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Development of a method for directed modification of thin-film nitride coatings to enhance resistance to corrosion processes of steels and alloys

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Abstract. In recent years, much attention has been paid to research in the field of increasing resistance to external influences, including the corrosion resistance of steels and alloys exposed to aggressive environments or high temperatures. Interest in this area is due to the need to increase the service life of steel products, which will reduce the cost of most technological processes. The most promising methods for improving the strength characteristics, as well as resistance to corrosion degradation, are the application of various protective coatings to steel, among the variety of which one can single out nitride coatings that are highly resistant to corrosion and degradation caused by exposure to aggressive media. Also, one of the ways to increase resistance to external influences is the methods of changing the dislocation density in the near-surface layer, which make it possible to create a barrier layer for the penetration of oxygen and moisture through the protective coating. Based on the proposed corrosion protection methods, this paper considers the possibilities of combining the methods of applying nitride coatings and subsequent ionic modification in order to create a barrier protective layer against corrosion of 316L stainless steel. During the studies conducted, it was found that the magnetron deposition of a nitride coating (TiN) on the surface of stainless-steel leads to a hardness growth by 10-13 %, and the subsequent ion modification, performed by the action of low-energy N²⁺ ions on the nitride coating with different irradiation fluence, leads to an increase in strength by 50-70 % compared to the initial value. As a result of corrosion tests to maintain the stability of strength properties, it was found that the most effective influence of ion irradiation is a fluence of 5×10¹³-10¹⁴ ion/cm², leading not only to maximum hardening, but also to an increase in the corrosion potential, the alteration of which indicates a decrease in the degradation rate.

Keywords: ion modification, TiN thin films, dislocation density, stress resistance, hardness, crack resistance.

1. Introduction

As is known, one of the most common types of steel is 316L stainless steel, which has high mechanical strength, electrical and thermal conductivity, which allows it to be used in a wide range of various practical applications, including operation in aggressive conditions (exposure to aggressive environments, acids, alkalis, high temperatures, etc.) [1-3]. At the same time, the possibility of forming a passivating film on the surface of the steel makes it possible to use this type of steel under aggressive conditions of exposure, including interactions with acids or alkalis [4, 5]. However, the protective film that does not always form on the steel surface leads to complete protection against corrosion processes, and in most cases the degradation of the steel surface occurs through the formation of pitting, accompanied by partial destruction of the surface layer and subsequent volumetric degradation [6, 7].

One of the most promising ways to increase the resistance to degradation processes caused by environmental influences or external influences, including aggressive media, is the use of thin-film coatings based on nitrides or carbides [8, 9]. This modification method is based on a number of technological solutions aimed at applying protective thin-film coatings to metal parts of devices that are subject to corrosion or external influences that can lead to surface degradation and the appear-

ance of destructive inclusions [10, 11]. In the case of using thin-film coatings that are highly resistant to degradation when interacting with most various external factors, this technology makes it possible to increase wear resistance, as well as service life, which entails a reduction in economic costs and energy resources [12, 13].

However, despite the prospects for using thin-film coatings as anti-corrosion protective materials, a number of problems remain, the solution of which will increase the service life of materials, as well as enhance their resistance to mechanical damage and high-temperature heating. One way to solve this problem is to use the methods of ion or radiation modification using low-energy ions or electron radiation [14-16]. This modification is based on the hypothesis that, during the interaction of incident charged particles (ions) with the crystal structure of thin films, energy transfer processes, accompanied by energy transformation of kinetic energy into thermal energy, lead to structural changes in film coatings and their hardening. Hardening effects in this case are associated with changes in dislocation density and size factors, which leads to an increase in resistance to external influences, a decrease in the rate of structural degradation as a result of corrosion or high-temperature heating.

The aim of this study is to explore the prospects for the use of ion modification (low-energy irradiation with heavy ions) to

change the strength properties of nitride coatings, and to determine the effectiveness of the influence of magnetron sputtering of titanium nitride followed by ion modification to increase corrosion resistance in aggressive environments.

