Research of Surface Wear Resistance of Aluminum Alloy Modified with Minerals using Sclerometry Method

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*Received 01.07.2019
Accepted for publication 14.08.2019*

Abstract

Improving the wear resistance of the surface of metal parts used in various industries is one of the relevant areas of materials science. The aim of this work was a comparative study of the wear resistance of a sample of an aluminum alloy (EN AW-2024, an aluminum alloy of the Al-Cu-Mg system) modified with ultrafine particles of minerals using the sclerometry method, which makes it possible to measure the physicomechanical properties of the material at the microscale, as well as determining some tribological parameters (hardness and elastic modulus) of a duralumin sample with a mineral coating.

Wear resistance was measured using a NanoScan-4D scanning hardness tester using the multi-cycle friction method using a sapphire sphere with control of the pressing force and the deepening of the tip into the sample. The use of such a measurement system is especially important when testing thin modified layers, when the layer thickness is comparable with the surface roughness parameters and the influence of the substrate is excluded.

The measurement results showed that the wear resistance of the surface of an aluminum alloy sample modified with ultrafine mineral particles increased by more than 12 times compared to the wear resistance of an aluminum alloy surface without modification. Also, measurements of the hardness and elastic modulus of the surface of the modified sample were performed taking into account the features of measuring the mechanical parameters of thin layers.

The obtained parameters of the modified surface of the aluminum alloy can be further used to build models of the processes of friction and wear of the surface modified by ultrafine particles of minerals. The lack of an acceptable explanation of the nature of the special properties of the surface modified by particles of minerals of natural origin does not exclude the use of the observed effects to significantly increase the resource of various parts and mechanisms.

Keywords: surface modification, aluminum alloy, wear resistance, hardness, mineral coatings.

DOI: 10.21122/2220-9506-2019-10-3-263-270

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Для цитирования:
A.V. Skazochkin, G.G. Bondarenko, P. Żukowski
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Приборы и методы измерений.
2019. – Т. 10, № 3. – С. 263–270.
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For citation:
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Исследование износостойкости поверхности алюминиевого сплава, модифицированного минералами, методом склерометрии

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Поступила 01.07.2019
Принята к печати 14.08.2019

Повышение износостойкости поверхности металлических деталей, используемых в различных отраслях промышленности, является одним из актуальных направлений материаловедения. Целью данной работы являлось сравнительное исследование износостойкости образца из алюминиевого сплава (EN AW-2024, алюминиевый сплав системы Al-Cu-Mg), модифицированного ультрадисперсными частицами минералов с использованием метода склерометрии, позволяющего измерить физико-механические свойства материала в микромасштабе, а также определение некоторых трибологических параметров (твердости и модуля упругости) образца из дюралюминия с минеральным покрытием.

Измерение износостойкости было выполнено с помощью сканирующего твердомера «НаноСкан-4D» методом многоциклового трения сапфировой сферой с контролем силы прижима и углубления наконечника в образец. Использование такой системы измерения особенно важно при испытании тонких модифицированных слоев, когда толщина слоя сопоставима с параметрами шероховатости поверхности и исключено влияние подложки.

Результаты измерений показали, что износостойкость поверхности образца из алюминиевого сплава, модифицированной ультрадисперсными частицами минералов, увеличилась более чем 12 раз по сравнению с износостойкостью поверхности из алюминиевого сплава без модификации. Также выполнены измерения твердости и модуля упругости поверхности модифицированного образца с учетом особенностей измерения механических параметров тонких слоев.

Полученные параметры модифицированной поверхности алюминиевого сплава могут быть в дальнейшем использованы для построения моделей процессов трения и износа поверхности, модифицированной ультрадисперсными частицами минералов. Отсутствие в настоящее время приемлемого объяснения природы особых свойств поверхности, модифицированной частицами минералов природного происхождения, не исключает использования наблюдаемых эффектов для значительного повышения ресурса различных деталей и механизмов.

