ISSN 2070-2051, Protection of Metals and Physical Chemistry of Surfaces, 2020, Vol. 56, No. 3, pp. 560–566. © Pleiades Publishing, Ltd., 2020.
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NEW SUBSTANCES, MATERIALS, AND COATINGS

Chemical Polishing of Aluminum Using Acid-Containing Reverse Microemulsions

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Received January 23, 2018; revised January 25, 2018; accepted February 1, 2018

Abstract—Chemical polishing of metals using nanostructured media—reverse microemulsions has been studied using the example of aluminum. It has been shown that the surface roughness of aluminum foil reduced after the treatment with reverse microemulsion of sodium bis-(2-ethylhexyi)phosphate in kerosene containing an aqueous solution of hydrochloric acid with a concentration of 0.11 mol/L inside the drops. At a polishing time of 2 h, the average roughness was reduced from 54 to 28 nm. Compositions that do not form microemulsions (aqueous solution of hydrochloric acid, solution of tributylphosphate adduct with hydrochloric acid in kerosene, or dispersion of sodium bis-(2-ethylhexyl)phosphate in an aqueous solution of hydrochloric acid), have not exhibited such an effect. Additional exposure to ultrasound at the same process duration, the introduction of tributylphosphate into the microemulsion, and the replacement of hydrochloric acid with acetic or nitric acid had virtually no effect on the results of chemical polishing.

Keywords: chemical polishing, microemulsion, sodium bis-(2-ethylhexyl)phosphate, surface treatment, aluminum, nanostructured media

DOI: 10.1134/S2070205120030259

INTRODUCTION

Nanostructured liquid media, such as microemulsions, lyotropic liquid crystals, and direct and reverse micelles, are promising systems for various branches of science and technology. Microemulsions are thermodynamically stable isotropic dispersions of nonpolar organic and aqueous phases stabilized by a surface-active substance (substances). The diameter of microemulsion drops ranges from a few to dozens of nanometers. Microemulsions are used for cleaning surfaces of solids from impurities, for improving oil recovery of wells, in construction as hydrophobizers, and in separating substances in analytical chemistry. Microemulsions can be used as media for enzymatic reactions, in polymerization producing nanoscale latex particles, in synthesis of inorganic nanoparticles, for liquid and membrane extraction of organic and inorganic substances, and as carriers for targeted drug delivery [1].

Microemulsions as nanostructured reagent carriers can be used for the development and improvement of various processes occurring in the "liquid–solid phase" systems. Earlier, a method was suggested for extraction of metals from solid phase particles using microemulsions (microemulsion leaching). The method of microemulsion leaching involved the extraction of metals from industrial raw materials (concentrates, sludges, ash, dust, etc.) by a contact with an extractant-containing microemulsion, which enabled one to combine leaching and extraction in one process. For leaching, it was suggested to use microemulsions based on sodium bis-(2-ethylhexyl)phosphate (NaDEHP) containing the known extractants bis-(2-ethylhexyl)phosphoric acid and tributylphosphate [2–4].

Microemulsion, as a carrier of a metal-dissolving reagent, can be used to reduce surface roughness of metals (chemical polishing). A mixture of concentrated acids with a small amount of water is often used for chemical polishing. Here, the result of the polishing was improved with the presence of a viscous diffusion layer adjacent to the metal surface, which contributed to the dissolution of protrusions on the surface and made it difficult for a reagent to access hollows [5]. If the acid was localized inside the microemulsion drops, it was possible to reduce its total concentration in the polishing liquid (microemulsion), as well as to achieve a better polishing effect due to diffusion restrictions. For chemical polishing, it is of interest to use a microemulsion of the "water-in-oil" type (reverse microemulsion), in which the dispersion medium is a nonpolar organic solvent that poorly conducts electricity. It has been suggested that this will avoid the occurrence of local currents that affect the chemical polishing process [5, 6]. Note that there are very few published data on using microemulsions for etching or chemical polishing.

