives are introduced into the charge, in particular, powders of aluminum, boron, beryllium, iron, yttrium, nickel, cobalt, etc. [1, 6]. Aluminum, beryllium, hafnium and rare earth metal oxides can also be effective additives. Proposed additives, as shown by the research results, provide the base material with high density, heat resistance and mechanical strength, stability in sulfuric and nitric acids, sufficient electrical conductivity and wear resistance. However, the use of ceramic materials in high temperature ranges (1200 °C and above) leads to a decrease in its technical and technological indicators. A significant increase in the physical and mechanical properties of disilicide molybdenum ceramics is achieved by introducing up to 15 wt. % of one or more of the following oxides: Al<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>3</sub>, Y<sub>2</sub>O<sub>3</sub>, MgO, Fe<sub>2</sub>O<sub>3</sub>, Br<sub>2</sub>O<sub>3</sub>, etc. An increase in the density of the pressed material, which can reach 97%, ensures an increase in its heat resistance and heat strength at 1400 °C, and the bending strength margin begins to exceed 55 MPa. At the same time, resistance to oxidation and thermal shocks strongly depends on the nature and amount of secondary phases that are formed at the grain boundaries due to sealing oxide additives. Also, conducted research demonstrated that it is possible to successfully use oxygen-free refractory compounds as additives: carbides, nitrides and borides [6].

**Strengthening.** A promising way that makes it possible to improve the physico-mechanical properties of molybdenum disilicide ceramics is its strengthening with dispersed particles, thread-like crystals and fibers [2, 4]. The introduction of dispersed particles of carbide and titanium diboride into molybdenum disilicide ceramics increases the heat resistance of the material within the temperature range of 1000–1400 °C. The technology of reinforcing the ceramic matrix with silicon carbide fibers with a length of 45–55 μm ensures a 2–3 times increase in the viscosity of the matrix, and with the subsequent introduction of 0.5 μm zirconium oxide particles, it starts to increase up to 4 times. Ceramics, after strengthening with silicon carbide fibers, have high thermal and chemical resistance, crack and impact resistance, as well as high viscosity. The ability to

destroy products made of molybdenum disilicide is close to the energy of destruction of aluminum alloys, which allows the use of this ceramic composition for the manufacture of internal combustion engine parts [5].

**Coating.** The biggest disadvantage of reaction-sintered molybdenum disilicide composites is their poor resistance to high-temperature oxidation. The technological stages of the molybdenum disilicide oxidation process are: 1) diffusion of oxygen through the  $\text{SiO}_2$  film; 2) reaction on the  $\text{MoSi}_2$ - $\text{SiO}_2$  distribution surface; 3) counter diffusion of gaseous CO through the  $\text{SiO}_2$  film. As a result, lumps and cracks appear on the surface of the composition, which is the reason for the low technical characteristics of such materials. When manufacturing products which are operated in the high temperature range, it is recommended to use sintered  $\text{MoSi}_2$ , which does not contain free silicon. The bending strength of such material can reach 480, and 3800 MPa in compression, the modulus of elasticity is 415 MPa. This material is resistant to oxidation and the action of a gas environment at temperatures up to 1650 °C, its thermal conductivity is 6 times higher than that of silicon nitride and 2.5 times higher than that of stainless steel. The discrepancy in strength indicators complicates its use in the design of mechanically loaded machine parts. Research results indicate that the heat resistance of reaction-sintered molybdenum disilicide depends on the nature and number of intergranular secondary phases, as well as the presence of residual porosity. It is possible to solve this problem by using various types of protective coatings [2].

In order to increase the resistance of molybdenum disilicide products to oxidation, they are coated with silicon and heated. At the same time, silicon fills the pores, reducing the porosity of the material to a level of less than 1.5%. Titanization and silicification of ceramic products not only protects it from the action of an aggressive environment, but also stops the propagation of cracks in the outer layer of the material, and also increases the viscosity before destruction by 2.5 times, for example, at room temperature it is 2.75, and at 1300 °C almost 5 MPa m<sup>1/2</sup>. For example, the company «Ceramic Refractory Co» offers to protect the structures of furnaces made of molybdenum disilicide ceramics ET-4 type coating, which consists of a crushed mixture of zirconium powders and zirconium dioxide in the form of a colloidal suspension. This coating with a thickness of 0.04–0.06 mm is applied to the surface of the products by spraying. Mechanical and chemical interaction with the base is necessary to obtain strong adhesion of the coating, while the coating is characterized by high stability during operation, in addition, the coating has a high emissivity. A peculiarity of the technological process is the coating not only onto the working part of the product, but also on the reinforcing fibers used to form the composition. Applying a 65 nm thick layer of zirconium dioxide to molybdenum disilicide fibers preserves the mechanical properties of the fibers during long-term thermal exposure to an oxidizing medium, thus creating an effective diffusion barrier at the fiber-matrix interface [6].

