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Combined Electron-Ion-Plasma Treatment of 40Cr Steel Surface

Yu. F. Ivanov, Yu. Kh. Akhmadeev, I. V. Lopatin, O. V. Krysina, E. A. Petrikova

Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences (2/3 Akademicheskii Ave., Tomsk 634055, Russian Federation)

Abstract. In the industry of most developed countries, complex alloying as a surface layer saturation with metal and gas atoms in a certain sequence is extensively used. This study identifies and analyzes the changes in the elemental and phase composition, defect substructure, mechanical (microhardness), and tribological (wear resistance and friction ratio) properties of alloyed carbon steel after complex treatment, consisting of surface layer saturation with Al atoms and subsequent nitriding. We studied 40Cr steel. Its initial structure contains plate-like ferrite and pearlite grains. A TRIO system with a 600×600×600 mm^3 vacuum chamber was used for complex alloying. The system was equipped with a control module for electron-ionic treatment. Aluminizing lasted for 4 hours at 963 K. The electric arc evaporator cathode was made of A7 aluminum alloy (98.8 % Al). Subsequent nitriding of the aluminized layer lasted for 2 hours at 803 K. It was found that such treatment results in a modified surface layer up to 70 μm thick. The complex alloying of steel forms multiphase submicro- and nanostructures with Al nitrides, Fe and Cr nitrides, and aluminides. We found that steel hardness is greatest at the modified surface. It exceeds the initial hardness by 300 %. Complex alloyed steel is less resistant to dry friction.

Keywords: 40Cr steel, complex treatment, aluminizing, nitriding, structure, phase composition, hardness, wear resistance, friction ratio

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Оригинальная статья

Комплексная электронно-ионно-плазменная обработка поверхности стали 40Х

Ю. Ф. Иванов, Ю. Х. Ахмадеев, И. В. Лопатин, О. В. Крысина, Е. А. Петрикова

Институт сильноточной электроники СО РАН (Россия, 634055, Томск, пр. Академический 2/3)

Аннотация. Комплексное легирование, сочетающее в определенной последовательности насыщение поверхностного слоя материала атомами металлов и газов, в настоящее время широко используется в большинстве промышленно развитых стран мира. Настоящая работа посвящена выявлению и анализу закономерностей изменения элементного и фазового состава, дефектной субструктуры, механических (микротвердость) и трибологических (износостойкость и коэффициент трения) свойств легированной углеродистой стали, подвергнутой комплексной обработке, сочетающей насыщение поверхностного слоя образцов атомами алюминия и последующее азотирование. В качестве материала исследования использована сталь 40Х, имеющая в исходном состоянии структуру, представленную зернами феррита и зернами перлита пластинчатой морфологии. Комплексное модифицирование осуществляли в едином вакуумном пространстве на установке «ТРИО» с размерами камеры 600×600×600 мм^3, дооснащенной блоком коммутации для реализации элионного (электронного и ионного) режима обработки. Алитирование проводили при температуре 963 К в течение 4 часов. Катод электродугового испарителя был изготовлен из алюминиевого сплава А7 (98,8 % Al). Последующее азотирование алитированного слоя проводили при температуре 803 К в течение 2 часов. Установлено, что в результате комплексной обработки формируется модифицированный слой толщиной до 70 мкм. Показано, что комплексное модифицирование стали сопровождается формированием многофазного субмикро- и наноструктурного состояния, содержащего нитриды алюминия, нитриды и алюминиды железа и хрома. Установлено, что твердость стали максимальна на поверхности модифицирования и превышает твердость исходного материала в три раза. Износостойкость стали в условиях сухого трения после комплексного модифицирования снижается.

Ключевые слова: сталь 40Х, комплексная обработка, алитирование, азотирование, структура, фазовый состав, твердость, износостойкость, коэффициент трения

Финансирование: Исследование выполнено при финансовой поддержке РФФИ и Госкорпорации «Росатом» в рамках научного проекта № 20-21-00111.

