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INVESTIGATION OF THE CHANGE IN TECHNOLOGICAL PROPERTIES OF REFRACTORY METALS AFTER DIFFUSION SATURATION

Vasiliy Kovbashyn[1]; Igor Bochar[2]

[1]Ternopil Ivan Puluj National Technical University, Ternopil, Ukraine
[2]Ternopil Volodymyr Gnatyuk National Pedagogical University, Ternopil, Ukraine

Summary. *The recommended ways of improving the technological and physico-mechanical properties of refractory metals (molybdenum and tungsten) after diffusion saturation with the elements of the same name are described. In order to increase significantly the performance characteristics and transition to stricter operating conditions for products made of refractory metals, it is necessary to improve the existing methods of processing metals and make significant changes in the development of new ones. Different directions of refractory metals processing, which involve diffusion saturation with elements of the same name, introduction of activating additives into the composition of the diffusion mixture, use of isothermal conditions during chemical-thermal treatment, and application of protective coating to improve technological and physical-mechanical properties, have been studied and analyzed. To evaluate changes in the mechanical properties of molybdenum and tungsten after diffusion saturation with the elements of the same name, appropriate tests were conducted. To study the changes in the technological and physical and mechanical properties of refractory metals after the process of diffusion saturation in active-aggressive environment, tests on static rupture and stretching, bending, impact bending and buckling were carried out. It has been established that it is possible to increase the technological characteristics and ensure the use of refractory metals in harsher operating conditions by introducing molybdenum and tungsten powders of the same name and activating additive (sodium fluoride) into the charge to obtain protective coating. Comparison of the magnitude of the temporary resistances values for of untreated samples breaks of refractory metals and samples that have undergone the appropriate heat treatment showed significant decrease in these indicators after diffusion saturation with the same elements (molybdenation and tungstenization). Based on the analysis of literature data and conducted research, a set of measures aimed at the improvement of technological and physico-mechanical properties of refractory metals (molybdenum and tungsten) after diffusion saturation with the same elements in powder medium is proposed.*

Key words**:** *molybdenum, tungsten, refractory metals, diffusion saturation.*

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Statement of the problem. The solution of existing technical problems of significant increase of technological characteristics and duration of operation of products, structures and technological equipment is carried out due to the research and development of protective coating. Protective coatings applied to the surface of refractory metals make it possible to increase significantly the service life of products in electronic, electrical, lighting, and electrovacuum industries. For significant change in operating temperatures and transition to more strict operating conditions for products made of refractory metals, improvements and changes in both existing processing methods and development of new ones are required. Therefore, the solution of such complex problems is currently carried out by various technical and technological ways.

Analysis of available investigations and publications. There are various technical and technological ways of improving technologies and improving chemical, physical, mechanical and technological properties of refractory metals [1, 2]. In the process of chemical and thermal treatment, there is a significant change in the operational capabilities of technological high-temperature equipment or specific products made of refractory metals due to the change in obtained structure, chemical composition, and change in properties in the near-surface layers is also observed. All the above listed changes are the result of physical and chemical processes caused by heating and isothermal aging of processed materials in aggressive high-temperature environment [3]. In high-temperature aggressive environment, active atoms capable of alloying the near-surface layers of the processed materials are formed, chemical adsorption of active atoms is observed and they are absorbed by the surface layers, and at the final stage, the adsorbed atoms diffuse into the depth of the processed material, while chemical bonds are established with atoms of the base metal. The correct choice of the saturating medium ensures obtaining a protective coating with the necessary properties and makes it possible to carry out chemical and thermal treatment using the optimal temperature. The use of optimal temperature and time regimes of chemical and thermal treatment can provide the best diffusion permeability of alloying elements with the simultaneous production of a protective coating with a minimum duration of the treatment process [3]. The technological process of manufacturing rolled molybdenum and tungsten has certain disadvantages, their surface layers may contain certain macroscopic defects in the form of cracks, pores, streaks or even shells, resulting in the deterioration of properties [1]. Identified technological macrodefects can be partially or completely eliminated using mechanical or chemical processing [4]. In our opinion, one of the promising ways to solve this problem is the diffusion saturation of the near-surface layers of molybdenum and tungsten with chemical elements of the same type.

