Technology of electrodepositing and properties of indium-containing chromium alloys

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Abstract The experimental results of the influence of electrolyte composition and electrodepositing regimes on the properties of galvanic gradient indium-containing composite coatings based on chromium are presented. The dependences of micro hardness, coefficient of friction and wear resistance of coatings on electrodepositing regimes and electrolyte composition were studied. The effect of coating thickness on properties of gradient coatings was investigated. It is shown that the gradient alloy chromium – indium – molybdenum – vanadium – boron nitride (elbor), obtained by electrolytic deposition, has a high wear resistance and is far superior to the wear resistance of the coating obtained from standard chromium sulphate electrolyte.

1. Introduction

Galvanic chromium plating has been widely used while producing details and instruments, to increase micro-hardness and wear resistance. Chromium coatings possess high micro-hardness (to 13 GPa) and wear resistance, they also have good anti-frictional characteristics and high corrosion resistance. More often, chromium plating is performed from standard sulphate electrolytes based on hexavalent chromium. Main disadvantage of these methods of obtaining is their ecological danger. Besides, the process of chroming has low dispensability and current output. In chroming, hydrogen absorption by metal (hydrogen uptake) occurs resulting in decreased fatigue strength. To improve properties of chromium coatings and increase chroming rate, supplements are introduced in electrolyte composition, ultrasound is used, impulse and reversible current is used [1, 2]. Chroming process from electrolytes based on trivalent chromium is ecologically more pure than that from standard sulphate electrolytes based on hexavalent chromium. These electrolytes have higher current output and, correspondingly, chromium steels absorb less hydrogen. However, mechanical properties of coatings obtained from electrolytes based on trivalent chromium is ecologically more pure than that from standard sulphate electrolytes based on hexavalent chromium. These electrolytes have higher current output and, correspondingly, chromium steels absorb less hydrogen. However, mechanical properties of coatings obtained from electrolytes based on trivalent chromium are inferior to those obtained from electrolytes based on hexavalent chromium [3, 4]. To increase wear resistance and hardness, composite electrochemical coatings (CEC) have been made, basing on chromium. However, obtaining CEC based on chromium is associated with a number of difficulties due to abundant hydrogen excretion on cathode; because of this the content of the second phase in CEC based on chromium does not exceed 2–3 %, as a rule. Nevertheless, even insignificant amount of the second phase in the coating can essentially improve its properties due to restructuring [5]. Use of chromium-based nano-composite galvanic coatings gives still more effect. Such coatings can be obtained by the way of introducing ultradisperse powder of
aluminum oxide in the chroming electrolyte. Herewith, fatigue of base metal decreases, and adhesion increases, along with the increase of micro-hardness [6-8]. Diamond-cluster galvanic coatings possess wear resistance 2.5–5.7 times more, compared with coatings obtained by the routine hard chroming plating, with their micro-hardness increasing to 14 GPa. Micro-hardness of the coatings modified by nanotubes increases to 1.5–3 times. Simultaneously, porosity and unevenness of distributing the coating on the surface of a detail decrease [9–11].

The given work is devoted to methods of improving properties of chromium coatings by the way of alloying, making the second phase in them, obtaining gradient coatings with increased content of indium in the upper surface.

2. Experiment: materials and methods

Samples were made of unalloyed carbonaceous steel with carbon content 1 %. Before coating, all samples were ground, polished, degreased with Viennese lime, pickled in 20% solution of sulfuric acid, washed first in tap, then in distilled water.

Making electrolytes, distilled water and chemically pure chemicals were used. To obtain a composite electrochemical coating, cubic boron nitride (cBN) with size of grains of base fraction (95%) 1 µm and finer was introduced into the electrolyte composition. Galvanic plating was performed in baths with volume 0.5, 1 and 3 liter, provided with thermo stating system, a propeller blade mixer, and sources with current stabilization systems.