2. Materials and methods

Thin-film coatings based on titanium nitride (TiN) obtained by magnetron sputtering on the surface of steel 316L were selected as objects for research. The thickness of the deposited coatings was no more than 500 nm. The thickness determination was carried out using the ellipsometry method.

Surface modification in order to create a barrier layer in the applied coating was carried out by ion irradiation, by irradiation with low-energy N_2^+ ions with an energy of 40 keV and fluences of 10^{13} - 10^{15} ion/cm². The choice of the irradiation fluence is due to the possibility of dislocation density formation as a result of the interaction of incident low-energy ions with the crystal structure of the deposited coating. The formation of the dislocation density in this case is due to the effects of energy transfer with subsequent transformation of the kinetic energy of the incident ions into thermal energy along the ion motion trajectory with the formation of structural changes, initialization of recrystallization processes, and grain crushing. The maximum possible projective range of ions in a film based on titanium nitride is no more than 100-150 nm, while 49% of the total incident ion energy is spent on ionization processes, which indicates that structural changes associated with the processes of electron density alteration (as a result of ionization during the interaction of incident particles with the electronic subsystem), and deformation distortions as a result of the interaction of incident particles with nuclei, leading to the formation of primary knocked-on atoms are equiprobable. In this case, the variation of the irradiation fluence can lead both to hardening processes due to structural distortions and changes in the dislocation density, and to the opposite effects associated with partial sputtering or peeling of the deposited coating at a high concentration of deformation distortions in the structure associated with ion implantation.

The hardness assessment was carried out using the indentation method. The measurements were performed on a Duroline M1 Metkon microhardness tester (Metkon, Turkey). The measurements were carried out using the Brinell hardness method at a maximum load value of 100 N. The measurements were performed in the form of series (at least 25 measurements on the sample), which made it possible to determine the average value of hardness, as well as the values of the measurement error and standard deviation. The hardening effect was calculated by comparing the hardness values of the modified coatings with the hardness value of 316L steel measured under the same conditions.

Corrosion resistance tests were carried out by measuring the corrosion potential (CP) using the corrosion potential versus corrosion current method. The experiments were carried out using a 0.1 M solution of sulfuric acid (H₂SO₄). The measurements were carried out using a three-electrode cell in which the sample was used as the working electrode, the platinum electrode was used as the counter electrode, and the silver chloride electrode was used as the reference electrode.

Measurements were made using a PulmSenc 4+ galvanostat potentiostat, corrosion current was measured using a galvanostat, and corrosion potential was measured using a potentiostat. According to a number of works [17, 18], it is

known that the corrosion potential is directly proportional to the corrosion resistance, i.e. the higher the CP value, the higher the resistance to corrosion and, accordingly, the lower the rate of degradation and corrosion.

3. Results and discussion

One of the important factors determining the modification efficiency of 316L steel is the determination of its strength characteristics, the change of which indicates a positive effect of the proposed surface modifications. The results of this assessment are presented in Figure 1 as a dependence of the change in the hardness values of the samples before and after ion irradiation. As a comparison, the hardness value measured for uncoated 316L steel is 1.53 GPa, which is in good agreement with the data on the strength characteristics of this steel grade. During magnetron deposition of a thin TiN film with a thickness of 500 nm, an increase in hardness from 1.53 ± 0.05 GPa to 1.74 ± 0.06 GPa is observed, which indicates a strengthening by more than 10% compared to the original steel (see data presented in Figure 2a). Such a small increase in hardness during the deposition of a thin film can be due to the fact that the thickness of the applied coating is rather small, and the coating itself, formed by magnetron sputtering, consists of rather large grains (more than 80 nm), which does not provide high resistance to external influences. In the case of ionic modification with a fluence of 10^{13} ion/cm², a more than twofold increase in hardness is observed in comparison with the initial value for steel and by more than 13% in comparison with the applied coating.

The maximum efficiency in hardness increase is achieved by irradiating thin film samples with a fluence of 5×10^{13} ion/cm², for which the hardness value is more than 2.6 GPa, which is more than 70% higher than the hardness value of the original steel and is 6 times higher than the hardness of coated samples. Such an increase in hardness can be explained by the effect of the so-called dislocation hardening associated with a rise in the dislocation density in the modified layer, as well as a decrease in grain size as a result of their crushing and recrystallization processes (see data in Figure 2b) [19, 20].

