

TESTING MECHANICAL STRUCTURAL CHARACTERISTICS OF Al_2O_3 OXIDE CERAMICS RESISTANT TO SLIDING FRICTION

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Summary:

Al_2O_3 is hard oxide ceramics, chemically bioinert and with good sliding properties; it has found wide application in the design of engineering components. The aim of the paper was to apply the APS process with the change of plasma currents in order to produce Al_2O_3 layers of such structural and mechanical properties which will find application in the manufacture of biomedical coatings alone or in combination with hydroxyapatite (HA) on the surfaces of alloys used for making implants. The coating was deposited with a plasma current of 700, 800 and 900 A. The mechanical properties of coatings were tested using the Pratt & Whitney standard. The shape of the surface of powder particles and the coating surface were examined by SEM. The metallographic analysis of the inner layers was carried out by light microscopy. The best structural and mechanical properties of the Al_2O_3 coating deposited with 900A were confirmed by testing the sliding properties of the coating deposited and polished to a mirror on the sealing ring paired with the graphite ring on the water brake.

Keywords: sliding, property, oxides, mechanical properties, deposits, coatings, Al_2O_3 .

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Introduction

Ceramic coatings are widely used to improve wear resistance of working surfaces of various mechanisms due to their high strength and structural stability (Toma et al, 2001, pp.149-158). Al₂O₃ ceramics is applied to many engineering components where good sliding properties and wear resistance to friction are required. Because being bioinert in living tissues, Al₂O₃ ceramics is applied to the surface modification of biomedical coatings deposited on titanium alloys. Good mechanical characteristics of Al₂O₃ ceramics allow it – since it is inorganic and bioinert - to be combined in different volume ratios with organic bioactive ceramics hydroxyapatite (HA) in the process of producing biomedical coatings. In this way, coating stiffness is reduced, tensile strength and fatigue resistance to friction are increased, which extends the life of the coating (Li et al, 2006, pp.1166-1172), (Liou, 2009). Applying such modified coatings on the surfaces of titanium alloys significantly enhance the biological activity of implants and increase the resistance of titanium alloys to bio corrosion (Gadow et al, 2010, pp.1157-1164). For the manufacture and modification of biomedical coatings, ceramics of Al₂O₃ and ZrO₂ are most frequently used, alone or in combination, as well as hydroxyapatite (HA) (Hsiung et al, 2012, pp.457-463).

In the plasma spray process, due to high speed of plasma particles and uneven distribution of temperature in plasma, plasma current changes significantly affect total or partial melting of Al₂O₃ powder particles, which is reflected in the structure and mechanical properties of the coating (Friis et al, 2001, pp.115-127), (Matejcek & Sampath, 2001, pp.1993-1999). Previous studies have shown that the properties of the coating are directly related to the parameters that influence the degree of melting of particles and the mechanism of deformation of molten drops in collision with the substrate (Li & Ohmori, 2002, pp.365-374), (Mrdak, 2016a, pp.1-25), (Mrdak, 2016b, pp.411-430). The coating quality is primarily defined by its microstructure (type of phases and homogeneity) and its mechanical properties that are critical for reliable behavior of the coating in exploitation (Tucker, 2002, pp.45–53), (Liao et al, 2000, pp.235–242), (Toma et al, 2001, pp.149–158). The share and the phase α -Al₂O₃/ γ -Al₂O₃ ratio depends on the amount of heat which drops of molten particles carry with them and which is directly dependent on the size of melting particles, cooling rate and substrate temperature (Kulkarni et al, 2004, pp.124-137), (Yang et al, 2006, pp.1649-1653). The γ -Al₂O₃ phase in the coating is desirable for increasing toughness and cohesive coating / adhesion strength. Resistance to sliding contact fatigue of the

Al_2O_3 coating depends on the phase composition, content and pore size, and presence of unmelted particles and cracks.

The paper presents an analysis of the results of tests of the mechanical properties and the microstructure of Al_2O_3 coatings deposited with 700, 800 and 900A. Because of economic effects, Al_2O_3 coatings were deposited on steel substrates. The aim was to use plasma in the manufacture of coating layers to be applied for the modification of biomedical coatings and the surface modification of alloys used to produce implants. The coating with the best properties is tested on the sealing ring of the water brake paired with the graphite ring. The investigation confirmed that the Al_2O_3 coating, polished to a mirror on the sealing ring, substantially improved the efficiency of the sealing and significantly reduced the sealing ring friction abrasion, thus allowing the use of Al_2O_3 coatings for implant surface modification.