Ключевые слова: модификация поверхности, алюминиевый сплав, износостойкость, твердость, минеральные покрытия.

DOI: 10.21122/2220-9506-2019-10-3-263-270

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Introduction

Aluminum alloys are widely used in industrial production due to the combination of physical and mechanical properties that provide sufficient performance characteristics along with low specific gravity [1–2]. Compared to steels and titanium alloys, aluminum alloys have lower hardness, which affects their wear resistance [2]. However, due to its special properties and low cost, aluminum alloys stably occupy the niche of low-inertia parts of various machines. Moreover, the service life of the parts is relatively small, which can lead to problems associated with the need to replace aluminum parts that fail as a result of wear [1–2].

The traditional ways to increase the wear resistance of wearing parts made of aluminum alloys is coating and surface modification [3–8]. Reliable enough ways to create a protective coating on the surface of aluminum and its alloys are chemical and electrochemical oxidation [3], the creation of cermet composite materials with an aluminum matrix hardened by refractory, high-strength ceramic particles [4–6], the technology of arc surfacing [7], the use of laser surface treatment methods [8]. But these methods are ineffective both from the point of view of technical parameters and economically, because they are accompanied by high-temperature heating, limitation of overall dimensions, and brittleness of the resulting layers. Therefore, it is urgent to develop other methods for modifying the surface of aluminum and its alloys with minimal or low thermal impact, in particular, methods of ion implantation, electrospark alloying, and technology of mineral coatings.

The methods that use low-temperature operations to create a modified layer include the socalled technology of mineral coatings [9]. The essence of the technology is to create a modified surface layer with a thickness of 5–30 μm by plastic deformation using ultrasonic and mechanical effects that activate the entry of ultrafine particles of minerals into the metal volume [9–10]. However, currently there are no models that correctly describe the effects that arise when creating layers containing ultrafine particles of minerals. Possible causes of changes in the physical properties of the surface with micro and nanoparticles of minerals are listed in [11]. It should be noted here that the choice of characteristics included in a particular model is determined mainly by the technical capabilities of their measurement. Until now, the surface parameters of aluminum samples modified with ultrafine particles of minerals remained unknown. This is partly why the real work was done.

There is also the problem of the correctness of the measurement of the physicomechanical properties of thin coatings and thin modified layers, arising due to the presence of factors leading to methodological errors for some methods of measuring wear resistance, hardness, and elastic modulus [12–13]. The most significant factors are surface roughness, residual stresses, and the effect of the substrate [14]. The effect of the substrate or the bulk of the base metal is that for the system a modified layer – the base metal – the recorded response of the material during measurement depends on the properties of the layer and the properties of the volume of the metal [14, 15].

The possibility of avoiding some of the shortcomings listed above when measuring wear resistance, along with other methods, is provided by the use of the multi-cycle friction method with a sapphire sphere with control of the pressing force and the deepening of the tip into the sample, which is provided, for example, by the scanning NanoScan-4D hardness tester [12].

The purpose of this work is a comparative study of the wear resistance of a sample of an aluminum alloy modified with ultrafine particles of minerals using the sclerometry method, which makes it possible to measure the physicomechanical properties of the material at the microscale, as well as the determination of some tribological parameters (hardness and elastic modulus) of a duralumin sample with a mineral coating. The parameters of the surface with a mineral coating can be further used to build models of the processes of friction and wear of a surface modified with ultrafine particles of minerals.

Materials and methods

Two samples of aluminum alloy bars EN AW-2024, aluminum alloy of the Al-Cu-Mg system (Al – 93 %, Cu – 4.2 %, Mg – 1.4 %, Mn – 0.4 %), were made by turning without grinding in the form of discs 10 mm thick, with a diameter of 33 mm, with a hole in the middle with a diameter of 11 mm. A modified mineral layer was created on the surface of one of the samples according to the basic technology of SPA «Geoenergetika» [9]. The thickness of the modified layer, on the basis of the technological parameters during its creation and earlier experiments, was about 10 µm [9]. Comparative measurements of the wear resistance and surface roughness, as well as the hardness and elastic modulus (Young) of the modified samples were performed on the samples.