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Introduction

There are many options of controlling the surface layer structure and properties of metals. The most promising methods combine impacting the defect substructure, chemical, and phase composition by introducing elements into the surface layer. Ion implantation [1 – 3], using laser beams [4], gas-phase saturation [5], diffusion saturation in liquid metal solutions [6, 7], liquid phase saturation [8, 9], irradiation with high energy ion beams [10], alloying during steel making [11], high-temperature nitriding in pure nitrogen [12], and high-temperature saturation with a saturating coating [13] are used. With regard to making surface alloys, complex alloying as a surface layer saturation with metal and gas atoms in a certain sequence is of particular interest. In most industrialized countries, complex plasma surface treatments which change the material structure and properties are extensively used [14 – 19]. Single vacuum chamber processes are still under development [20 – 23].

Statement of problem. This study analyzes the complex treatment of carbon-alloyed steel in a single vacuum chamber combining diffuse saturation of the surface layer with aluminum and nitrogen.

Materials and Methods

We used 40Cr steel (composition: 0.36 – 0.44 % C; 0.17 – 0.37 % Si; 0.5 – 0.8 % Mn; up to 0.3 % Ni; 0.8 – 1.1 % Cr; up to 0.035 % S; up to 0.035 % P; up to 0.3 % Cu; the rest in Fe (wt. % are indicated)) and treated polished specimens 5 mm thick, 12 mm dia. A TRIO ion-plasma system with a 600×600×600 mm^3 vacuum chamber was used [17, 18]. The chamber was evacuated by a turbomolecular pump with 500 l/s capacity. The system has two generators producing gas (PINK module) and metallic (arc evaporator) plasma. The arc evaporator cathode was made of the A7 aluminum alloy (98.8 % Al). The system supports electron-ion beam treatment. For this, the discharge is alternatively switched between the main anode (chamber) and the processed part. It ensures efficient sample heating by means of the electron component of plasma without ion etching of the surface. The sample surface was cleaned by ions from plasma in between electron heating sessions. The bias voltage for this treatment was selected, in order to achieve minimal required surface cleaning, rather than maintaining a required temperature. The samples were attached to a holder in the center of the vacuum chamber at the plasma sources axis, in such a way that the holder was at 60° to each plasma source. The samples were on the front side of the holder. Temperature was measured using a type K thermocouple installed in a quartz tube on the sample holder. The sample temperature during aluminizing (963 K) was set to be slightly higher than the aluminum melting temperature (950 K). Before aluminizing, the samples were etched with 700 eV argon ions for 10 min. Then they were heated with argon plasma (electron heating) to the required temperature (963 K). The ion etching and electron heating alternated at 10 – 50 Hz. The table lists the treatment modes of the 40Cr steel samples where: p_{Ar} is the argon pressure; I_{PINK} and I_{arc} are the discharge currents in the PINK plasma generator and arc evaporator; U_{cm} is the negative bias for ion etching; k_{el} is the fraction of time for electron etching; t is the process duration; and T is the temperature of the samples during the process.

We applied X-ray phase analysis, optical, scanning, and transmission electron microscopy, in order to study the structure, elemental, and phase composition of the treated steel. The following mechanical and tribological properties were measured: microhardness (indenter load: 0.5 N); wear resistance; and friction ratio. The tribological tests (dry friction) were conducted at room temperature as follows: counter body – 6 mm diameter SiC ball; wear track diameter – 4 mm; load – 5 N; and track length – 2,000 m.

Results and Discussion

Before treatment the 40Cr steel structure included plate pearlite grains and ferrite grains. A dislocation substructure (chaotically distributed dislocations) was ob-

Treatment modes of 40Cr steel samples

Режимы обработки образцов стали 40Х

p_{Ar}, Pa	I_{PINK}, A	I_{arc}, A	U_{cm}, V	t, h	T, K	k_{el}, %
Aluminizing						
0.20	45	45	300	4	963	27
Nitriding						
0.65	45	0	300	2	803	22

served in the ferrite plates within the pearlite colonies and ferrite grains. Such structures have been analyzed in detail in many works [24, 25] and will not be covered in this paper.