Materials and experimental details

Al_2O_3 oxide powder used for coating deposition is Metco 105NS produced by the technique of melting / grinding molded blocks to a granulation of 15 - 45 μm . (Material Product Data Sheet, 2012). Figure 1 (SEM) shows the surface of Al_2O_3 powder particles used in this work; the particles are of irregular shapes with sharp edges.

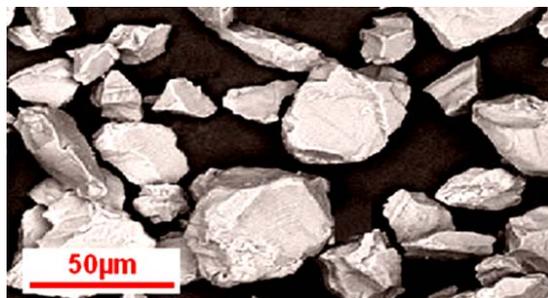


Figure 1 – Surface of Al_2O_3 particles

Рис. 1 – Изображение поверхности частиц Al_2O_3

Слика 1 – Изглед површине Al_2O_3 честица

To test the mechanical properties and the structure of the layers, the powder is deposited on steel samples (X15Cr13 EN10027), the characteristics and dimensions of which are prescribed by the Pratt & Whitney standard (Pratt & Whitney, 2002). To measure the microhardness and adhesion strength, the Pratt & Whitney standard was used, providing the measurement procedure relating to the number of samples and the number of measuring points (Pratt & Whitney, 2002). The paper presents the

minimum and maximum values of microhardness and average adhesion strength. The metallographic analysis of the coatings and the content of pores in the coatings was performed under a light microscope. The paper presents the average values of a share of pores. The analysis of the surface of the powder particles and the surface of the deposited coating is carried out by the SEM method.

The Al₂O₃ powder was deposited at atmospheric pressure with a SG-100 plasma gun and a power supply of 40kW. The Al₂O₃ powder deposition parameters are shown in Table 1. The deposited layers formed a coating thickness from 0.18 to 0.20 mm.

Table 1 – Plasma spray parameters
Таблица 1 – Параметры плазменного напыления
Табела 1 – Плазма спреј параметри

Deposition parameters	Values		
Plasma current, I (A)	700	800	900
Plasma Voltage, U (V)	35	37	42
Primary plasma gas flow rate, Ar (l/min)	50	50	50
Secondary plasma gas flow rate, He (l/min)	32	32	32
Carrier gas flow rate, Ar (l/min)	7	7	7
Powder feed rate, (g/min)	40	40	40
Stand-off distance, (mm)	115	115	115

Results and discussion

Figure 2 illustrates the results of the microhardness of Al₂O₃ coatings deposited with a change in plasma current amperage. The measured values of coating microhardness were directly related to the applied current amperage. The lowest microhardness value of 780 – 1010HV_{0.3} belongs to the coating deposited with 700A, and the maximum value of 990 - 1180HV_{0.3} characterizes the coating deposited with 900A. Nonuniform and different coating microhardness values are the result of uneven distribution of micro pores, different pore content and pore size in the coating layers. The plasma current amperage significantly influenced the melting of powder particles. Lower current intensity was not enough to melt injected particles throughout the complete cross section, which resulted in the deposited particles having a smaller intercon-

tact surface and a higher intercontact content of micro pores. Due to poorer melting of particles and a larger share of pores, the coating had a range of microhardness of 230HV_{0.3}. The coating deposited with 900A had a range of microhardness lower than 190HV_{0.3}. High amperage affects a greater degree of ionization of the He monoatomic gas, which increases the plasma temperature and heat transfer from plasma to powder particles.