A coating is formed on products from reaction-sintered molybdenum disilicide by using a suspension containing 80 wt. % of high-quality silicon carbide and 20 wt. % of bentonite, which is applied to ceramics and then clinkered in air at 1400 °C [1]. At the same time as the resistance of the base material to oxidation increases, its electrical parameters improve. We also studied the process of applying a paste containing molybdenum silicide powder, water and an adhesive additive – methylcellulose, to products made of molybdenum disilicide ceramics, followed by annealing the coating in air within the range of 1300–1500 °C [1]. The research findings showed that this coating reliably protects the ceramic structure from high-temperature gas corrosion. When using such a composition for heating elements made of molybdenum disilicide, which work at high temperatures, a significant increase in the specific load (W/cm<sup>2</sup>) is observed. You can increase the service life of molybdenum disilicide heating elements by impregnating them with a suspension that includes molybdenum disilicide powder, water,

polyvinyl alcohol, and disodium dibutylorthophenol disulfide acid, the technology also involves annealing in an inert environment at 1500 °C [1]. The obtained coating can ensure normal operation of heaters for 2200 hours at 1500 °C. Further increase in operating temperatures leads to a sharp increase in the specific load.

The technology for increasing the heat resistance of molybdenum disilicide structures consists in applying a gas-tight protective layer of silicon carbide with a thickness of up to 0.02 mm to their surface, but this method is very difficult in technical execution. Heating elements made of molybdenum disilicide with high thermal stability and high specific resistance can be obtained by diffusion saturation of them with boron carbide [2]. The essence of the technology is as follows, it is necessary to wet the surface with bakelite varnish, coverage of the entire surface is possible due to rolling with a layer of technical boron carbide powder or a mixture of boron carbide with 10 wt. % silicon and subsequent heat treatment (first in air, and then in a protective atmosphere; the final stage is heating in a hydrogen atmosphere at 2200 °C for 1.5–2 hours), a layer of cubic silicon carbide is deposited on the surface of the product. The mechanical strength of the surface layer is due to the fact that the coefficients of thermal expansion of molybdenum disilicide and the ternary compound of silicon with boron and carbon have approximate values, as well as a smooth transition from the diffusion layer to the ceramic base of the heater [2]. Taking into account the advantages of this method, it is necessary to take into account the disadvantages, namely, a large number of intermediate technological operations and the impossibility of technically processing tubular products.

The use of protective coatings sometimes does not give the desired results, due to the specifics of the technology for obtaining products from reaction-sintered molybdenum disilicide materials, they may contain up to 10 wt. % of free silicon. The operation of ceramic products in high temperature intervals revealed the aggressive effect of unbound silicon, which leads to the appearance of a certain amount of SiO<sub>2</sub>. The inequality of the coefficients of linear expansion of silicon dioxide and molybdenum disilicide when the temperature changes causes local destruction of bonds between grains and loss of technological properties of the material, in particular, silicon dioxide accumulates in molybdenum disilicide heaters, as a result of which the electrical resistance of the elements increases (so-called aging) and overheating occurs in products. The technology of operation of such products causes additional difficulties when using molybdenum disilicide heaters, since if one element fails, it is necessary to replace all furnace heaters, since aging processes begin in operating rods and their electrical parameters differ drastically from the parameters of new heaters [7].

**Removal of free silicon.** It is possible to partially increase the effectiveness of the protective action of the coating applied to the surface by removing silicon from molybdenum ceramic disilicide. If we take into account that the rate of evaporation of silicon from the surface of molybdenum disilicide is somewhat higher than from other ceramic materials [8], then its removal from the base material can be carried out thermally. Our preliminary studies have shown that the removal of unbound silicon can be carried out in a non-oxidizing atmosphere in the range of 1750–2100 °C or in a vacuum ( $p = 0.1$  Pa) at 1650 °C. To prevent contamination of the vacuum furnace with evaporating silicon, as well as impurities contained in the base material, it is recommended to conduct annealing in an environment capable of absorbing silicon and other impurities. But with certain advantages, this method also has significant disadvantages: high labor intensity of the technological process and complexity during processing of long products, in addition, the samples may also contain up to 4 wt. % of free Si.

Free silicon can be removed chemically, because it can actively interact with lithium, sodium, potassium hydroxide in comparison with molybdenum disilicide, which has greater stability in various alkali solutions [3]. The authors of the mentioned work studied the effectiveness of using chemical etching of silicon from reaction-sintered disilicide

molybdenum ceramics, which contains up to 4 wt. % of free silicon. The samples were immersed in a 35% aqueous solution of lithium hydroxide and kept for 50 hours. As shown by the results of chemical analysis, samples after digestion may contain up to approximately 1.5 wt. % of unbound silicon. The advantage of this method is the simplicity of technological process and the ability to deal with products of various configurations. But chemical treatment and subsequent thermal annealing of reaction-sintered molybdenum disilicide ceramics leads to an increase in porosity, possible loss of technological properties of the material (strength), and also does not have complete removal of silicon. Therefore, additional processing of ceramics is recommended, as a result of which free silicon and other impurities present in refractory joints would bind. After this treatment, the porosity decreases and the operational characteristics of the molybdenum disilicide material improve.