The modified layer thickness (measured by metallographic etching) reaches its max value of about 30 μm after a complex treatment combining aluminizing and subsequent nitriding. The X-ray spectral microanalysis revealed a monotonic decrease of the aluminum concentration in the surface layer of steel (Figure 1). The concentration of chromium in the aluminized layer remains virtually without change and matches the Cr concentration in the sample body. It suggests that the aluminizing process used forms an aluminum-saturated layer, not an aluminized surface layer.

X-ray phase analysis indicated the formation of a structure containing an α iron solid solution, Fe_4N, Fe_3N iron nitrides, and an AlN aluminum nitride (Figure 2) in the surface layer after complex alloying. The total fraction of the nitride phases in the surface layer by volume was 98 %. Of this: AlN is 2 %, and Fe_3N is 20 %.

Using transmission electron diffraction microscopy, we found that a polycrystalline structure with 1.0 – 2.5 μm grain size forms in the surface layer. The second phase particles are along the boundaries and within the grain volume. Darkfield analysis showed that particles of iron and chromium carbonitrides, Al_2Cr chromium aluminides, and Al_2Fe iron aluminides are formed along the α iron grain boundaries after the complex alloying. The particles are mostly globular. The sizes range

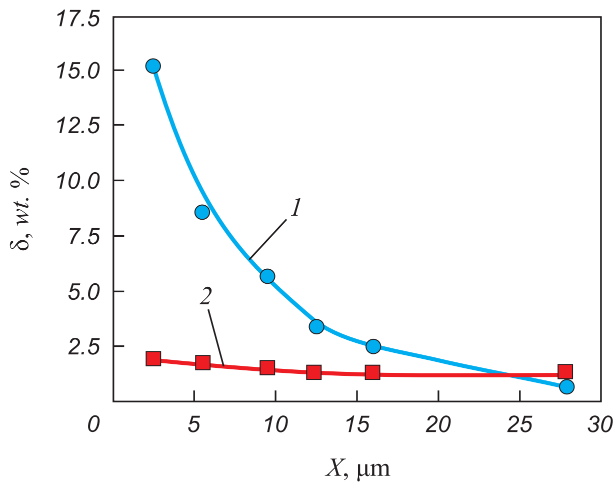

Figure 1. Dependence of aluminum (*1*) and chromium (*2*) concentrations on distance from aluminazing surface of 40Cr steel

Рис. 1. Зависимость концентрации алюминия (*1*) и хрома (*2*) от расстояния от поверхности алитирования стали 40Х

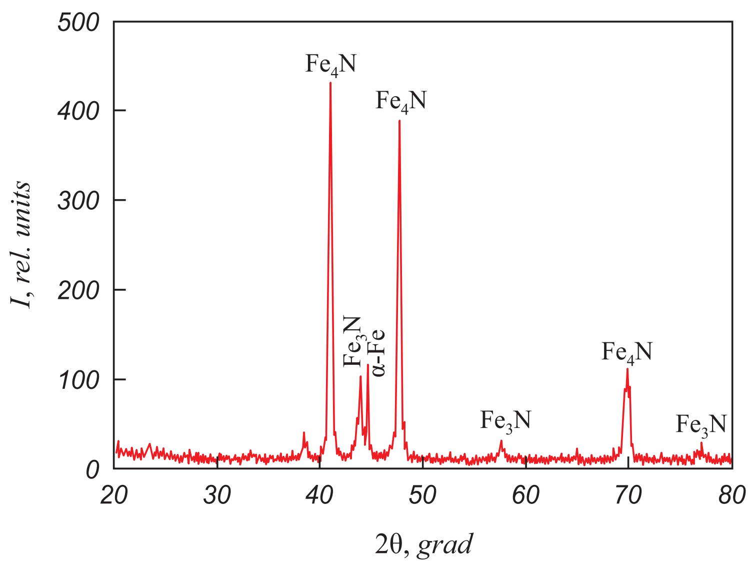

Figure 2. X-ray section obtained from 40Cr steel surface after complex treatment (aluminazing and consequent nitriding)