The dislocation density alteration in the case of irradiated samples is associated with the following processes that occur when the samples are irradiated. Firstly, during the interaction of low-energy ions with the crystal structure, due to the equiprobable contribution of the ionization processes and displacement of atoms, as well as the transformational transition of kinetic energy into thermal energy, the damaged layer structure undergoes large changes associated both with a change in the electron density distribution and with the displacement of atoms from the crystal lattice nodes, which leads to the accumulation of deformation distortions. In this case, the effect of the irradiation fluence or the density of interacting ions with a crystal lattice per unit area plays a very important role. When the calculated values of the diameters of the damaged areas that appear along the ion trajectory are equal to 3-5 nm, the effect of deep overlapping of these areas will be observed at fluences of 5×10^{13} ion/cm² and higher. As a result, a sufficient concentration of deformation distortions and residual stresses associated with the displacement of atoms can accumulate in the structure of the damaged layer due to deep overlap, which can lead to the initialization of recrystallization processes and grain fragmentation, which in turn leads to an increase in dislocation density (see data in Figure 2b).

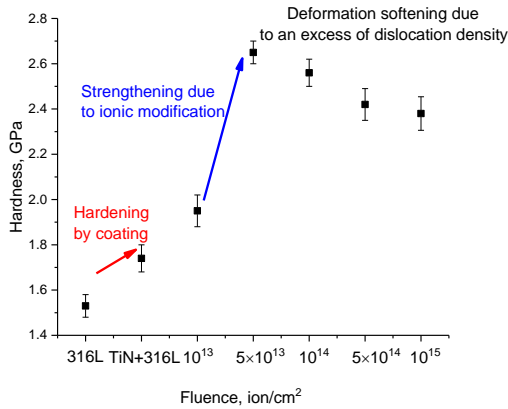


Figure 1. The evaluation results of the change in the hardness value of the samples depending on the modification type (after coating and after ion modification with different fluences)

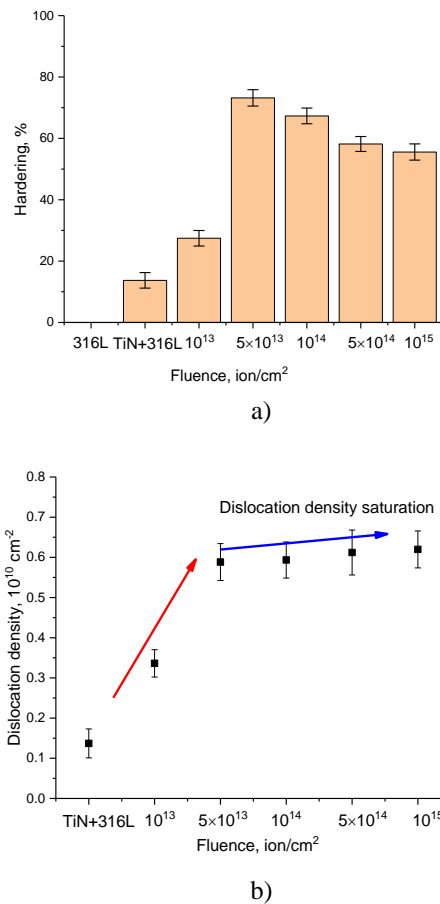


Figure 2. a) Results of the samples' hardening as a result of the deposition of thin films and subsequent ion modification with varying irradiation fluence (the data are presented as a percentage of the strengthening efficiency in comparison with the hardness value for the original 316L steel); b) The evaluation results of the change in dislocation density depending on the irradiation fluence

An increase in the dislocation density, the value of which is inversely proportional to the square of the grain size, leads to the creation of additional obstacles to the propagation of microcracks under external influences, which leads to strengthening and the creation of a modified layer of increased hardness. However, at a high concentration of deformation distortions and residual stresses arising from high irradiation fluences, the effect of supersaturation with dislocations can

occur, which will lead to the appearance of deformed inclusions in the damaged layer, which are in a metastable state, the impact on which can lead to embrittlement or partial peeling of the near-surface layer. In this case, hillocks can form on the surface of the coatings, which are characteristic of the deformation extrusion of the volume onto the film surface due to structural distortions or the implantation effect.