The possibility of using reverse microemulsions for copper etching was shown. Microemulsions in the systems sodium bis-(2-ethylhexyl)sulfosuccinate (AOT)–isooctane–water and Pluronic L62–xylene–water were used: their drops contained an aqueous solution of a mixture of copper(II) chloride and potassium chloride at a molar ratio of 1 : 1. The average roughness of the foil after etching with microemulsions based on Pluronic L62 (containing 0.05 M KCl + 0.05 M $CuCl_2$) and AOT (containing 1.0 M KCl + 1.0 M $CuCl_2$) was 44 and 70 nm, respectively. After etching with aqueous solutions of KCl and $CuCl_2$ with the same concentration as in the aqueous phase of microemulsions, the average roughness values were higher: 84 and 88 nm, respectively. The authors attributed this effect to the fact that microemulsion drops, unlike salts dissolved in water, interacted better with protruding surface defects rather than with microcracks. A drop of reverse microemulsion has a large size compared to salt ions in an aqueous solution. This made it difficult for the reagent from the microemulsion to access the microcracks on the surface [7].

The use of reverse microemulsion containing an aqueous solution of KH_2PO_4 in drops with a concentration from 0.01 to 0.1 mol/L for the chemical-mechanical polishing of KH_2PO_4 crystals was shown. The average surface roughness of the KH_2PO_4 crystal decreased from 4.6 to 1.5 nm after the treatment by microemulsion in the Triton X-100–decanol–0.03 M aqueous solution of KH_2PO_4 system [8].

We demonstrated that it was possible to reduce the surface roughness of nickel by using a reverse microemulsion based on NaDEHP containing an aqueous solution of hydrochloric acid inside the drops. The concentration of HCl, expressed in terms of the entire volume of microemulsion, was 0.026 mol/L, the process was carried out for 2 h. Under the same conditions, treatment of the nickel surface with an aqueous HCl solution of a concentration of 0.026 mol/L resulted in an increase of roughness [9]. This can be explained by the fact that the diffusion coefficients of a substance in a molecular solution were by two orders of magnitude higher than for a substance localized in microemulsion drops. Access of the microemulsion drops to the hollows on the metal surface was obstructed due to the diffusion restrictions. A reagent localized in the droplets was expected to interact better with the protruding areas of the surface, which was hypothesized would lead to the predominant dissolution of the protrusions and reduce the average roughness.

The objective of the present work was to study the chemical polishing of metal by means of nanostructured media (reverse microemulsions) on the example of polishing of aluminum. The earlier well-studied [3, 10, 11] microemulsion based on sodium bis-(2-ethylhexyl)phosphate containing hydrochloric, nitric, or acetic acids was selected as a polishing medium.

EXPERIMENTAL

Bis-(2-ethylhexyl)phosphoric acid by Acros Organics brand (the main substance content was not less than 95 wt %) and sodium hydroxide of pure grade were used to fabricate the microemulsion. Kerosene of Osvetlitelniy (Lightening) KO-25 grade (Expert-Oil, Russia) was used as an organic solvent. Hydrochloric, nitric, and acetic acids of pure grade were added to the microemulsion. In some cases, tributylphosphate by Acros Organics (the content of the main substance was not less than 99 wt %) was added to the microemulsion. Distilled water was prepared by the standard method.

NaDEHP microemulsion was produced according to the method described in [2, 3]. An aqueous phase of the microemulsion was represented by a solution of sodium hydroxide in distilled water. An organic phase was represented by a solution of bis-(2-ethylhexyl)phosphoric acid, and, if necessary, tributyl phosphate in kerosene. The required amount of water and organic phase was mixed in order to prepare the microemulsion. During the mixing process, a neutralization reaction occurred between sodium hydroxide and bis-(2-ethylhexyl)phosphoric acid with the formation of a surface active substance—sodium bis-(2-ethylhexyl)phosphate—which stabilized the microemulsion drops. Here, we observed a transformation of the turbid emulsion into a transparent homogeneous microemulsion. The necessary amount of concentrated hydrochloric or nitric or acetic acid was then added to the resulting microemulsion.

The hydrodynamic diameter of microemulsion droplets was determined by the method of dynamic light scattering using a Zetasizer Nano ZS particle size analyzer (Malvern, United Kingdom). To remove dust, the sample was prepared for measurements by centrifugation on an OPN-8 centrifuge (Russia) at 8000 rpm (acceleration of 150 g) for 30 min. To obtain statistically reliable results, each measurement was performed at least five times.