To increase wear resistance of the chromium coatings, chromium alloying with indium, as well as chromium-molybdenum and chromium-molybdenum-vanadium micro-alloys were used. To precipitate chromium-molybdenum alloys, stable sulphate electrolytes have found applications in industry. Such coatings have wear resistance about 2 times higher, compared with pure chromium ones, also their protective ability is better, inner tensions are lower, and micro-hardness is higher. Vanadic acid introduced into chroming electrolytes contributes to increased current output, finer grains of the coating, as well as eliminates negative influence of increased concentration of sulphate ions due to their binding into sulphate-vanadic complex. Further improvement of the coatings was performed by the way of additional alloying of chromium-molybdenum-vanadium coatings with indium. Chromium, molybdenum and vanadium on the one hand, and indium on the other hand, are metals possessing a number of opposite properties. If Cr-Mo-V coatings have high melting temperature and hardness, rather often they possess significant inner tensions (though lower than chromium does) and are easily hydro treated. Unlike them, indium is fusible, soft, plastic, and its coatings are not hydro treated. Besides, indium possesses the lowest friction coefficient among all metals – 0.05–0.06 [12]. Use of such materials was aimed at removing these disadvantages and obtaining anti-frictional and wear-resistant alloys with improved mechanical properties. In order to use more fully advantages of each component of the alloy, a gradient coating with increased indium content in upper layers was made on their base. In gradient coatings, the coating composition varies smoothly across the coating thickness. Correspondingly, micro-hardness of coatings and friction coefficient (unlike multi-layer coatings which have alternate layers of different materials with distinct borders) may vary smoothly by the coating’s thickness. Making of gradient or thickness modulated coatings is a very actual problem, as it allows to improve essentially the performance properties of coatings (wear and corrosion resistance), and eliminate the negative effect of “thin ice”, etc. [13]. On the base of the electrolytic chromium-indium-molybdenum-vanadium alloy, a composite electrochemical gradient coating was made due to introducing cubic boron nitride into its composition, in order to improve further micro-hardness and wear resistance. As the experimental results have shown, such a coating is significantly superior in wear resistance to the coatings obtained from the standard sulphate electrolyte of chroming.

All coatings obtained were examined for composition, micro-hardness, and wear resistance. The composition of the obtained electrolytic alloys was determined by the method of micro X-ray spectral analysis (MRSA) using an X-ray microanalyzer MR-7. Micro-hardness was defined on steel samples covered with studied alloys. Initial samples had shape of plates sized 20x10x1.5 mm. Microhardness
was determined on steel samples coated with the studied alloys. The initial samples had the form of plates with dimensions of 20x10x1.5 mm. The microhardness of the coatings was determined on a microhardometer by the Vickers method, by measuring the print diagonals obtained from indentation of the diamond pyramid. Wear resistance of the coating was studied with a rotary-type friction machine and reciprocating stand of construction. The reciprocating unit allows wearing out two samples simultaneously. As an opposite body, the plate made of high-carbon steel U10A (Russian mark, English analog BW1B, Chinese analog T10A), was used. The steel was hardened and tempered at low temperature. In a rotating-type friction machine, plastic strips made of glass-filled phenol-formaldehyde polymer AG-4C (Russian mark) were used as a counter body. In preliminary experiments, plastic was stuck on copper or steel pads. It was found that it was impossible to provide conditions for uniform wear process of coatings on a roller, by using a rigidly fixed opposite body. As the coatings were wearing out locally, wear process of the support plate material introduced an error in measurements of the coatings’ wear amount. In order to achieve uniform wearing of the coatings, the plastic tape was laid in two layers on 6 mm thick rubber gasket and held in tight state on a cylindrical drum with a clip of the holder with which the rotary-type friction machine was equipped. Run-in was first performed with force 10 N, then the loading gradually increased to 100 N. Tests were carried out at frequency of the roller’s rotation 300 rpm (sliding velocity 0.785 m/s). The coatings’ wear was evaluated by measuring their mass which was defined with an analytical balance, accuracy to 0.1 mg. To evaluate the wear scar, a microscope MPB-2 (Russian mark) was used.