Figure 3 reveals the data on the change in the corrosion potential value obtained by analyzing electrochemical corrosion curves using a potentiostat-galvanostat. As is known, an upward change in the corrosion potential (shift to the passive region) is due to an increase in degradation resistance and a decrease in the rate of formation of corrosion inclusions in the form of pits or passive films. According to the data obtained, the most dramatic increase in the value of the corrosion potential is observed for modified samples with fluences above 10¹³ ion/cm², for which, as shown earlier (see data in Figures 1-2), an increase in hardness and hardening values is observed. At the same time, the corrosion potential increase in the case of unmodified coatings is no more than 4.5%, while for samples with ionic modification, the corrosion potential increase is more than 20%. Such a difference in the values of corrosion potentials may be due to the fact that the modified coatings have a barrier layer consisting of dislocations and defective inclusions in the form of small grains, which leads to an increase in degradation resistance during corrosion.

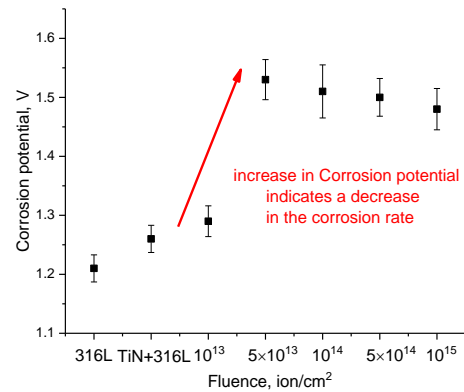


Figure 3. Dependence of the change in corrosion potential on the modification type

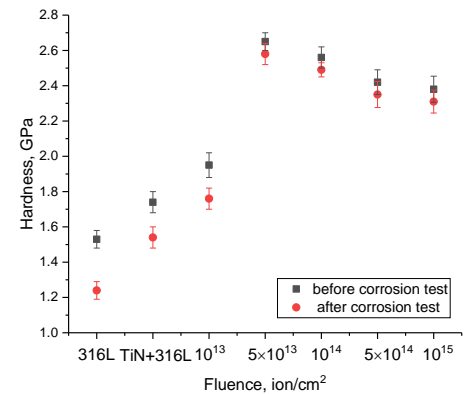


Figure 4. Comparative analysis of changes in hardness before and after corrosion testing

Figure 4 shows the data on the dependence of the change in the hardness values of the test samples before and after corrosion tests, reflecting the dependence of the change in hardness and the degree of softening of the samples in the case of corrosion and degradation. As can be seen from the data presented, the most pronounced differences in hardness values before and after corrosion tests are observed for unmodified steel and coated samples. At the same time, in the case of samples modified by ion irradiation at fluences above 10^{13} ion/cm², the differences in hardness values are no more than 2.5-3%, which indicates a high resistance of these samples to degradation of strength properties and softening during exposure to aggressive media.

Figure 5 shows the data on changes in the values of the corrosion potential and the softening of TiN films after corrosion tests. The softening value was calculated based on a comparison of data on the change in hardness values before and after corrosion testing and is presented as a percentage. In general, the presented data have a good correlation between changes in the corrosion potential and the softening value, which also characterizes the resistance of materials to corrosion processes. An increase in the corrosion potential, indicating a decrease in the rate of degradation and the formation of pits, leads to a decrease in the spread in the values of hardness before and after corrosion tests, which in turn indicates an increase in resistance to degradation. In this case, it is also worth to note that the maximum efficiency of stability and degradation resistance growth is observed for the case when the samples were irradiated with a fluence of 5×10^{13} ion/cm², for which, according to the data on hardness changes, the maximum increase in strength parameters is observed. In this case, despite the decrease in the value of the corrosion potential with a subsequent increase in the irradiation fluence, the amount of disordering remains practically unchanged, which indicates that the effect of hardening and an increase in the corrosion resistance of the modified films is retained even with a decrease in hardness values in comparison with the maximum achievable effect of hardening with ionic modification.