The purpose of the paper is to investigate the technological features of the improvement of physical, mechanical and chemical properties of refractory metals after high-temperature diffusion saturation.

Peculiarities of experiment conducting methods. The process of diffusion saturation of molybdenum and tungsten is carried out on previously refined samples [4, 5]. The in vestigations are carried out with samples of rectangular shape (100x20x0.5 mm) made of rolled steel: molybdenum (MChVP). tungsten (WA). Chemical and thermal treatment is performed in vacuum furnace ($p=10^{-5}$ *Pa*) in the powder mixture which included: ground molybdenum or tungsten powders in quantity no more than 80%, fluoride activator no more than 2%, the rest inert additive Al_2O_3 [6, 7]. The temperature mode is maintained within the range of 700–1000^{0}C, the process duration is no longer than three hours.

Changes in the surface structures are detected by the methods of metallographic and microdurometric investigations. X-ray phase analysis of the internal stresses of molybdenum and tungsten is performed using monochromatic radiation from DRON-3M diffractometer. Comparing the reference samples with the obtained research results, the obtained phase transformations are identified. X-ray phase analysis of molybdenum and tungsten samples is carried on flat grinds. The value of elasticity modulus is determined by the static method on Instron-1126 experimental machine (with the accuracy of deformation degree registration within 0.01 *mm*). Prepared samples with 50x10x0.5 *mm* size are subjected to elastic loads for five times. Elastic properties are investigated using at least ten samples in each experiment. The load is applied through wedge-shaped fasteners, changing the movement of the traverse with V=0,2 *mm/min*. applying the stress not higher than *MPa*. The values of elastic deformations of the investigated samples are measured by means of highly sensitive strain gauge. Under the action of bending forces, errors may occur in the samples, but they are compensated by the design of the strain gauge. The amplification factor of the deformation measurement channel is 1000, and the measuring base of the strain gauge is 29, all investigations are carried out at room temperature. Using S-55IM high-frequency calibrator with permissible error of up to one micron, the strain gauge is constantly calibrated before and after each measurement. While investigating the effect of diffusion saturation on refractory metals (*Mo, W*), the samples are subjected to bending, stretching and impact bending in accordance with the requirements [8] on the breaking machine (force up to 5 *kN*) and KM-4 installation (with pendulum energy up to 2 *J*).

Investigation results. The conducted metallographic analysis of molybdenum and tungsten samples shows that the coating with fine-grained structure having from 5 to 30 microns thickness is formed on the surface, the grains of the metal base are oriented in the rolling direction. At the same time, no clear boundary between the base and the resulting coating is found, only the heterogeneous structure enables to determine approximately the thickness of the protective coating layer. The resulting protective coating is characterized by density, the surface is clean and the presence of macrodefects is not observed. The fine-grained structure of the metal-based protective coating is obtained due to the fact that refractory metals in crystallographic planes have high adsorption capacity. The maximum adsorption of molybdenum atoms that reached the surface during diffusion saturation is observed. The coating layer starts to grow in three directions and forms the required fine-grained structure. The carried out X-ray phase analysis shows that in the absence of macrodefects on the surface of refractory metals, their internal stresses can decrease almost to 20%, which is not typical for molybdenum and tungsten, the samples of which were not subjected to annealing.