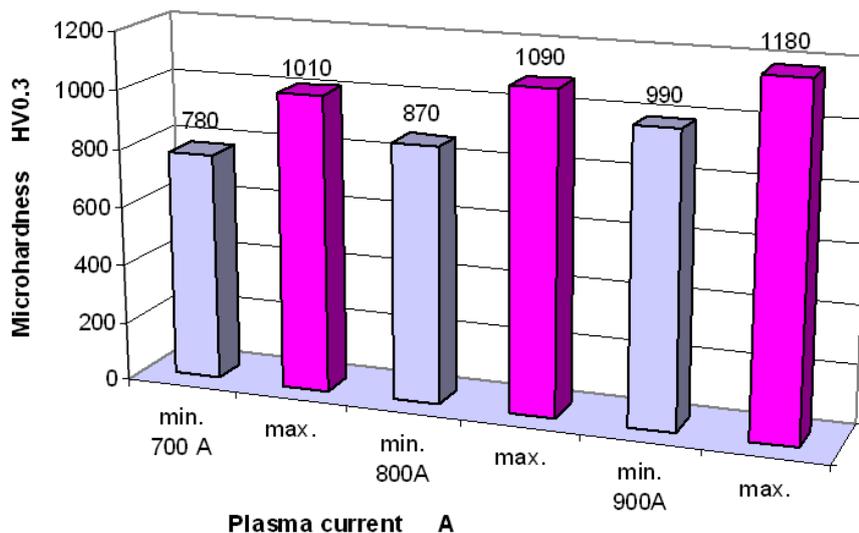


Figure 2 – Results of the microhardness of Al₂O₃ coatings
 Рус. 2 – Результаты микротвердости покрытия Al₂O₃
 Слика 2 – Резултати микротврдоће превлака Al₂O₃

Consequently, powder particles melt better and more evenly throughout the cross section. Liquid drops of molten particles are better bonded to one another achieving a greater bonding area and stronger cohesive strength with a smaller proportion of micro pores. Figure 3 shows the results of the adhesion strength of the substrate / Al₂O₃ coating bond depending on the amperage. The Al₂O₃ powder deposited with 700A achieved the lowest value of adhesion strength of 38MPa with the substrate. Due to uneven and incompletely melted powder particles in plasma with 700A, particles carry a small amount of heat. Therefore, incompletely melted particles transfer a smaller amount of heat to the substrate, which is why their bond to the substrate is weak.

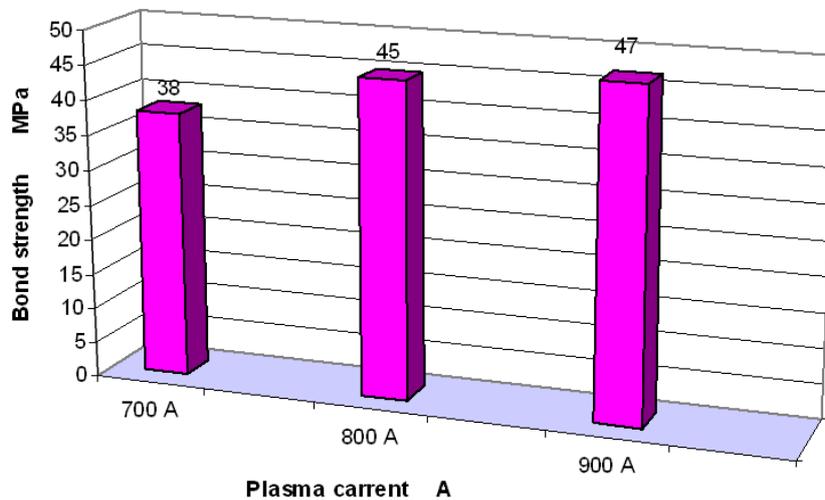


Figure 3 – Results of the bond strength of Al_2O_3 coatings
 Рис. 3 – Результаты твердости связи покрытия Al_2O_3
 Слика 3 – Резултати чврстоће споја превлака Al_2O_3

Heating the metal substrates from liquid drops of completely melted Al_2O_3 particles deposited with 900A increases the adhesive bond strength to 47 MPa. At the fracture of the samples, it was established that the fracture mechanism was adhesion at the substrate / Al_2O_3 coating interface.