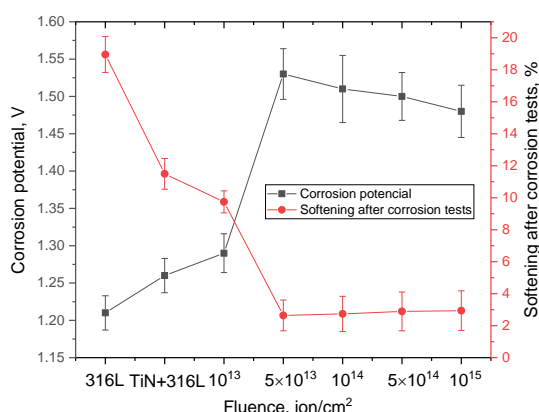


Figure 5. Comparison of data on changes in the potential of corrosion and softening after corrosion tests

Analyzing the data obtained, it can be concluded that the use of methods of magnetron sputtering of thin TiN films leads to an increase in degradation resistance when interacting with sulfuric acid by 10%, while ion modification of thin

films leads to an almost 10-fold increase in resistance to corrosion and degradation. This effect is due to the presence of a high dislocation density, which leads to the creation of a barrier layer near the surface, which creates obstacles for the penetration of oxygen ions and the subsequent formation of oxide inclusions or passive films in the surface layer, an increase in the thickness of which characterizes corrosion processes.

4. Conclusions

In this paper, the prospects of using ionic modification to increase resistance to the external influences of titanium nitride-based thin films obtained by magnetron sputtering have been studied. N₂⁺ ions with an energy of 40 keV and fluences of 10^{13} - 10^{15} ion/cm² were chosen for modification. During experiments, it was found that an increase in the irradiation fluence leads to an increase in the hardness values of coated steel from 1.74 GPa to 2.38-2.65 GPa, depending on the irradiation fluence, while the steel hardness in the initial state was 1.53 GPa. In this case, the strengthening of the films is primarily due to a change in the dislocation density, as well as a change in the size factor associated with the recrystallization and grain fragmentation processes caused by irradiation.

In the course of corrosion tests, it was found that the modification of TiN thin films by ion irradiation leads to an increase in the corrosion potential and, as a result, a decrease in the corrosion rate and degradation of strength properties. The established dependences of the change in the value of the corrosion potential and the resistance to softening of the near-surface layer with the modified coating showed good agreement between the change in the corrosion potential value and the increase in stability.

Based on the data obtained, it was found that the most effective way to modify thin TiN films in order to increase resistance to external influences and increase corrosion resistance in an acidic environment is to irradiate films with fluences of 5×10^{13} - 10^{14} ion/cm². At these irradiation fluences, the most effective improvement in the properties of anticorrosion coatings is observed, while an increase in the irradiation fluence above 10^{14} ion/cm² leads to a decrease in stability, which is due to the effects of supersaturation of the damaged layer with dislocations, which leads to the occurrence of deformation stresses and distortions in the structure of the coating, and also reduces the stress resistance of coatings to external influences.

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Болаттар мен қорытпалардың коррозиялық процестеріне төзімділігін арттыру үшін жұқа пленкалы нитридті жабындарды бағытталған модификациялау әдісімен әзірлеу

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Андатпа. Соңғы жылдары коррозиялық ортаға немесе жоғары температураға ұшыраған болаттар мен қорытпалардың коррозияға төзімділігін қоса алғанда, сыртқы әсерлерге төзімділікті арттыру саласындағы зерттеулерге көп көңіл бөлінді. Бұл бағытқа деген қызығушылық болат өнімдерінің қызмет ету мерзімін арттыру қажеттілігімен байланысты, бұл көптеген технологиялық процестердің өзіндік құнын төмендетеді. Беріктік сипаттамаларын, сондай-ақ коррозиялық деградацияға төзімділікті арттырудың ең перспективалы әдістері болатқа әртүрлі қорғаныс жабындарын қолдану болып табылады, олардың арасында агрессивті орталардың әсерінен коррозияға және деградацияға жоғары төзімділігі бар нитридті жабындарды ажыратуға болады. Сондай-ақ, сыртқы әсерлерге төзімділікті арттырудың бір әдісі-қорғаныс қабаты арқылы оттегі мен ылғалдың енуіне тосқауыл қабатын құруға мүмкіндік беретін беткі қабаттағы дислокациялық тығыздықты өзгерту әдістері. Коррозиядан қорғаудың