The chemical polishing of aluminum foil (foil for packaging, GOST (State Standard) 745-2003) was carried out according to the following method. Thirty milliliters of microemulsion was placed in a 50-mL weighing bottle. A 20 × 20-mm metal plate was then immersed in the microemulsion so that the entire test surface was covered in the liquid. The metal was dissolved in a closed container at a temperature of 80°C with constant mechanical stirring (300 rpm) for 1–3 h. Ultrasound was used in addition to mechanical mixing in some experiments. Ultrasound with a power of 10 W was generated by a UZG 13-0.1/22 ultrasonic disperser. During the polishing process, the microemulsion remained stable, with no turbidity and exfoliation being observed. After the polishing, the metal surface was cleaned of adsorbed surface-active substances by sequential washing of the plate in hexane, ethanol, and water. The polishing results were compared to a control sample—an aluminum foil plate washed sequentially in hexane, ethanol, and water.

The surface of the samples was analyzed by atomic force microscopy (AFM) using an Ntegra Prima atomic force microscope (NT-MDT, Russia, Zelenograd). A flint cantilever with a gold coating NSG10 (NT-MDT, Russia) was used. The semi-contact operating mode was used. The average roughness (S_a) was calculated for an area of 10 × 10 μm according to the ISO 4287/1 standard using the software of the device.

RESULTS AND DISCUSSION

Earlier, a decrease in the nickel surface roughness was shown after interaction with reverse microemulsion in the NaDEHP–kerosene–HCl water solution system [8]. Therefore, sodium bis-(2-ethylhexyl)phosphate in kerosene microemulsion was chosen for chemical polishing of aluminum in the same composition as used earlier: C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, and C_{HCl} = 0.026 mol/L. This corresponded to a content of the water phase in the microemulsion of 22.5 vol % and a concentration of hydrochloric acid in the water phase of 0.11 mol/L. The hydrodynamic diameter of the drops of such microemulsion determined by the method of dynamic light scattering was 13.3 ± 0.6 nm (Fig. 1). Note that, during the process of chemical polishing of nickel, a drop structure of the reverse microemulsion was characteristic, while only a slight change in the hydrodynamic diameter of the drops was observed: 13.3 ± 0.6 nm before polishing and 8.4 ± 0.7 nm after polishing for 2 h. The diameter of microemulsion drops of the same composition, which did not contain hydrochloric acid, was 3.0 ± 0.1 nm. This change in the diameter of the microemulsion drops during the chemical polishing was explained by a decrease in the concentration of hydrochloric acid, which was expended on dissolving the metal.

Since the surface of aluminum was coated with an oxide film, the following chemical reactions can occur during the polishing of aluminum:

$$2Al + 6HCl \rightarrow 2AlCl_3 + 3H_2, \quad (1)$$

$$Al_2O_3 + 6HCl \rightarrow 2AlCl_3 + 3H_2O. \quad (2)$$

The effect of the process time on the reduction of roughness was investigated. The results of the AFM study of the surface of the aluminum foil samples before polishing (control) and after 1, 2, and 3 h of the process were shown in Fig. 2. The values of average roughness depending on the time of chemical polishing with microemulsion were shown in Table 1. As can be seen from the presented data, the average surface roughness of the aluminum foil decreased during the first 2 h of polishing, and then increased slightly. Therefore, for further experiments, the time of the polishing process was chosen equal to 2 h.

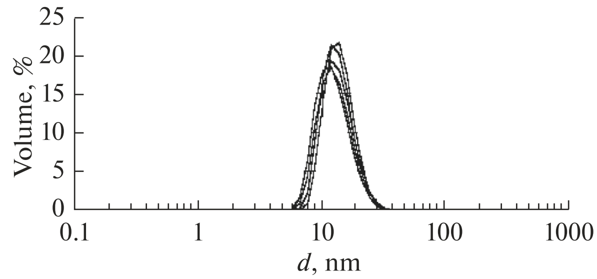

Fig. 1. Hydrodynamic diameter of droplets of the microemulsion used for chemical polishing. Composition of the microemulsion: C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, C_{HCl} = 0.026 mol/L.