Figure 4 illustrates the cross section of the microstructure of the Al_2O_3 coating deposited with 900A. The substrate / coating bond is very good because contamination from corundum, coarse pores and micro cracks are not visible at the interface. High amperage of plasma current allowed the drops of completely melted particles to overlap completely in collision with the substrate and form thin lamellae and inter lamellar pores of small size and small content in the coating. In the coating, there are micro pores of 5 - 10 μ m with a mean share of 4.2%. The structure of the coating consists of phases of mixed crystals α - Al_2O_3 + γ - Al_2O_3 (Kulkarni et al, 2004, pp. 124-137), (Yang et al, 2006, pp.1649-1653).

Figures 5 and 6 illustrate the cross sections and the microstructures of the Al_2O_3 coatings deposited with 800A and 700A.

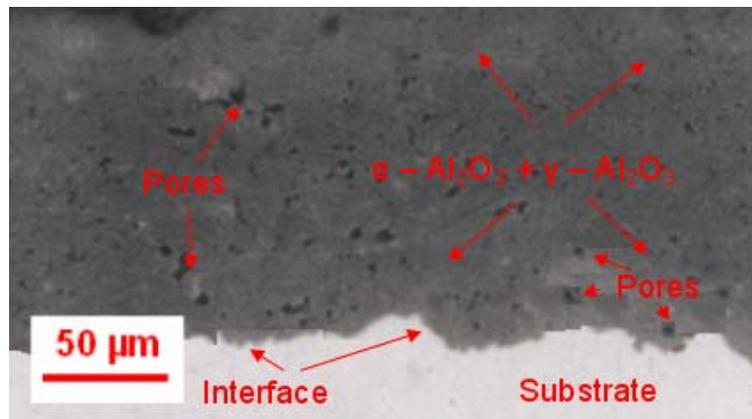


Figure 4 – Cross section and microstructure of the Al₂O₃ (900A) coatings
Рис. 4 – Поперечное сечение и микроструктура Al₂O₃ покрытия (900A)
Слика 4 – Попречни пресек и микроструктура Al₂O₃ превлаке (900 A)

Due to low thermal conductivity of the oxide powder, lower plasma current amperage heats and melts powder particles to a lower extent.

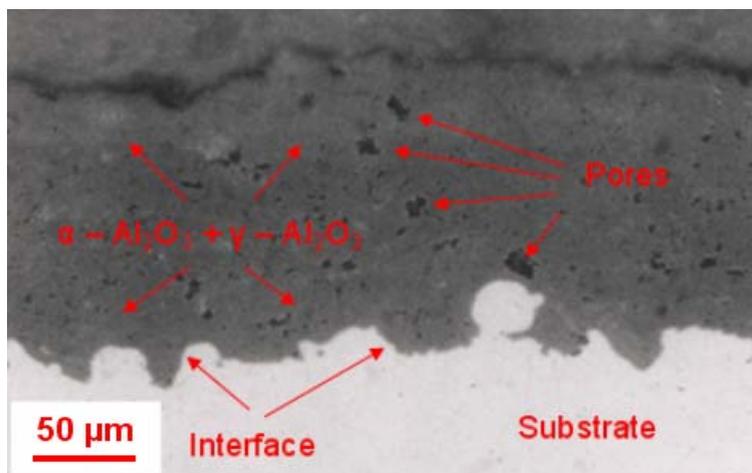


Figure 5 – Cross section and microstructure of the Al₂O₃ (800A) coatings
Рис. 5 – Поперечное сечение и микроструктура Al₂O₃ покрытия (800A)
Слика 5 – Попречни пресек и микроструктура Al₂O₃ превлаке (800 A)

Such semi-melted particles are poorly and improperly deformed in collision with the substrate thus forming pores larger in size and share in the coatings. Coatings with a higher share of pores of irregular shapes and larger pores significantly reduce the cohesive strength of the coating and the coating time and resistance to fatigue friction. Because of their lower density and inhomogeneity, such coatings will wear faster and are unreliable in operation.