Рис. 2. Фрагмент рентгенограммы, полученной с поверхности стали 40Х, подвергнутой комплексной обработке (алитирование и последующее азотирование)

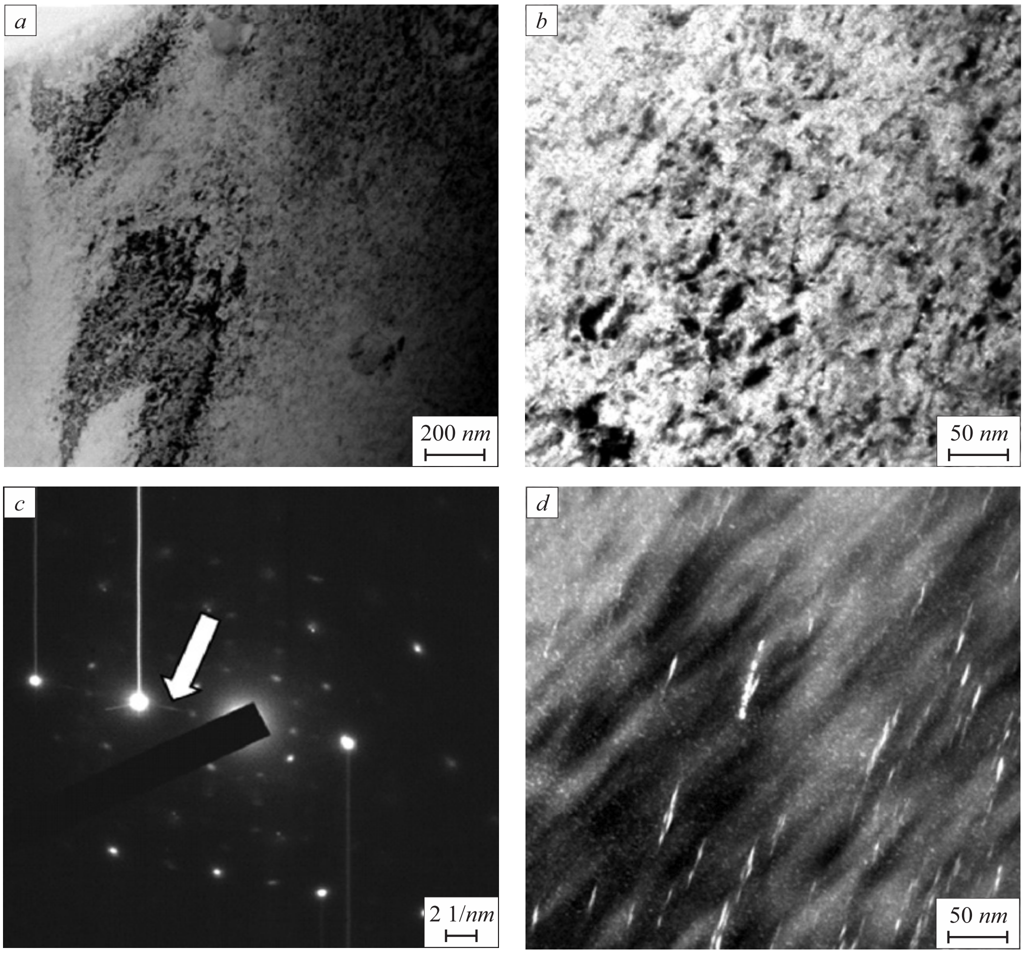

Figure 3. TEM images of second phase particles formed in the grain bulk of 40Cr steel after complex treatment:
a, *b* – bright field; *c* – microdiffraction pattern (arrow indicates the reflection in which dark field was obtained);
d – dark field obtained in [330] Al_9Cr_4 reflection

Рис. 3. ПЭМ-изображение частиц второй фазы, формирующихся в объеме зерен стали 40Х в результате комплексной обработки:
а, *b* – светлое поле; *c* – микроэлектронограмма (стрелкой указан рефлекс, в котором получено темное поле);
d – темное поле, полученное в рефлексе [330] Al_9Cr_4