In the case of chemical polishing with mixtures of concentrated acids, the process takes minutes or even fractions of minutes. Such a short polishing time is by caused a number of technological difficulties associated with the necessity for rapid washing of items from a polishing solution [5]. When using microemulsions, the duration of chemical polishing lasts for hours. On the one hand, such a slow process is easier to control. On the other hand, 2 h is a very long time in terms of creating an effective technology. Therefore, it was necessary to develop measures to accelerate the chemical polishing using microemulsions.

The process of interaction of copper oxide with an extractant-containing NaDEHP microemulsion (microemulsion leaching) occurred in a mixed regime; i.e., it was controlled by both diffusion and chemical reaction [12]. It can be suggested that the rate of the chemical polishing process by means of microemulsions must be controlled by diffusion and chemical reaction. In this case, one can increase the rate in the following ways:

— by increasing the temperature,

— by additional activation by means of ultrasound,

— by increasing the concentration of a metal-dissolving reagent (acid), and

— by facilitating the diffusion of acid from a drop of the aqueous phase to the metal surface through a layer of the organic phase.

In the present work, the chemical polishing process was underway as early as at 80°C. Further increase in temperature was not desirable due to the possible intensive evaporation of the solvent (kerosene) and the limited region of existence of microemulsions. A significant increase in the concentration of acid in the

Table 1. Average surface roughness of samples before and after chemical polishing with microemulsion containing HCl, according to AFM data