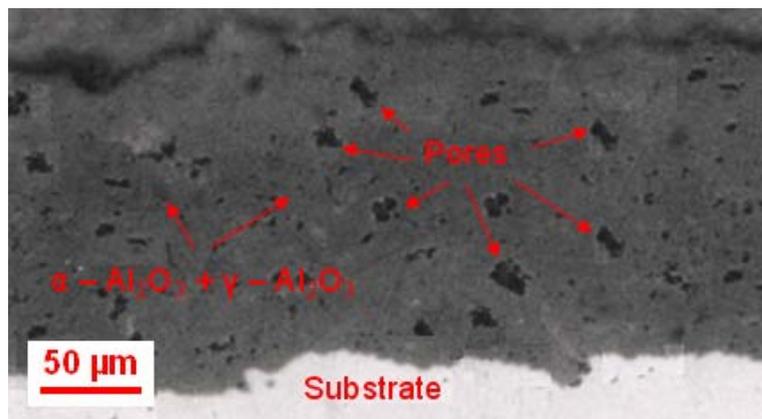


Figure 6 – Cross section and microstructure of the Al₂O₃ (700A) coatings
 Рис. 6 – Поперечное сечение и микроструктура Al₂O₃ покрытия (700A)
 Слика 6 – Попречни пресек и микроструктура Al₂O₃ превилаке (700 A)

In the structure of the coating deposited with 800A, there are small and rough micro pores of irregular shape marked with red arrows. Micro pores in the coating had a size of 10 - 20μm with a total content of 6.4%. The microstructure of the worst coatings deposited with 700A is illustrated in Figure 6. The highest content of micro pores of 8.6% and formed coarse pores over 20μm resulted in the coating having the lowest microhardness, the highest microhardness range and the lowest adhesion strength.

Figure 7 illustrates the surface morphology of the Al₂O₃ coating deposited with 900A and analyzed by the SEM method. On the surface of the Al₂O₃ coating, overlapped particles can be seen, deformed in a characteristic way in collision with the substrate. Drops of molten Al₂O₃ particles cooled to the substrate temperature and formed a disk-like shape. The morphology of a well-molten particle is marked with a red line in the Figure. Complete overlapping of particles is proof that the Al₂O₃ particles are completely molten in plasma and as such allow deposition of layers of coating with thin disks - lamellae on whose edges (interfaces) fine micro pores and precipitates are present. Micro pores in the Figure are rounded in

yellow, while precipitates are in red and green. Full overlapping of deposited particles, due to a large contact area, indicates a good bond between individual particles in a layer deposited in a single plasma gun shot.

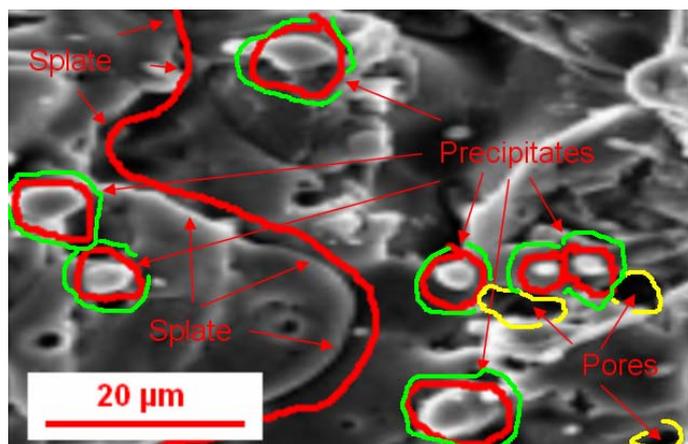


Figure 7 – Morphology surface of the Al_2O_3 coating in the sprayed state, (900A)
 Рис. 7 – Морфология поверхности покрытия Al_2O_3 , нанесенного напылением, (900A)
 Слика 7 – Морфологија површине Al_2O_3 премаке у напрсаном стању (900 A)

It also indicates good bonding between the layers in the coating as well as good adhesion of ceramic particles with the roughened substrate on whose surface liquid Al_2O_3 ceramic drops stay during cooling to the substrate temperature. Such deposition of fully molten powder particles enables the production of coatings of good adhesion / cohesion strength and provides the coating with good sliding properties and wear resistance to friction.

Conclusion

On the basis of the results of examining the mechanical, structural and sliding properties of the Al_2O_3 coatings deposited with 700A, 800A and 900A, it can be concluded:

The results of coatings microhardness were directly related to the applied current amperage. The current of 900A melted injected Al_2O_3 particles in plasma over the complete cross section, which caused the deposited particles to have a large intercontact surface (good cohesive strength) and maximum microhardness. The transfer of heat from the liquid drops of completely molten Al_2O_3 particles to the metal substrate enabled that the coating has the highest strength of bond adhesion - 47 MPa.