Saturating capacity of powder mixture when the concentration of the element that diffuses on the surface of molybdenum or tungsten during diffusion saturation of rolled metal (*Mo, W*) increases, starts to increase in direct proportion to the amount of ground molybdenum or tungsten powder introduced into the mixture. With diffusion saturation in powder mixtures where the concentration of molybdenum or tungsten does not exceed 60%, sharp increase in the specific weight gain Δg of the samples is observed. As a result, with the increase in the specific weight of the samples, the thickness of the applied coating layer on the treated surface starts to increase. Diffusion saturation of refractory metals in mixtures containing molybdenum or tungsten powders up to 60% ensures the increase in coating thickness up to 18 μm on *Mo* samples and up to 14 μm on *W* samples. Carried out investigations show that further increase in the concentration of molybdenum or tungsten powders in the saturating mixture does not lead to the coating thickness growth. Therefore, in order to obtain the optimal coating thickness on the surface of refractory metals (*Mo, W*), it is reasonable to use saturating mixtures with the concentration of elements no more than 60%.

During chemical-thermal treatment, diffusion coatings are applied in isothermal conditions using active-aggressive media [9, 10]. Carried out investigations of the processes of diffusion saturation of molybdenum and tungsten in temperature modes of 700–1000^oC show the increase in the specific gravity of the treated samples with increasing temperature. In the temperature range of 700–850^oC, the specific weight of molybdenum and tungsten samples starts to increase, the thickness of protective coating layer increases (Fig. 1).

Diffusion saturation is carried out in the mixture which includes the following components: tungsten (molybdenum) powder – 60%, sodium fluoride (*NaF*) – 2%, the rest – inert filler aluminum oxide (Al_2O_3). Duration of heat treatment while reaching the specified temperature mode does not exceed one hour. The conducted investigations show that the subsequent increase in temperature mode above 900^oC results in the adhesion of *Mo* and *W* powders to the treated surface, worsening the cleanliness of the surfaces and complicates the further use of the treated samples. Therefore, the temperature modes of diffusion saturation of refractory metals (molybdenum and tungsten) with similar elements should not exceed 950^oC for *Mo* and 900^oC for *W* (Fig. 1). Bending tests of molybdenum and tungsten samples treated at different temperature regimes show that the maximum number of bends for *Mo* ranges from 18±1, and for *W* – 9± cycles.

Investigation of the technological process of surface saturation of molybdenum and tungsten samples the similar elements shows that there is the increase in the specific weight gain of the samples. The initial increase starts with the treatment duration of 30 minutes, and exposure within one hour provides the increase in Δg for molybdenum to 18 mg/cm_2, and for tungsten to 28 mg/cm_2. Further increase in the exposure of the process of diffusion saturation of the refractory metals surface does not provide significant increase in the specific gravity of the samples, and the growth of the thickness of the coating layer is not practically observed.