The surface roughness measurement was carried out on a standard profilometer. The following parameters of the measurement procedure were used: profile length – 12.5 mm, profile measurement speed – 0.5 mm/s.

The measurement of hardness and modulus of elasticity was carried out using a NanoScan-4D nanohardness meter, which allows indentation in accordance with the recommendations of ISO 14577 [12]. The measurement method is the indentation of the diamond pyramid with the registration of the indentation diagram and the subsequent calculation of the hardness and modulus of elasticity in accordance with ISO 14577, which is often called the instrumental indentation method.

The measurements were carried out by the indenter in the form of a three-sided pyramid of the Berkovich type, the loading time was 10 s, the unloading time was 10 s, the time for maintaining the maximum load was 2 s, and the maximum load was 20–1500 mN.

The wear resistance was measured using the NanoScan-4D scanning hardness tester using the method of multi-cycle friction with a sapphire sphere with control of the pressing force and deepening of the tip into the sample [12, 13]. The NanoScan nanohardness tester combines the capabilities of classic nanoindenters and scanning probe microscopes, which have a number of additional features. In this device, the use of sclerometry during abrasion allows using a high-speed feedback system to actively maintain a given load at each point of the trajectory during abrasion. If the tip moves from a local peak to a depression, the load system presses or pulls the measuring head so that the contact conditions are maintained constant. In addition, the possibility of local shock loads characteristic of the passive task of a normal load is excluded. The use of such a measurement system is especially important when testing thin layers, when the layer thickness is comparable with the surface roughness parameters.

The hard-tip is a spheroconic sapphire crystal with a tip radius of 42 µm (Figure 1).

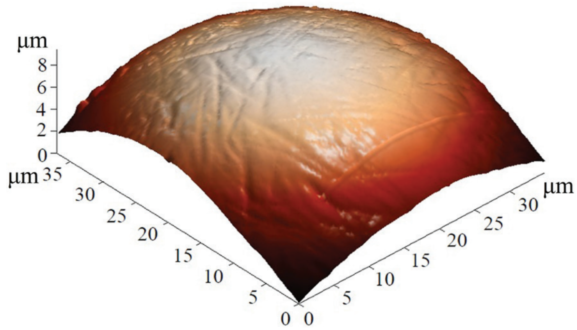

Figure 1 – Image of tip edge, received by means of the Scanning Probe Microscope

The measurement procedure parameters: the radius of the sapphire tip tip is 42 µm, the wear track length is 1 mm, the number of cycles of reciprocating movement is 100, the loading force during testing is 100 mN and 500 mN, the speed of movement of the tip is 120 µm/s. One cycle consisted of two scratches, successively created by direct and reverse stroke. The speed of movement of the tip in the above parameters of the measurement procedure is indicated in the middle of the abrasion area, at the edges of the test area (approximately 5 % of the length) there are areas of acceleration and deceleration. Three friction tests were performed on each sample.

Research results and discussion

The roughness measurement was carried out by measuring the surface profiles of the samples. At least three profiles with a length of 12.5 mm were drawn on each sample; roughness parameters are shown in Table 1.

Table 1

Roughness of the samples[+]

Sample	*Ra*, µm	*Rq*, µm	*Rz*, µm
Aluminium alloy	1.144 ± 0.039	1.393 ± 0.036	6.38 ± 0.15
Aluminum alloy with a modified layer	1.26 ± 0.16	1.69 ± 0.285	11.2 ± 2

[+] In Table 1: *Ra*, µm – arithmetic mean deviation; *Rq*, µm – standard deviation; *Rz*, µm – height of irregularities at 10 points

It is obvious that the modification of the surface with minerals led to a relatively small increase in the roughness parameters of the samples. The maximum relief difference is close to the intended coating thickness (10 µm). According to ISO 14577, the hardness measurement should be carried out at a depth of 20 times the parameter *Ra*, in which case the influence of roughness on the measured value will be less than other sources of error. If this ratio is not observed, a significant increase in the scatter of the measured hardness values is expected due to the deviation of the actual contact area of the tip with the sample from that provided for in the theoretical model.