**Binding of impurities.** Bonding of impurities is a type of treatment that represents the diffusion saturation of reaction-sintered molybdenum disilicide ceramics with boron, resulting in the formation of borides of silicon and other impurities. Silicon borides have high scale resistance, with a combination of high resistance to thermal shock, they are chemically inert, capable of good radiation and have high thermoelectric parameters [1]. As a result, the physical and chemical properties of ceramics are significantly improved.

In practice, the powder method of boration is more often used. Therefore, the possibility of using this method to bind silicon and other impurities into the molybdenum disilicide material was studied, as well as the effect of saturation on changing the properties of the treated material. Boration of ceramics was carried out in a mixture containing boron carbide, an inert filler and a fluoride activator [2]. Tests of borated samples in air at 1500 °C showed that after exposure for 100 hours, their mass and electrical conductivity slightly changed, while the mass and electrical conductivity of untreated samples greatly increased [9].

The next stage is binding of other impurities present in the material by silicification. The conducted studies showed a general disadvantage of diffusion saturation processes, in particular, their long duration. It is possible to reduce the technological processing time, without impairing the properties of the disilicide molybdenum material, by combining the processes of boronization and silicification, simultaneously carrying out joint borosilication.

Binding of impurities after chemical and thermal treatment significantly improves the physical and chemical properties of reaction-sintered ceramics. However, it is also possible to increase the operating temperature of ceramic materials by using a multi-layer heat-resistant coating. The applied protective layer should have good adhesion with the ceramic base and have high thermoelectric parameters, and what is important, the values of their coefficients of thermal expansion should not differ significantly. The components for forming a high-temperature protective coating can be: silicon carbide, molybdenum disilicide, zirconium dioxide and yttrium oxide. The use of the composition of the above-listed components provides good electrical properties and reliably protects against high-temperature oxidation of structures operating in air in thermocyclic mode at 1550 °C and above. The formation of a layer of silicide-oxide composition on the surface of products made of reaction-sintered molybdenum disilicide improves their temperature operating mode and service life, especially in conditions of multiple fluctuations of the thermal mode [2].

**Conclusions.** Based on the study of literature, as well as conducted research, it is possible to propose a set of measures aimed at improving of the chemical and physico-mechanical properties of reaction-sintered ceramic materials. The combination of certain methods of processing molybdenum disilicide ceramics allows you to significantly expand the range of its use and increase the thermal operating mode.

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## ДОСЛІДЖЕННЯ ТЕХНОЛОГІЧНИХ ОСОБЛИВОСТЕЙ ПОКРАЩЕННЯ ХІМІЧНИХ ТА ФІЗИКО-МЕХАНІЧНИХ ВЛАСТИВОСТЕЙ РЕАКЦІЙНО-СПЕЧЕНИХ КЕРАМІЧНИХ МАТЕРІАЛІВ НА ОСНОВІ ДИСИЛІЦИДУ МОЛІБДЕНУ

Василь Ковбашин<sup>1</sup>; Ігор Бочар<sup>2</sup>

<sup>1</sup>Тернопільський національний технічний університет імені Івана Пулюя,  
Тернопіль, Україна

<sup>2</sup>Тернопільський національний педагогічний університет імені Володимира  
Гнатюка, Тернопіль, Україна

**Резюме.** Описано рекомендовані шляхи покращення хімічних та фізико-механічних властивостей реакційно-спечених керамічних матеріалів на основі дисиліциду молібдену. Для значного підвищення робочих температур і зміни на більш жорсткі умови експлуатації керамічних виробів необхідне вдосконалення уже існуючих способів обробки кераміки та суттєва зміна розроблення нових. Досліджено й проаналізовано різні напрями обробки керамічних матеріалів, які передбачають введення активуючих добавок, зміцнення дисперсними частинками, ниткоподібними кристалами і волокнами та нанесення захисного покриття для запобігання швидкого окислення приповерхневих шарів. Проведення часткового очищення початкових вихідних компонентів від різних домішок суттєво може збільшувати деякі характеристики дисиліцид молібденової кераміки. Досліджено, що значний вплив на фізико-механічні властивості (теплопровідність, питомий електроопір, коефіцієнт термічного розширення та міцність) дисиліцид молібденової кераміки мають домішки, котрі внесені в матеріал основи як з вихідними складовими, так і в процесі його технологічного отримання. Встановлено, що підвищити експлуатаційні температури й забезпечити використання у жорсткіших умовах роботи керамічних матеріалів на основі дисиліциду молібдену можна шляхом введення у шихту порошків алюмінію, бору, берилію, заліза, ітрію, нікелю та кобальту, зміцненням дисперсними частинками дигориту титану і нанесенням захисного покриття, яке включає технологію силіціювання й титанування. Встановлено, що за високих температур у присутності кремнієвого розплаву відбувається синтез і кристалізація дисиліциду молібдену на зернах у приповерхневих шарах, а також розчинення й перекристалізація субмікронних частинок дисиліциду молібдену на певній глибині. На основі аналізу літературних даних і проведених досліджень запропоновано комплекс заходів, направлених на покращення хімічних та фізико-механічних властивостей реакційно-спечених керамічних матеріалів на основі дисиліциду молібдену.

**Ключові слова:** дисиліцид молібден, жаростійкість, керамічні матеріали, захисне покриття.

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