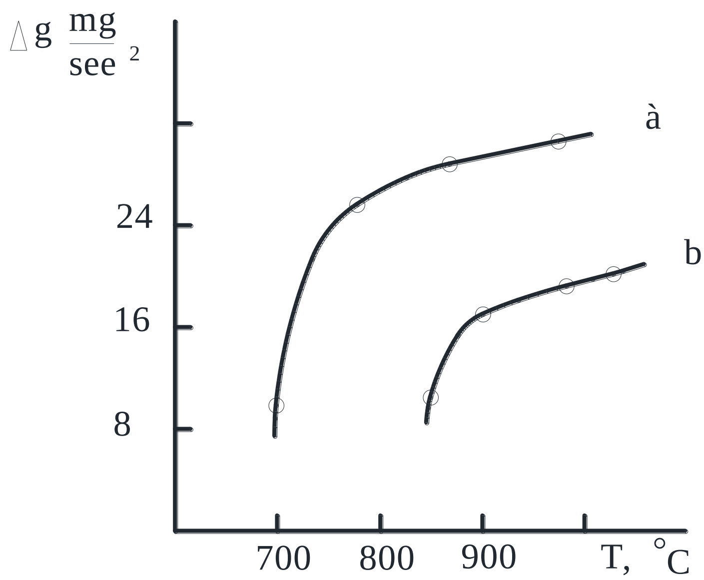

Figure 1. Specific weight gain of molybdenum (a) and tungsten (b) samples

Technological process of saturation of molybdenum and tungsten with similar elements in diffusion medium with their concentration up to 60% and temperature mode within 1000^oC is quite slow. Cariied out invetigations show that it is possible to increase the intensity of saturating mixture by adding to its composition active components capable to form gas phase in the reaction volume and transfer them to the treated surface. Alkaline and alkaline earth metal fluorides have optimal effect of activating additives on the processes of diffusion contact saturation of metals in powder state, sodium fluoride is used [4]. Based on the results of the investigation, it is recommended to add no more than two percent of sodium fluoride (*NaF*) to the composition of saturating diffusion medium. At this concentration of activating additive, the growth of the thickness of protective coating ranges from 5 to 25 *μm*, herewith there are no surface macrodefects.

In order to evaluate the change in mechanical properties of molybdenum and tungsten samples after diffusion saturation as the result of chemical and thermal treatment, appropriate tests are conducted.

For industrial production, mechanical properties of refractory metals, in particular, their elasticity, are of greatest interest. Therefore, a number of investigations are conducted in order to study the influence of diffusion saturation of molybdenum and tungsten in powder mixtures on the value of Young's modulus. The obtained results show that chemical-thermal treatment improves elastic properties of the treated metals. The values of Young's modulus for molybdenum are within the range of 315–335 *GPa*, and for tungsten 360–415 *GPa* (Table. 1). Analysis of the carried out investigations testifies the increase in the values of the modulus of elasticity by 10–20% for the samples made of refractory metals after high-temperature treatment in comparison with the samples of metals which are not treated.

Table 1

Alternation of parameters of the modulus of elasticity for molybdenum and tungsten

Refractory metal	Samples before treatment	Samples after refining	Samples after CTT
Mo	325	316	333
W	368	361	389

In order to investigate changes in physical and mechanical properties of refractory metals after the process of diffusion saturation, tests for static rupture or stretching, bendin and impact bending are carried out.

Investigations of refractory metals samples subjected to static stretching show that tensile strength value for molybdenum is within 1070±25 *MPa*, and for tungsten 1225±30 *MPa*. The fractures are brittle without significant residual deformations in the rupture area. The fracture surface in all carried out experiments is uniform and directed at the angle of approximately 80^o to the longitudinal axis. Molybdenum and tungsten samples have no extreme surfaces with the signs of yield strength for refractory metals. Such indicators as δ (relative elongation, %) and Ψ (relative narrowing, %) for molybdenum and tungsten samples are almost the same.

After diffusion saturation of the surface of refractory metal samples, the values of temporary tensile strengths ranges from 660±20 *MPa* for molybdenum and 765±30 *MPa* for tungsten, that is almost 25% less than the values of unannealed sheet metal samples. The samples destruction occurres to be fragile, so there is no need to determine the indicators of relative elongation (δ) and relative narrowing (Ψ), the surface of the samples subjected to destruction is similar to the surface of the original samples. The distinctive feature is that the destruction is carried out not perpendicular to the longitudinal axis of the samples, but at a certain angle, this angle constantly changes depending on the thickness of the sample and it is difficult to determine it due to the small thickness value. Comparison of values of temporary breaking resistances of untreated samples of refractory metals and samples that have undergone the appropriate heat treatment shows the significant decrease in these indicators after diffusion saturation with the same elements (molybdenation and tungstenization).

According to the results of the investigation, the values of static tensile tests show the decrease in the strength of molybdenum samples by 25–30% after chemical-thermal treatment, and tungsten ones by 20–25%. Analysis of the destruction nature gives reason to conclude that plasticity of refractory metals (*Mo, W*) after diffusion saturation increases by 25–30% in comparison with the samples not subjected to chemical-thermal treatment.

Evaluation of the plasticity of molybdenum sheet rolling is carried out using one of the most difficult methods - bending test, particularly, contacting the opposite sides of the sheet samples. Investigated samples successfully pass these tests after diffusion treatment. According to the results of visual inspection and small increase up to 10–15 times, the absence of cracks on the bent surfaces is determined.