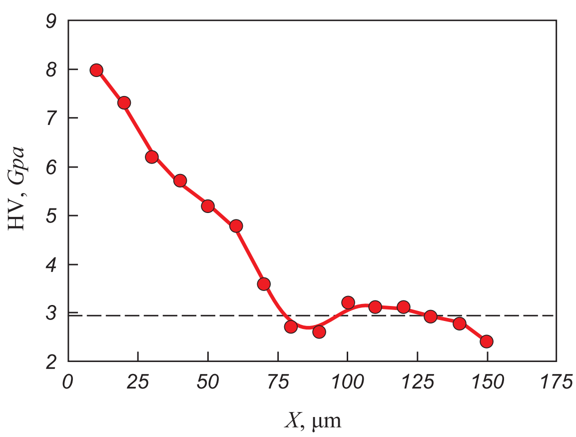

Figure 4. Microhardness profile of 40Cr steel samples after complex treatment including aluminazing and consequent nitriding (dotted line – microhardness of steel in initial state)

Рис. 4. Профиль микротвердости образцов стали 40Х, подвергнутых комплексной обработке, сочетающей алитирование и последующее азотирование (штриховой линией обозначена твердость стали в исходном состоянии)

from 80 to 100 nm. Spiky particles were found in the volume of the grains (Figure 3). Electron diffraction image analysis suggests that these particles are Al_9Cr_4 chromium aluminides. Their cross size varies from 1.2 to 2.5 nm, and the longitudinal size is from 15 to 32 nm (Figure 3, *d*).

It has been shown that treatment reduces wear resistance (Figure 5). One possible reason is brittle damage to the surface layer. Another one is the solid phase particles. They act as an abrasive powder which results in wearing. The friction ratio (μ) is virtually unchanged and ranges from 0.49 to 0.51.

Conclusions

We studied the complex alloying of 40Cr alloyed steel samples. This included aluminizing and subsequent nitriding in a single 600×600×600 mm^3 vacuum chamber of the TRIO ion-plasma system. Aluminizing was at 963 K

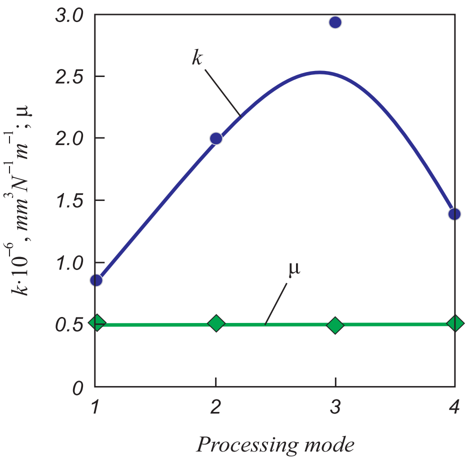

Figure 5. Dependences of friction coefficient (μ) and wear rate (*k*) on the treatment mode of 40Cr steel

Рис. 5. Зависимости коэффициента трения (μ) и параметра износа (*k*) от режима обработки стали 40Х

for 4 h. Subsequent nitriding of the aluminized layer was at 803 K for 2 h. The system incorporates two generators producing gas (PINK module) and metallic (electric arc evaporator) plasma. The arc evaporator cathode is made of the A7 aluminum alloy (98.8 % Al). The system supports ion-electron treatment for efficient sample heating by the electronic component of the plasma without ion etching of the surface. It was found that before aluminizing, 40Cr steel is a polycrystalline substance formed by plate ferrite and pearlite grains. It was shown that complex treatment forms a surface layer with a grain size in the range of 1.0 – 2.5 μm. The layer contained a multiphase microtructure with aluminum nitrides, nitrides, and iron and chromium aluminides. It was found that complex alloying of steel forms a hardened surface layer up to 70 μm thick. Steel hardness is greatest at the surface and exceeds base metal hardness by 300 %. Complex alloyed steel is less resistant to dry friction.