3. Results and discussion

At first, the content of indium in chromium alloys obtained by electrodeposition, and their appearance depending on the concentration of indium compounds in initial electrolytes were examined. For this electrolytes for electrodepositing the alloys were prepared, their composition being presented in table 1. Electrolytic chromium-indium-molybdenum-vanadium (Cr-In-Mo-V-BN) alloy is a gradient composite coating. Cubic boron nitride was selected as the second phase to introduce into the coating, since it is inferior only to diamond in hardness and contributes to wear resistance increase [14]. Herewith, cubic boron nitride manifests hydrophilic properties in the electrolytes used, so it can be quickly and well wetted by the electrolyte. Indium was introduced in the electrolyte as indium sulphate, or in the acidified electrolyte as indium oxide. Concentration of indium-containing compounds varied in electrolytes N1-3. The content of indium in the electrolyte was calculated on the base of concentrations of indium-containing compounds in the electrolyte. In order to gradually increase the content of indium in the coating, cathode current density in electrolyte N3 has been decreasing smoothly from 70A/dm² to 20A/dm².

| Components and regimes | Electrolyte N 1 | Electrolyte N 2 | Electrolyte N 3 |
|------------------------|----------------|----------------|----------------|
| Chromic anhydride, g/l | 80             | 250            | 80-150         |
| Sulfuric acid, g/l     | 0,8            | 2,5            | 0,8-1,5        |
| Indium sulphate, g/l   | -              | -              | 0,5-37,5       |
| Indium oxide, g/l      | 2-50           | 0,5-20         | -              |
| Molybdic acid, g/l     | -              | 15             | 10-30          |
| Vanadic acid, g/l      | -              | -              | 10-30          |
| Cubic boron nitride, g/l| -            | -              | 10-50          |
| Cathodic current density A/dm² | 25           | 25            | 70-20          |
| Electrolyte temperature, C | 50           | 50-65         | 55-65          |
The concentration of indium in the electrolytic alloys based on chromium has been changed quite a lot, depending on the indium concentration in initial electrolytes. The experimental results are presented in figure 1.

As can be seen from figure 1, at the increase indium concentration in the electrolytes, its content in the chromium-based indium-containing coatings also increases. Herewith, at first, the indium content in the coatings increases more sharply at lower concentrations of indium in the electrolytes. Then, as the double electrolytic layer was being saturated with indium ions, the concentration increased slower. As it follows from figure 1, when the concentration of indium ions in the electrolytes becomes similar, more indium is deposited in the electrolytic alloy Cr-In-Mo. It is known that introduction of molybdic acid into composition of the standard electrolyte of chroming impacts essentially on kinetics of electrodepositing chromium. The higher was the concentration of molybdic acid, the lower was the current output observed. Decreased rate of depositing chromium with increased concentration of molybdate-ions has been conditioned by decreased content of ions of hexavalent chromium in a cathode film due to increased concentration of more hardly restored molybdenum ions in it. Also, molybdate-ions contribute to formation of a less hard, looser, easily hydrogen-penetrated film. This loosing action of molybdate-ions on a cathode film is likely to contribute to deposition of indium ions into the electrolytic alloy. Despite the fact that electrolytes for electrodeposition of the alloy Cr-In-Mo-V-BN contain molybdate-ions, in this case it is necessary to consider possible impact of other electrolyte components as well. Decreased indium content in Cr-In-Mo-V-BN coatings deposited from electrolyte N2 is associated with the impact of vanadic acid on cathodic process. As vanadic acid is being introduced into the standard chromium solution to studied concentration 30 g/l, a more compact, hard and less porous film is formed, compared with a film formed in presence of molybdenum ions. This is correlated with the impact of vanadic and molybdic acids on the current output. Thus, the current output was equal to 17–18% in the standard chromium solution at current cathode density 50 A/dm² and temperature 50°C. As vanadic acid of concentration 30g/l was being added into this electrolyte, the current output became equal to 22–23%, and, in similar conditions of electrolysis and with the same concentration of molybdenum acid, the current output decreases to 16–17% [15]. Presence of vanadic acid in electrolytes results in decreased indium content in coatings which is aggravated at increased concentration of sulphate ions, since indium is introduced into electrolyte N3 as indium sulphate. Besides, it is necessary to consider that Cr-In-Mo-V-BN electrodeposition was...
performed at gradual decrease of current cathodic density from 70 A/dm\(^2\) to 20 A/dm\(^2\) made to obtain a gradient coating. Electroplating of alloys Cr-In-Mo and Cr-In was made at constant current cathode density 25 A/dm\(^2\).