In contrast to molybdenum samples, due to high fragility of tungsten samples, the above-mentioned tests cannot be carried out. The plasticity of tungsten samples is evaluated by determining the permissible value of the bending angle. Carried out investigations on samples that have not been annealed show that cracks begin to occur at $4–5^o$ bending angles (Fig. 2).

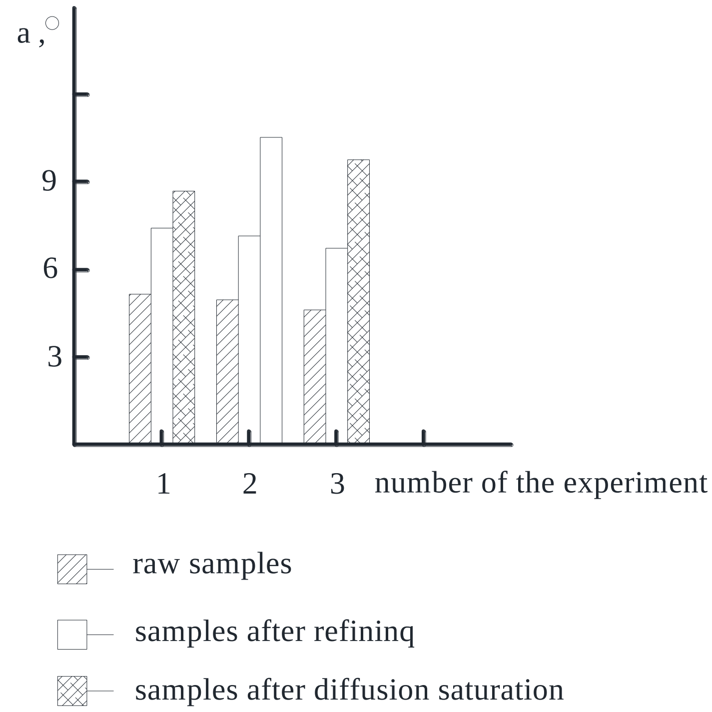

Figure 2. Bending angles of tungsten samples

Cracks appear on the samples that underwent the technological process of refining at 7–8^o bending angles. Samples that underwent diffusion saturation withstand the bending test at 10–11^o angles, only at these values cracks occur.

Therefore, analysis of the carried out tests gives reason to confirm that diffusion saturation of molybdenum and tungsten with the same elements provides significant increase in refractory metals plasticity. For example, the bending angle of tungsten samples after this chemical-thermal treatment increases almost two times in comparison with samples that have not been treated.

In order to evaluate the service life of molybdenum and tungsten samples under sign-changing loads and the possibility of brittle fractures, impact strength investigations are carried out. The obtained data of the investigations show that the values of impact strength for untreated molybdenum samples are within the range of 17±3 J/cm^2. In the samples of molybdenum that underwent chemical and thermal treatment, the value of impact strength reaches 25 $J/cm^2$25, which is almost 20–25% higher than the values of untreated metal samples. Also, unlike untreated molybdenum samples, the investigated samples do not split into parts and do not fail after the first impact. The occurrence of cracks to one millimeter depth is observed, and then the samples are deformed without failure.

Comparing the value of impact strength of unannealed tungsten samples with molybdenum ones, we can notice that it is much lower and is within the range of 2,8 J/cm^2. The investigated failures have complex fractures and follow the broken lines in three clearly defined sections. At the initial stage of failure, the cracks on the surface of the samples are at angles approximately 40–45^o, and at the crushing areas, the cracks occur at angles of 75–80 ^{o}, and metal layering is insignificant. The process of chemical-thermal treatment of tungsten samples reduces its impact strength by almost 55%, but all the investigated samples have expressed metal layering and complex fracture on the surface.