Plasma current of 900A resulted in Al₂O₃ powder particles melting completely and, in collision with the substrate, they completely overlapped and formed a coating of the best microstructure. In this way, thin lamellae and interlamellar pores of small size with a small share in the coating were formed in the layers. Micro pores had a size of 5 - 10µm with a mean share of 4.2% in the coating. The structure of the coating consisted of the phases of mixed crystals of α-Al₂O₃ + γ-Al₂O₃.

The coating deposited with 900A was tested on the sealing ring of the water brake paired with its counter pair made of graphite. Testing the sliding properties showed that the Al₂O₃ coating brushed and polished to a mirror on the sealing ring has good properties of sliding fatigue since the ring sealing efficiency is significantly improved and the ring wear is reduced.

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ИСПЫТАНИЯ МЕХАНИЧЕСКИХ СТРУКТУРНЫХ
ХАРАКТЕРИСТИК ОКСИДНОЙ КЕРАМИКИ Al_2O_3 НА
ИЗНОСОСТОЙКОСТЬ ПРИ ТРЕНИИ СКОЛЬЖЕНИЯ

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ВИД СТАТЬИ: оригинальная научная статья

ЯЗЫК СТАТЬИ: английский

Резюме:

Al_2O_3 является твердой оксидной керамикой, которая обладает биоинертными свойствами и отличается высокой

стойкостью на скольжение, благодаря чему она широко применяется в проектировании инженерных компонентов. Цель исследования заключалась в приспособлении APS процесса нанесения плазменного напыления, путем изменения плазменного потока, формирующего слой Al₂O₃, обладающие такими структурными и механическими характеристиками, которые могли бы найти применение в производстве биомедицинских покрытий, как в отдельности, так и в комбинации с гидроксиапатитом, в частности для поверхности сплавов, используемых в изготовлении имплантов. Покрытие нанесено плазменной струей 700, 800 и 900А. Механические характеристики покрытия испытаны в соответствии со стандартами Pratt & Whitney.

Форма частиц порошка и поверхность покрытия испытаны методом SEM. Металлографический анализ внутренних слоев проведен с помощью световой микроскопии.

Лучшие структурные и механические характеристики Al₂O₃ покрытия выявлены у покрытий, нанесенных при 900А, испытана стойкость на скольжение, путем нанесения на уплотнительное кольцо в паре с графитовым кольцом, защищающее гидравлический тормоз и путем шлифования покрытия до зеркального блеска.

Ключевые слова: скольжение, свойство, оксиды, механические свойства, нанесение, покрытие, Al₂O₃.

ИСПИТИВАЊЕ МЕХАНИЧКИХ СТРУКТУРНИХ КАРАКТЕРИСТИКА ОКСИДНЕ КЕРАМИКЕ Al₂O₃ ОТПОРНЕ НА КЛИЗАЊЕ ТРЕЊЕМ

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ВРСТА ЧЛАНКА: оригинални научни чланак

ЈЕЗИК ЧЛАНКА: енглески

Сажетак:

Al₂O₃ је тврда оксидна керамика, хемијски биоинертна са добрим клизним својствима, која је нашла широку примену у пројектовању инжењерских компоненти. Циљ рада био је да се APS процесом са променом плазма струје произведу слојеви Al₂O₃ структурних и механичких карактеристика који ће наћи примену за производњу биомедицинских превлака засебно или у комбинацији са хидроксиапатитом (ХА) на површинама легура које се користе за израду импланата. Превлака је депонована

са плазма струјом 700, 800 и 900 А. Механичке карактеристике превлаке испитане су применом стандарда Pratt & Whitney. Облик површине честица праха и површина превлаке испитана је методом SEM. Металографска анализа унутрашњих слојева испитана је светлосном микроскопијом. Најбоље структурне и механичке карактеристике Al_2O_3 превлаке депоноване са 900 А потврђене су испитивањем клизних својстава превлаке депоновне и полиране до огледала на заптивном прстену упареном са графитним прстеном на воденој кочници.

Кључне речи: клизање, својство, оксиди, механичка својства, депозити, превлаке, Al_2O_3 .

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