ұсынылған әдістеріне сүйене отырып, бұл жұмыс 316L тот баспайтын болаттан тосқауыл қорғаныш қабатын құру мақсатында нитридті жабындарды қолдану әдістерін және кейіннен иондық модификациялауды біріктіру мүмкіндіктерін қарастырды. Жүргізілген зерттеулер тот баспайтын болаттың бетіне нитридті жабынды (TiN) магнетронды қолдану қаттылықтың 10-13% жоғарылауына әкелетінін анықтады, ал төмен энергиялы N²⁺ иондарының әртүрлі сәулелену флюенсі бар нитридті жабынға әсер етуі арқылы жасалған кейінгі иондық модификация бастапқы мәнмен салыстырғанда беріктіктің 50-70%-ға артуына әкеледі. Беріктік қасиеттерінің тұрақтылығын сақтауға арналған коррозиялық сынақтардың нәтижесінде иондық сәулеленудің ең тиімді әсері 5×10¹³-10¹⁴ ион/см² флюенсі болып табылатыны анықталды, бұл максималды беріктікке ғана емес, сонымен қатар коррозия потенциалының жоғарылауына әкеледі, оның өзгеруі деградация жылдамдығының төмендеуін көрсетеді.

Негізгі сөздер: иондық модификация, TiN жұқа пленкалар, дислокация тығыздығы, стресске төзімділік, қаттылық, сынуға төзімділік.

Разработка способа направленной модификации тонкопленочных нитридных покрытий для повышения сопротивляемости коррозионным процессам сталей и сплавов

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Аннотация. В последние годы большое внимание уделяется исследованиям в области повышения устойчивости к внешним воздействиям, включая коррозионную стойкость сталей и сплавов, подвергающихся эксплуатации в условиях воздействия агрессивных сред или высоких температур. Интерес к данному направлению обусловлен необходимостью повышения сроков эксплуатации стальных изделий, что позволит снизить себестоимость большинства технологических процессов. Наиболее перспективными методами повышения прочностных характеристик, а также устойчивости к коррозионной деградации является нанесение на сталь различных защитных покрытий, среди многообразия которых можно выделить нитридные покрытия, обладающие высокой устойчивостью к коррозии и деградации, вызванной воздействием агрессивных сред. Также одним из способов повышения устойчивости к внешним воздействиям, являются методы изменения дислокационной плотности в приповерхностном слое, позволяющие создать барьерный слой для проникновения кислорода и влаги сквозь защитное покрытие. Основываясь на предлагаемых методах защиты от коррозии в данной работе рассмотрены возможности совмещения методов нанесения нитридных покрытий и последующей ионной модификации с целью создания барьерного защитного слоя от коррозии нержавеющей стали 316L. В ходе проведенных исследований было установлено, что магнетронное нанесение нитридного покрытия (TiN) на поверхность нержавеющей стали приводит к повышению твердости на 10-13%, а последующая ионная модификация, выполненная путем воздействия низкоэнергетических ионов N²⁺ на нитридное покрытие с различным флюенсом облучения приводит к повышению прочности на 50-70% в сравнении с исходным значением. В результате коррозионных испытаний на сохранение стабильности прочностных свойств было установлено, что наиболее эффективным воздействием ионного облучения является флюенса 5×10¹³-10¹⁴ ион/см², приводящие не только к максимальному упрочнению, но и повышению потенциала коррозии, изменение которого свидетельствует о снижении скорости деградации.

Ключевые слова: ионная модификация, TiN тонкие пленки, плотность дислокаций, стрессоустойчивость, твердость, сопротивляемость разрушению.

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