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Information about the authors

Yurii F. Ivanov*, Dr. Sci. (Phys.-Math.), Chief Researcher of the Laboratory of Plasma Emission Electronics,* Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences
ORCID: 0000-0001-8022-7958
E-mail: yufi55@mail.ru

Yurii Ch. Akhmadeev*, Cand. Sci. (Eng.), Leading Researcher of the Laboratory of Plasma Emission Electronics,* Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences
ORCID: 0000-0001-8084-2854
E-mail: ahmadeev@opee.hcei.tsc.ru

Il'ya V. Lopatin*, Cand. Sci. (Eng.), Senior Researcher of the Laboratory of Plasma Emission Electronics,* Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences
ORCID: 0000-0002-5192-871X
E-mail: lopatin@opee.hcei.tsc.ru

Ol'ga V. Krysina*, Cand. Sci. (Eng.), Senior Researcher of the Laboratory of Plasma Emission Electronics,* Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences
ORCID: 0000-0001-9265-2644
E-mail: krysina_82@mail.ru

Elizaveta A. Petrikova*, Junior Researcher of the Laboratory of Plasma Emission Electronics,* Institute of High Current Electronics, Siberian Branch of the Russian Academy of Sciences
ORCID: 0000-0002-1959-1459
E-mail: elizmarkova@yahoo.com

Сведения об авторах

Юрий Федорович Иванов*, д.ф.-м.н., главный научный сотрудник лаборатории плазменной эмиссионной электроники,* Институт сильноточной электроники СО РАН
ORCID: 0000-0001-8022-7958
E-mail: yufi55@mail.ru

Юрий Халяфович Ахмадеев*, к.т.н., ведущий научный сотрудник лаборатории плазменной эмиссионной электроники,* Институт сильноточной электроники СО РАН
ORCID: 0000-0001-8084-2854
E-mail: ahmadeev@opee.hcei.tsc.ru

Илья Викторович Лопатин*, к.т.н., старший научный сотрудник лаборатории плазменной эмиссионной электроники,* Институт сильноточной электроники СО РАН
ORCID: 0000-0002-5192-871X
E-mail: lopatin@opee.hcei.tsc.ru

Ольга Васильевна Крысина*, к.т.н., старший научный сотрудник лаборатории плазменной эмиссионной электроники,* Институт сильноточной электроники СО РАН
ORCID: 0000-0001-9265-2644
E-mail: krysina_82@mail.ru

Елизавета Алексеевна Петрикова*, младший научный сотрудник лаборатория плазменной эмиссионной электроники,* Институт сильноточной электроники СО РАН
ORCID: 0000-0002-1959-1459
E-mail: elizmarkova@yahoo.com

Contribution of the authors

Yu. F. Ivanov – studies of steel structural-phase state by methods of conducting electron diffraction microscopy, discussion of the results, writing the article.

Yu. Kh. Akhmadeev – complex processing of steel, discussion of the results, writing the article.

I. V. Lopatin – complex processing of steel, discussion of the results, writing the article.

O. V. Krysina – studies of steel structural-phase state by x-ray diffraction, mechanical testing of steel (definition of microhardness), discussion of the results, writing the article.

E. A. Petrikova – studies of steel structural-phase state by optical and scanning electron microscopy, tribological testing of steel (determination of wear resistance and friction coefficient), discussion of the results, writing the article.

Вклад авторов

Ю. Ф. Иванов – исследования структурно-фазового состояния стали методами провечивающей электронной дифракционной микроскопии, обсуждение полученных результатов, написание статьи.

Ю. Х. Ахмадеев – комплексная обработка стали, участие в обсуждении полученных результатов, написание статьи.

И. В. Лопатин –комплексная обработка стали, участие в обсуждении полученных результатов, написание статьи.

О. В. Крысина – исследования структурно-фазового состояния стали методами рентгенофазового анализа, механические испытания стали (определение микротврдости), обсуждение полученных результатов, написание статьи.

Е. А. Петрикова – исследования структурно-фазового состояния стали методами оптической и сканирующей электронной микроскопии, трибологические испытания стали (определение износостойкости и коэффициента трения), обсуждение полученных результатов, написание статьи.

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