Measurements in the instrumental nanoindentation mode were carried out with a Berkovich pyramid-shaped tip and were performed as follows: on each of the samples obtained, a series of injections with various maximum loads were applied. The load was chosen so that the depth of penetration of the tip into the material was from 500 nm to 3 μm. The measurements were carried out without preliminary selection of the indentation site, the distance between the points is not less than 100 μm.

The results of measuring the hardness H and the modulus of elasticity E of the modified sample are shown in Table 2.

Table 2

Mechanical characteristics of the samples

Sample	Load, mN	Depth, nm	Hardness H, GPa	Module elasticity E, GPa
Aluminum alloy with a modified layer	20	740 ± 240	2.4 ± 1.8	83 ± 46
	50	1170 ± 330	2.1 ± 0.8	75 ± 32
	100	1600 ± 700	2.5 ± 1.2	84 ± 37
	250	2600 ± 700	2.0 ± 0.8	93 ± 35
	500	3900 ± 1000	1.7 ± 0.7	87 ± 30

As mentioned above, the thickness of the modified layer was about 10 μm [9], and these measurements were made in the depth range from 1 to 4 μm. At small depths, the influence of the substrate on the measured values is small and, therefore, it could be neglected when determining the average values. The scatter of data is 40–60 % of the measured values, which is expected and due to the relatively large roughness of the samples compared to the indentation depth.

In these measurements using instrumental indentation, it is necessary to focus on the average measured values, since the roughness greatly increases the spread of the measured values. The increase in the spread is due to the fact that the actual contact area can vary at the point of contact of the indenter with the surface, depending on the local topology of the relief.

The specificity of the instrumental indentation technique is that observation of the footprint area is not performed. The hardness values are calculated using the loading diagram recorded during the experiment, which is the dependence of the load applied to the indenter on the depth of indentation of the indenter into the surface of the material. In the model on the basis of which the treatment is carried out in accordance with ISO 14577, the surface is assumed to be normal to the direction of indentation and is absolutely smooth. For such a surface, the calibration function of the indenter shape is preserved and the calculation of hardness is characterized by maximum accuracy. However, without exception, all real surfaces are rough and wavy, what introduces an error in the calculated values of hardness. At the same time, depending on the contact scheme, the actual contact area can be either larger or smaller than the calibration one. Thus, only because of the surface roughness, the measured value may differ from the true one up or down, since the hardness in this method is calculated as the ratio of the applied load to the contact area of the indenter with the sample. In the present experiment, since the pricking was done at random points on the surface, it is advisable to operate and compare the average measured values for the initial and modified surface, rather than the extreme points, taking into account the error.

Figures 2–5 show photographs of the areas of surface wear after the test and curves for the tip deepening into the sample as a function of test time. Depth values are counted from the surface level during the first abrasion cycle. The tests are marked with numbers in the photographs and the color of the curve in the graphs (1 – black, 2 – red, 3 – blue).

Table 3 shows the average linear wear of the specimen between 50 and 100 test cycles, Δh – linear wear, average tip groove per friction test cycle.