The experiments have proved that the appearance of a coating is greatly influenced by the indium content in alloys. Table 2 gives the dependence of metallic luster on the indium content in the coating.

**Table 2. Dependence of coatings’ appearance on indium content in electrolytic indium-containing alloys of chromium.**

| Indium content in coating, % | Cr-In | Cr-In-Mo | Cr-In-Mo-V-BN |
|-----------------------------|-------|----------|--------------|
| 1.4-1.5                     | gl/lu | -        | lu/sl        |
| 4                           | -     | -        | lu/sl        |
| 8.8-11.7                    | lu/sl | lu/sl    | sl/mt        |
| 15.2-19.9                   | sl/mt | sl/mt    | mt/mt        |
| 26.4–27.8                   | mt/mt | mt/mt    | -            |

As table 2 shows, chromium-indium coatings with low indium content (1.5% by weight) had the highest luster (lu) degree. Such, 15.8 µm thick, coatings electrodeposited as glassy (gl) ones. Then, as the coating became thicker, the degree of luster decreased a little, and 30.3 µm thick coatings became lustrous. The same effect, namely, decreased luster of the coatings with increased thickness has been observed for all studied indium-containing alloys of chromium. The electrolytic alloys Cr-In–Mo–V–BN at low indium concentration in the coating (1.4% and 4%) and the alloys Cr-In(9.6%) and Cr-In(8.8%)-Mo at the coating’s thickness 15.5±1 µm were lustrous. In 30.8±1.6 µm thick coatings, these alloys became semi-lustrous (sl). Coatings Cr-In (19.1%) and Cr-In(19.9 %)-Mo deposited from semi-lustrous to mat ones, depending on thickness of the coatings. At indium content from 26.4% to 27.8% in these coatings, in the whole studied range of thicknesses, mat coatings deposited. No doubt, indium content in chromium-based coatings is the most significant factor influencing the degree of luster of coatings. Besides, the luster degree of coatings decreases if there is vanadic acid and boron nitride in the electrolyte. Thus, if the coatings Cr-In(15.2%)-Mo-V-BN deposited as mat ones in the whole studied range of thicknesses, the coatings Cr-In (19.1%) and Cr-In(19.9%)-Mo with thicknesses less than 15.5 µm deposited as semi-lustrous ones.

Composition impact of the electrolytes and regimes of electrolysis (table 3) on composition of the gradient coatings Cr-In-Mo-V-BN and rate of their deposition (table 4) have been studied. Electrolyte with minimal concentrations of components (electrolyte N3 in table 3), electrolyte with average concentrations of components (electrolyte N4 in table 4), and electrolyte with maximum concentrations of components (electrolyte N5 in table 4) were used for this purpose.