Time of process, h	0 (control)	1	2	3
Average roughness, nm	54	35	28	32

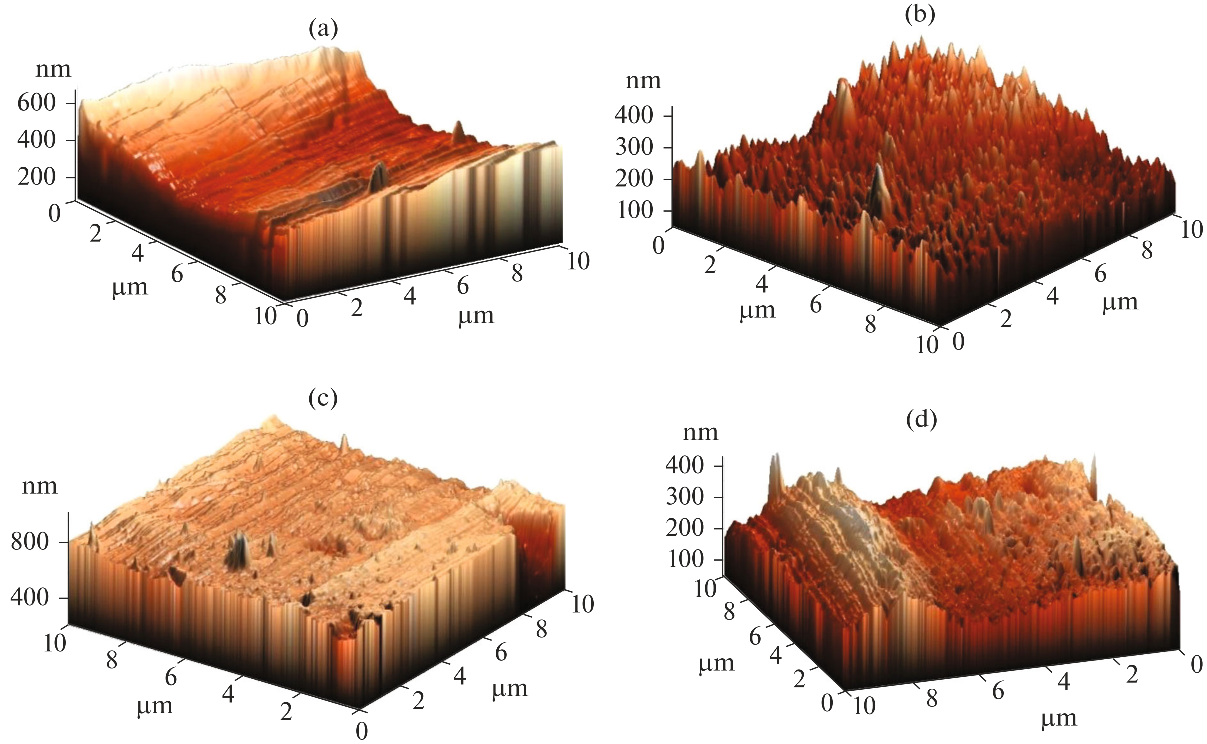

Fig. 2. Aluminum surface relief before and after chemical polishing with a microemulsion containing hydrochloric acid: (a) control, (b) 1, (c) 2, and (d) 3 h. Composition of the microemulsion: $C_{NaDEHP} = 1.22$ mol/L, $C_{water} = 13.2$ mol/L, $C_{HCl} = 0.026$ mol/L.

microemulsion was difficult to achieve due to low solubilization capacity of the studied microemulsion towards acids. Therefore, we studied the possibility of conducting the polishing process under effect of ultrasound and conditions of facilitating the diffusion of acid through the organic phase layer by adding tributyl phosphate.

The effect of ultrasonic mixing of the polishing medium (microemulsion) on the results of chemical polishing was investigated. The composition of the microemulsion was the same as in the earlier experiment: $C_{NaDEHP} = 1.22$ mol/L, $C_{water} = 13.2$ mol/L, and $C_{HCl} = 0.026$ mol/L. The time of the process was 2 h. Table 2 shows the values of the average surface roughness of the samples after chemical polishing with no mixing of the microemulsion, with mechanical mixing, and under the action of mechanical mixing and ultrasound.

Table 2. Average surface roughness of samples after chemical polishing with microemulsion containing HCl under various mixing modes, according to AFM data

Mode for mixing of the microemulsion	Average roughness, nm
Without mixing	49
Mechanical mixing	28
Mechanical mixing and ultrasound exposure	25
Control (before polishing)	54

As can be seen from the data presented in Table 2, in the absence of mixing, there was a significantly lower reduction in roughness than in the case of mechanical mixing. The combination of ultrasonic and mechanical mixing had a little effect on the result of the chemical polishing. When subjected to ultrasonic exposure during polishing, the microemulsion remained stable, no turbidity or destruction into separate phases was observed. This enabled one to combine mechanical and ultrasonic stirring during the polishing by means of the microemulsion. More detailed further studies on kinetics of the process is required in order to better understand the role of the ultrasonic exposure in the chemical polishing of metal by means of microemulsions.

Another approach to accelerate chemical polishing by means of microemulsions implied facilitating the diffusion of a reagent from a drop to the metal surface through an organic phase layer. Tributylphosphate (TBP) is a well-known industrial extractant that extracts inorganic acids from aqueous solutions into the organic phase by forming adducts [13]. Therefore, tributylphosphate can facilitate the transfer of HCl molecules from the water drop through the organic phase layer to the metal surface. In addition, the introduction of TBP in the microemulsion made it possible

Table 3. Solubilization of acids in microemulsion. Composition of microemulsion: C_{NaDEHP} = 1.22 mol/L, C_{TBP} = 0.15 mol/L, C_{water} = 13.2 mol/L. T = 20°C

Acid	Nitric	Hydrochloric	Sulfuric	Acetic
Maximum concentration of acid in microemulsion, mol/L	0.073	0.091	0.035	0.310

to increase the amount of acid that can be solubilized in the microemulsion. This will allow the use of microemulsions with a higher concentration of acid for chemical polishing. For example, with the same water content, the maximum concentration of acetic acid in NaDEHP microemulsion that did not contain TBP was 0.25 mol/L; in the presence of 0.45 mol/L TBP, the maximum concentration of CH_3COOH increased up to 0.51 mol/L. Table 3 shows the data on acid solubilization in NaDEHP microemulsion containing 0.15 mol/L TBP.

The effect of introducing TBP to the microemulsion on the process of polishing the aluminum surface was investigated. A microemulsion of the following composition was used: C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, C_{HCl} = 0.026 mol/L, and C_{TBP} = 0.15 mol/L. The polishing time was 1 and 2 h. The values of the average surface roughness of the samples were shown in Table 4.

As can be seen from the data presented in Tables 1 and 4, the introduction of TBP did not virtually affect the chemical polishing of the aluminum foil with NaDEHP microemulsion containing hydrochloric acid. In further experiments, we used a microemulsion containing TBP and various acids.