Table 3

Linear wear

Sample	Load, mN	Linear wear Δh, nm
Aluminium alloy	100	75 ± 15
Aluminum alloy with a modified layer	100	5.9 ± 1.2

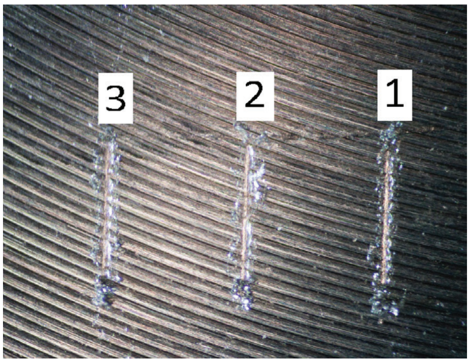

Figure 2 – A photograph (x40) of the surface of aluminium alloy sample without modification after testing with a load of 100 mN

From the data of Table 3 and Figures 3 and 5 it can be seen that the wear resistance of the surface of a sample of aluminum alloy, modified by minerals according to the technology of mineral coatings, increased by more than 12 times compared to the wear resistance of the surface of the aluminum alloy without modification. The observed wear schedules are very similar to the traditional curve of normal wear at the run-in and normal wear stages [14–15]. Small fluctuations can be explained by the role of microparticles of minerals, deflecting the needle of a scanning probe microscope. Thus, the present study suggests that at the micro level, wear should follow the general patterns characteristic of macrosystems, adjusted for the increasing role of surface forces and adhesion [11, 14].

The lack of standardized test methods is explained by the complexity of the processes and the presence of a large number of factors that affect friction and wear of materials, the divergence of views and theoretical positions from which they are considered. The processes that occur during friction and wear can be very different from materials, environmental conditions, the presence and properties of the lubricant. The frictional properties of materials are affected by the physicochemical state of the surface, temperature, specific load, sliding speed, elastic modulus, hardness, brittleness, roughness, and fatigue coefficient [14–15]. Depending on the relative penetration depth, the microroughness of the friction surfaces and the relationship between the cohesion and adhesion forces on the actual contact spots, the following can be realized: elastic deformation, plastic displacement of the material, microcutting, setting of films or surfaces (adhesive or cohesive detachment) [15]. All this causes the emergence of many test methods, many of which later become standardized [16]. Numerous factors affecting friction and wear necessitate the study of their combined effect and mutual influence to obtain the most complete information about the material and require, as a rule, the use of several measurement methods.

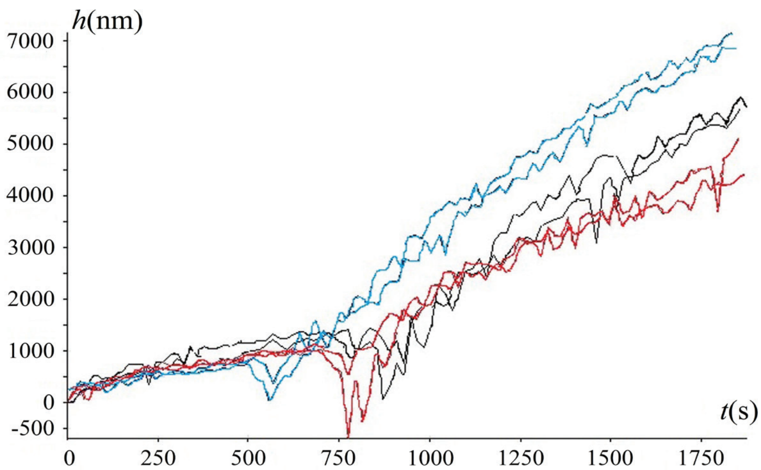

Figure 3 – Wearing diagram of the surface of a aluminium alloy sample without modification when using a sapphire tip with a load of 100 mN. In the diagram: h (μm) – value of the tip deepening, t (s) – test duration. The depth value is measured from the surface level during the first abrasion cycle. The tests are indicated by numbers on the photographs and the colour of the curve on the graphs (1 – black, 2 – red, 3 – blue)