**Table 3. Compositions of electrolytes and regimes of electrolysis for electrodeposition of gradient composite coatings Cr-In-Mo-V-BN.**

| Components and regimes | Electrolyte N4 | Electrolyte N5 | Electrolyte N6 |
|------------------------|----------------|----------------|----------------|
| Chromic anhydride, g/l | 80             | 100            | 150            |
| Sulfuric acid, g/l     | 0.8            | 1.0            | 1.5            |
| Indium sulphate, g/l   | 0.5            | 5.0            | 10             |
| Molybdc acid, g/l      | 10             | 20             | 30             |
| Vanadic acid, g/l      | 10             | 20             | 30             |
| Cubic boron nitride, g/l | 10           | 25             | 50             |
| Cathode current density, A/dm\(^2\) | 70-20        | 70-20         | 70-20          |
| Electrolyte temperature, °C | 60            | 60             | 60             |
Table 4. Dependence of compositions of galvanic alloys and rate of plating them upon composition of electrolytes.

| Alloy composition and electrodeposition rate | Electrolyte N4 | Electrolyte N6 |
|--------------------------------------------|----------------|----------------|
| Indium, %                                  | 1.4            | 11.7           |
| Molybdenum, %                              | 0.5            | 0.7            |
| Vanadium, %                                | 0.02           | 0.04           |
| Cubic boron nitride, %                     | 0.37           | 0.51           |
| Chromium                                   | rest           | rest           |
| Electrodeposition rate, µm/hour            | 32.4           | 29.2           |

Table 5. Impact of thickness of gradient coatings Cr-In-Mo-V-BN electrodeposited from electrolytes N4, N5 and N6 on microhardness and static friction coefficient (of resting state).

| Mechanical properties                        | Electrolyte N4 | Electrolyte N5 | Electrolyte N6 |
|----------------------------------------------|----------------|----------------|----------------|
| Friction coefficient, t₁/t₂                  | ≈0.20          | ≈0.17          | 0.16/0.14      |
| Micro-hardness, GPa, t₁/t₂                   | ≈10.9          | ≈10.4          | 10.1/8.94      |

In the table 5 there are results of measurement at thickness of the coating t₁=5.5±1µm, and in the denominator – at thickness t₂=30.8±1.6 µm.

Results of measuring micro-hardness of the coatings have shown that, despite the increased content of hard particles of cubic boron nitride in the coating from 0.37% to 0.51% and increased content of molybdenum and vanadium, the indium content is a major factor determining the amount of micro-hardness. The indium concentration increased from 1.4% to 11.7%, correspondingly, despite the other factors, the micro-hardness decreased from 10.9 GPa to 8.94 GPa. As table 6 shows, the increased indium content in chromium galvanic alloys decreases the coefficient of friction. The decreased friction coefficient and micro-hardness, along with the increased thickness of coatings Cr-In-Mo-V-BN, is related to the increased indium content in upper layers of the coating caused by smooth decrease of cathodic current density.

In this work, much consideration has been given to the studies of wear resistance of indium-containing chromium alloys. Results of the testing presented in table 6 have demonstrated perceptivity of using indium-containing chromium alloys in the friction conditions examined. The given data have shown that, at first, as the indium content increased in the alloys, the wear process decreased, despite the decreased micro-hardness. The greatest wear resistance paired with steel friction belonged to sample N2 coated with the Cr-Mo-In alloy, indium content 19.9%, and sample N5 coated with the Cr-In-Mo-V-BN alloy, indium concentration 11.7%, this is likely to be due to the lower friction coefficient.

Table 6. Dependence of wear of coatings paired with hardened steel friction upon their composition and conditions of obtaining.