Table 4. Average surface roughness of samples before and after chemical polishing with microemulsion containing TBP and HCl, according to AFM data

Time of process, h	0 (control)	1	2
Average roughness, nm	54	47	29

The effect of the acid nature on the chemical polishing of the aluminum surface with a microemulsion was investigated. The concentration of acids in the microemulsion equaled to 0.026 mol/L, which was significantly lower than their maximum content (Table 3); the concentration of other components: C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, and C_{TBP} = 0.15 mol/L. The process time was 2 h. The results of the AFM study of the samples surface were shown in Fig. 3, and the average roughness values were shown in Table 5.

From the presented data (Fig. 3; Table 5), it can be seen that all the studied microemulsions reduced the roughness of the samples at the approximately equal

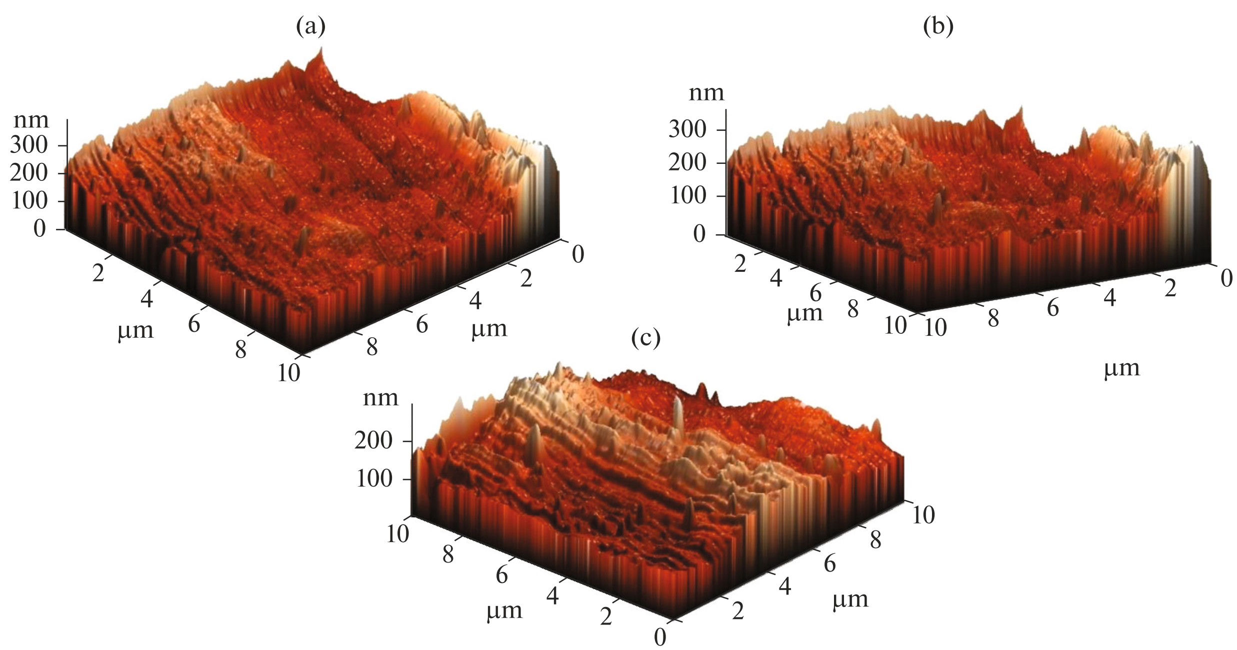

Fig. 3. The relief of the aluminum surface after chemical polishing with a microemulsion containing TBP and acid: (a) hydrochloric acid, (b) acetic acid, and (c) nitric acid. Composition of the microemulsion: C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, C_{TBP} = 0.15 mol/L. Concentration of acids in the microemulsion was 0.026 mol/L. Polishing time was 2 h.