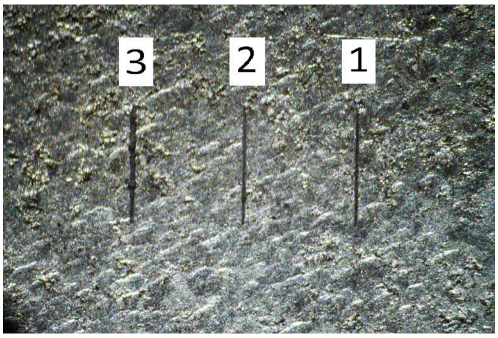

Figure 4 – A photograph (x40) of the surface of a aluminium alloy sample with modification of the surface by minerals after testing with a load of 100 mN

In the future construction of a model of the process of friction of metal layers modified with ultrafine particles of minerals, the interconnectedness of physical processes occurring at different scales should be taken into account. For example, in the process of friction of two surfaces, changes occur in the macro-volumes of matter, the micro-volumes of matter, and in the electronic shells of their atoms. The miniaturization of the models of rubbing bodies leads to a transition from the bulk properties of the material, on which averaging over the main inhomogeneities and the description by methods of the elastic-plastic medium are possible, to their surface properties, estimated by measuring adhesion, micro and nanoindentation. The lack of an acceptable explanation of the nature of the special properties of the surface modified by ultrafine particles of minerals of natural origin does not preclude the use of the observed effects to significantly increase the resource of various parts and mechanisms.

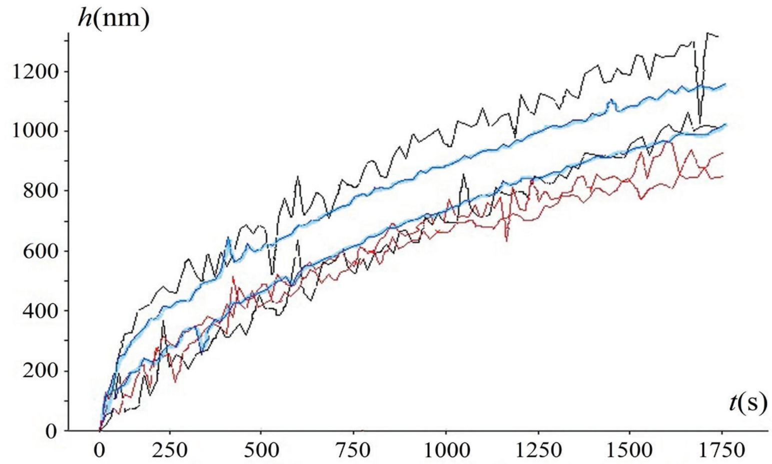

Figure 5 – Wearing diagram of the surface of a aluminium alloy sample with modification of the surface by minerals when using a sapphire tip with a load of 100 mN. In the diagram: *h* (μm) – value of the tip deepening, *t* (*s*) – test duration. The depth value is measured from the surface level during the first abrasion cycle. The tests are indicated by numbers on the photographs and the colour of the curve on the graphs (1 – black, 2 – red, 3 – blue)

The relevance of measurement accuracy in the micro and nanometer range is confirmed by the international standard published in 2012 on methods for conducting micro and nanotribological tests [16]. It focuses on the general requirements for the testing machine and basic test methods. Further development of the standard may concern the features of the choice of measuring methods and types of tips, depending on the tasks assigned to the researcher.

Conclusions

A comparative study of the wear resistance of a sample of an aluminum alloy EN AW-2024 (aluminum alloy of the Al-Cu-Mg system) modified by ultradispersed mineral particles was performed using the sclerometry method. This method measures the physicomechanical properties of the material in microscale, and also determined the hardness and elastic modulus specimen of aluminum alloy with mineral coating. After the modification of the surface of the sample of aluminum alloy by the technology of mineral coatings using low-temperature technological operations (local heating to 80 °C), the wear resistance of the surface of the sample of aluminum alloy modified by mineral particles increased by more than 12 times compared to the wear resistance of the surface of aluminum alloy without modification. The obtained parameters of the modified surface of the aluminum alloy can be further used to build models of the processes of friction and wear of the surface, modified by ultradispersed particles of minerals.

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