The plasticity of refractory metals is one of the main mechanical properties, as it provides the ability of metals to various shapings. Having carried out the investigations with unannealed samples of sheet molybdenum with 0,5 *mm* thickness, it is determined that they can withstand 3–4 bendings, and then rapid brittle failure. After diffusion saturation of the samples surface with the same element, its plasticity changes significantly and the samples withstand up to 15 bends without failure (Table. 2).

Table 2

Results of molybdenum and tungsten samples bending

Refractory metal	Samples of untreated metal	Samples after annealing in vacuum	Samples annealed in vacuum in heteric mixture	Samples after CTT
Mo	3–4	5–6	12–13	18–19
W	1	2–3	3–4	8–9

Samples of sheet tungsten behave during bending tests in the same way. Samples of unannealed tungsten fail immediately during the first bending, and samples that underwent the process of diffusion saturation with the same element can withstand up to 7 bendings (Table 2).

Conclusions. Carried out investigations indicate that chemical-thermal treatment of refractory metals (*Mo*, *W*), in particular, diffusion saturation with the same elements, is reasonable to perform in working powder mixtures that include almost 60% of molybdenum and tungsten powders with exposure at high temperature in one hour interval. Such diffusion environment ensures the formation of both molybdenum and tungsten coatings with no macrodefects on the surface. Chemical and thermal treatment significantly improves mechanical properties of tested refractory metals samples, ensuring the increase in operating characteristics.

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ДОСЛІДЖЕННЯ ЗМІНИ ТЕХНОЛОГІЧНИХ ВЛАСТИВОСТЕЙ ТУГОПЛАВКИХ МЕТАЛІВ ПІСЛЯ ДИФУЗІЙНОГО НАСИЧЕННЯ

Василь Ковбашин[1]; Ігор Бочар[2]

[1]Тернопільський національний технічний університет імені Івана Пулюя, Тернопіль, Україна
[2]Тернопільський національний педагогічний університет імені Володимира Гнатюка, Тернопіль, Україна

Резюме. *Описано рекомендовані шляхи покращення технологічних та фізико-механічних властивостей тугоплавких металів (молібдену і вольфраму) після дифузійного насичення однойменними елементами. Для значного підвищення робочих характеристик і переходу на жорсткіші умови експлуатації виробів із тугоплавких металів необхідне вдосконалення уже існуючих способів обробки металів та внесення суттєвих змін у розроблення нових. Досліджено та проаналізовано різні напрями обробки тугоплавких металів, які передбачають дифузійне насичення* *однойменними елементами, введення до складу дифузійної суміші активуючих добавок, використання ізотермічних умов під час проведення хіміко-термічної обробки та нанесення захисного покриття для покращення технологічних та фізико-механічних властивостей. Для оцінювання змін механічних властивостей молібдену і вольфраму після дифузійного насичення однойменними елементами проведено відповідні випробування. Для вивчення змін технологічних та фізико-механічних властивостей у тугоплавких металів після процесу дифузійного насичення в активно-агресивному середовищі проведено випробування на статичний розрив і розтягування, згинання, ударне згинання і перегинання. Встановлено, що підвищити технологічні характеристики та забезпечити використання у жорсткіших умовах експлуатації тугоплавкі метали можна шляхом введення в шихту однойменних порошків молібдену і вольфраму та активуючої добавки (фторид натрію), отримавши захисне покриття. Порівняння величин значень тимчасових опорів розривів необроблених зразків тугоплавких металів і зразків, які пройшли відповідну термічну обробку, показало значне зменшення цих показників після дифузійного насичення однойменними елементами (молібденування і вольфрамування). На основі аналізу літературних даних і проведених досліджень запропоновано комплекс мір, направлених на покращення технологічних та фізико-механічних властивостей тугоплавких металів (молібдену і вольфраму) після дифузійного насичення однойменними елементами у порошковому середовищі.*

Ключові слова: *молібден, вольфрам, тугоплавкі метали, дифузійне насичення.*

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