| Composition of alloys | HV, GPa | Wear, µm/km | Conditions of obtaining                  |
|----------------------|---------|-------------|-----------------------------------------|
| 1 Cr                 | 10.3    | 3.9         | Electrolyte N1 without indium, Cathode current density 50 A/dm² |
| 2 Cr-Mo-In(19.9%)     | 8.57    | 0.41        | Electrolyte N2 with indium 8.3 g/l      |
| 3 Cr-In(1.4%)-Mo-V-BN | 10.9    | 0.82        | Electrolyte N4                          |
| 4 Cr-In-Mo-V-BN      | 10.4    | 0.49        | Electrolyte N5                          |
| 5 Cr-In(11.7%)-Mo-V-BN | 8.84   | 0.36        | Electrolyte N6                          |
| 6 Cr-Mo-V            | 10.6    | 1.03        | Electrolyte recommended [16]             |
One of main purposes of this work is to increase service of molds used to produce details from glass-filled plastics of brands AG-4S (Russian mark) due to increased wear resistance of chromium coatings. Concerning this, the wear resistance of developed coatings paired with plastic AG-4S (Russian mark) friction has been studied, using unit SMTs 2 (Russian mark) (see table 7).

**Table 7.** Impact of conditions of obtaining and composition of coatings on their wear paired with glass-filled plastic AG-4S (Russian mark) friction after 20000 rounds of roller.

| Composition of alloys | HV, GPa | Wear, g/m·10^-6 | Conditions of obtaining |
|-----------------------|---------|------------------|-------------------------|
| 1 Cr                  | 10.3    | 2.8              | Electrolyte N1 without indium |
| 2 Cr-Mo-V             | 10.6    | 0.6              | Electrolyte recommended [16] |
| 3 Cr-Mo-In(8.8%)      | 9.92    | 0.33             | Electrolyte N2 with indium 1.65 g/l |
| 4 Cr-Mo-In(19.9%)     | 8.57    | 0                | Electrolyte N2 with indium 8.3 g/l |
| 5 Cr-Mo-In(26.4%)     | 6.62    | 0.76             | Electrolyte N2 with indium 16.5 g/l |
| 6 Cr-In(9.6%)         | 9.35    | 1.64             | Electrolyte N1 with indium 8.3 g/l |
| 7 Cr-Mo-In(11.7%)-V-BN| 8.94    | 0                | Electrolyte N6 |

Despite decreased micro-hardness of the coatings, alloying with indium can lead to increased wear resistance of the coatings, compared with pure chromium coatings. This is most likely to be related to decreased friction coefficient of such coatings paired with glass-filled plastic AG-4S (Russian mark) friction. Besides, degree of hydrogen-charging coatings (which can be less for indium-containing coatings), graininess of a coating, whether there are structure defects, and other factors should be considered. Concerning the wear resistance of galvanic Cr-Mo-In alloys, you can see in table 8 that the alloys containing 19.9% of indium possess the maximum wear resistance. The roller coated with such alloy does not demonstrate the sign of losing the coating’s mass even after 20000 rounds. At further increase of indium content in the Cr-Mo-In alloys to 26.4%, their wear resistance decreases due to decreased micro-hardness of the coatings (composition N5, table 7). Good results have been obtained by using a gradient composite coating Cr-Mo-In(11.7%)-V-BN, it having a zero wear after 20000 rounds of the roller with such coating.

**4. Conclusion**

As a result of the work done, technology of electrodepositing indium-containing chromium-based alloys with maximum indium content 27.8% has been suggested. It has been found that molybdate-ion contributes to indium electrodepositing in a chromium-based alloy, and vanadate-ion, on the contrary, hinders. It has been defined that, as indium content in the alloys and thickness of the coatings increase, luster degree and micro-hardness of the coatings decrease. However, after polishing, the alloys with higher indium concentration have lower friction coefficient. We succeeded in making a gradient chromium-based coating. In these coatings, micro-hardness of lower layers neighboring directly the metal-base (steel) has high hardness which decreases gradually as thickness of the coating increases. And upper layers possess low friction coefficient, thank to which they can work for a long time without lubricating. Galvanic alloy Cr-Mo-In (11.7%)-V-BN paired with glass-filled plastic friction as well as with steel friction has the greatest wear resistance. A gradient composite coating Cr-Mo-In (11.7%)-V-BN may be recommended to increase resistance of molds and other details working in the conditions of dry friction, especially of those contacting with glass-filled plastics.

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