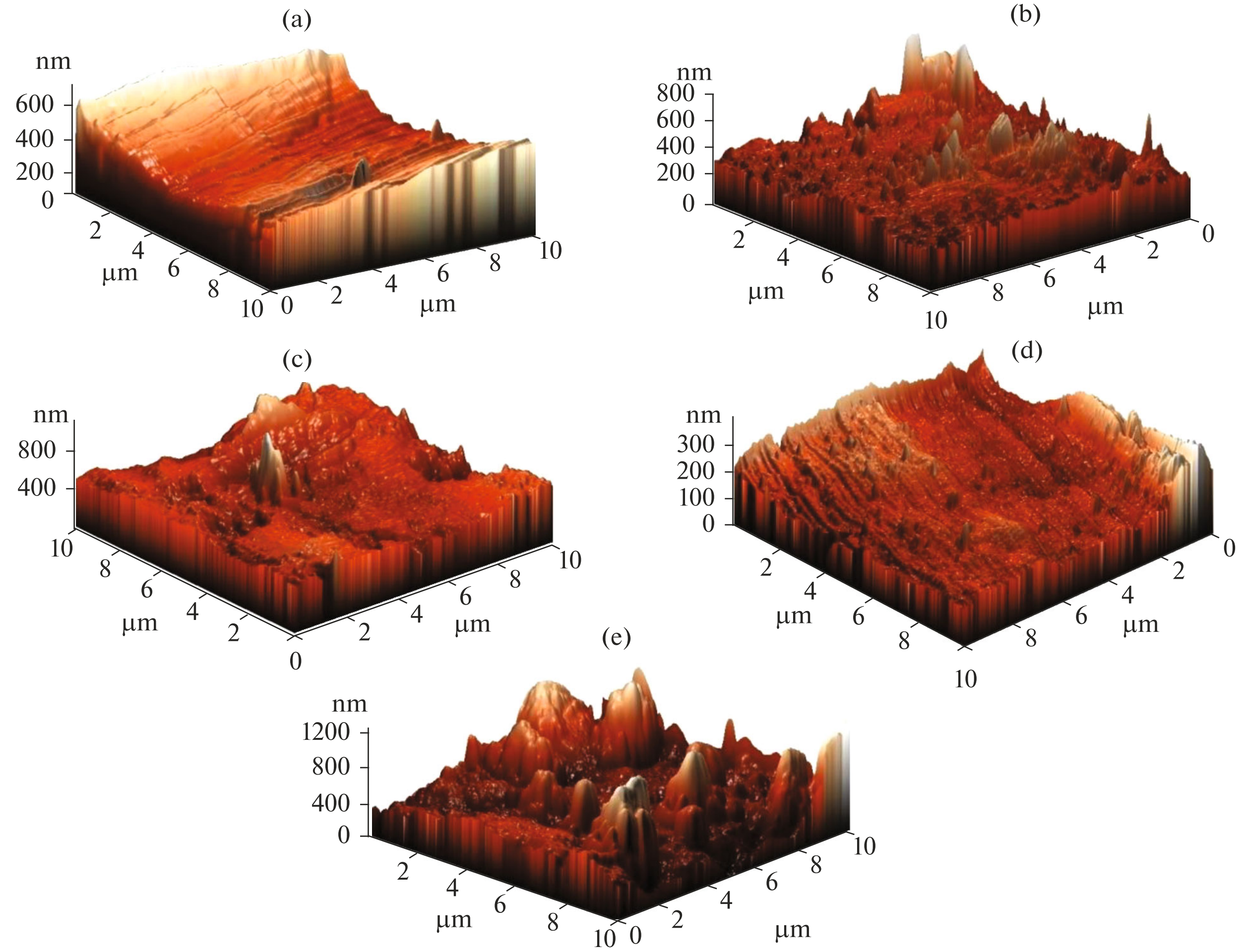

Fig. 4. The surface relief of aluminum after treatment with reagent mixtures: (a) control (before treatment), (b) water dispersion of NaDEHP containing HCl (C_{NaDEHP} = 0.0013 mol/L, C_{HCl} = 0.026 mol/L), (c) TBP solution in kerosene containing HCl (C_{TBP} = 0.15 mol/L, C_{HCl} = 0.026 mol/L), (d) microemulsion (C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, C_{HCl} = 0.026 mol/L, C_{TBP} = 0.15 mol/L), (e) water solution of HCl (C_{HCl} = 0.026 mol/L). Treatment time was 2 h.

degree. Therefore, all three acids can be used as parts of microemulsions for the chemical polishing of metals. Here, the highest content in the microemulsion (the highest solubilization capacity) was observed for acetic acid (Table 3). Increasing the concentration of acid in the microemulsion allowed to increase the rate of the chemical polishing process. Acetic acid was probably the most promising agent for further development of processes for chemical polishing of metals by means of reverse microemulsions.

To demonstrate that it was the nanostructured medium (microemulsion) that provided the conditions for chemical polishing, an experiment was conducted on polishing the aluminum surface with the following mixtures of reagents.

1. Aqueous dispersion of NaDEHP containing hydrochloric acid. Composition of the dispersion: C_{NaDEHP} = 0.0013 mol/L; C_{HCl} = 0.026 mol/L.

2. Solution of TBP in kerosene containing hydrochloric acid. Composition of the solution: C_{TBP} = 0.15 mol/L; C_{HCl} = 0.026 mol/L.

3. Aqueous solution of hydrochloric acid C_{HCl} = 0.026 mol/L.

The results were compared with a sample not subjected to the polishing (control) and a sample treated with a microemulsion containing TBP and hydrochloric acid. Composition of the microemulsion: C_{NaDEHP} = 1.22 mol/L, C_{water} = 13.2 mol/L, C_{HCl} = 0.026 mol/L, and C_{TBP} = 0.15 mol/L. The relief of the aluminum surface and the values of the average roughness of the samples are shown in Fig. 4 and Table 6.

Table 5. Average surface roughness of samples before and after chemical polishing with microemulsion containing TBP and varied acids, according to AFM data

Acid	Hydrochloric	Acetic	Nitric
Average roughness, nm	29	29	30

Table 6. Average surface roughness of the samples after treatment with reagent mixtures, according to AFM data

Composition of the reagent mixture	Average roughness, nm
Control (before treatment)	54
NaDEHP microemulsion containing HCl and TBP	29
Water dispersion of NaDEHP containing HCl	56
Solution of TBP in kerosene containing HCl	74
Aqueous solution of HCl	60

As can be seen from the data presented in Fig. 4 and Table 6, none of the used combinations of components that did not form a microemulsion caused no significant reduction in the roughness of the metal surface. Treatment of the aluminum surface with water dispersion of NaDEHP containing HCl caused a slight change in the average roughness compared to the control sample, and there were noticeable irregularities in the form of peaks on the surface (Fig. 4b). When treating metal with a solution of TBP in kerosene containing HCl and an aqueous solution of HCl, an increase in the roughness (etching) of the aluminum surface was observed (Fig. 4c, 4e). Only the impact of the nanostructured medium (microemulsion) led to smoothing of the aluminum surface (Fig. 4d).

To sum up, the possibility of chemical polishing of metal using a reverse microemulsion containing acid was demonstrated. The mechanism of polishing probably consisted in limiting the diffusion of acid to hollows on the metal surface and the predominant dissolution of protrusions due to the localization of the reagent in drops with a size of about 10 nm.

CONCLUSIONS

The chemical polishing of metals by means of nanostructured media—reverse microemulsions—has been studied on the example of aluminum. The possibility of reducing the surface roughness of aluminum foil treated with the NaDEHP microemulsion containing hydrochloric acid has been demonstrated. With the polishing time of 2 h, the average roughness has been reduced from 54 to 28 nm. Additional exposure to ultrasound during the process has appeared to slightly affect the result of polishing. The introduction of TBP to the microemulsion has had almost no effect on the chemical polishing of the aluminum foil with the microemulsion containing acid. Replacing hydrochloric acid in the microemulsion with acetic or nitric acid has not affected the result of the chemical polishing.

It has been demonstrated that the use of a nanostructured medium (microemulsion) for chemical polishing has been enabled the surface roughness of aluminum foil to be reduced significantly. Compositions not forming microemulsions (aqueous solution of hydrochloric acid, solution of tributylphosphate adduct with hydrochloric acid in kerosene, and dispersion of sodium bis-(2-ethylhexyl)phosphate in an aqueous solution of hydrochloric acid) have not exhibited such an effect.

The results obtained may be a basis for further development of new methods of chemical polishing of metals.

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Translated by D. Marinin