

Application of ultrasound for the systems of electroless copper plating: Influence of ultrasonic irradiation on the plating rate and the surface of coatings

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Ultrasound produces specific conditions for reactions in electroless copper plating systems. Electroless deposition of copper using the systems with different ligands (EDTA, saccharose, pentaethylenhexamine) and different reducing agents (formaldehyde, Co(III)/Co(II) redox couple) proceeds differently under ultrasonic irradiation. Depending on the plating system, the plating rate varies from modest decrease (or stopping of the process) to five or more times higher rate, compared with conditions without ultrasound. The dependencies of the surface roughness of the copper coatings obtained on various factors (ultrasonic irradiation, pH, ligands etc.) were determined and discussed.

Key words: electroless copper deposition, ultrasound, saccharose, EDTA, Co(II)/Co(III)

INTRODUCTION

Sound is compression and expansion waves passing through the gaseous, liquid and solid phases. Sound with oscillation frequency which exceeds the limits of human sensory system (more than 16–20 kHz) is called ultrasound (US). Ultrasound wavelengths 10^{-2} – 10^{-5} m are not close to the normal molecular dimensions, therefore the ultrasound cannot directly affect the molecules and chemical reactions. However, ultrasound provides a large amount of energy for chemical reactions taking place in the liquid phase. This is the result of an ultrasound induced physical phenomenon known as cavitation. Cavitation phenomenon is a dynamic formation, growth and implosion of cavities (bubbles), accompanied by high temperatures and high pressures. Collapse of cavities in the liquid and liquid-solid phase boundary is different. Ultrasonic waves, moving microvolumes of fluid via groups of molecules, and the compression-expansion cycles can form cavities when the negative pressure exceeds the local grav-

ity forces in the liquid. Cavities are usually formed in areas of liquid where dissolved gases are found or formation of gases occurs. Microcavities initiated by ultrasound absorb energy and grow. After reaching a critical size a bubble implodes – bursts inward (at 20 kHz frequency ultrasound the critical size of bubble is about 170 μm) (Fig. 1) [1, 2].

Implosion of bubbles produces high temperatures and high pressures in microvolumes that cause formation of specific conditions for chemical reactions. Cavity implosion releases large amounts of energy, leading to formation of short-term (10^{-6} s) localized micro-hot points where the temperature can reach 5000 °C. The difference of pressure at those points can reach 1000 atmospheres, and cooling-heating rates up to 10^9 °/s. Cavity collapse can be accompanied by a small flash of light, so it is possible to take emission spectrum analysis [1, 2].

Cavitation phenomenon is significantly different at the boundary of liquid-solid surface. Fluid flow is formed and directed into the solid phase during implosion near the surface. The flow speed can reach 400 km/h. Surfaces exposed to an enormous force and surface impacts lead to a formation of active reaction points after bubble implosion. Such fluid

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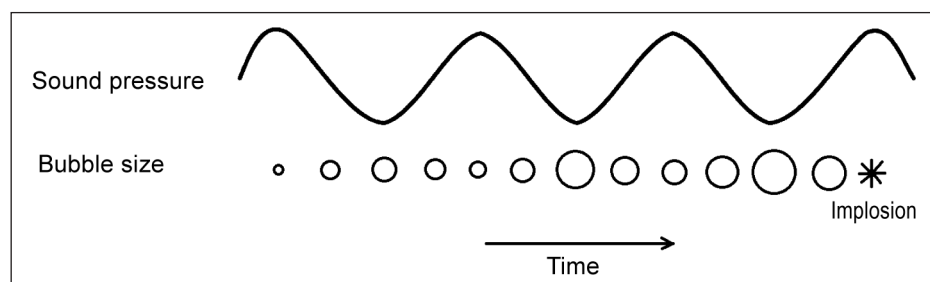


Fig. 1. Cavitation effects initiated by ultrasound

flows cannot be formed in the powdery substance (fluid current can be formed when the solid phase surface is several times larger than the critical size of the cavity). During homogeneous cavitation in the liquid containing solid particles, particles strongly confronted with each other and released enough energy for welding of metal particles [1, 2].

Organic compounds and water molecules exposed to ultrasound can be divided into radicals resulting in formation of new forms, also unusual molecular clusters can be created, homogeneous catalysis can be initiated, which can increase the reaction rate by hundreds of thousands times [1, 2].

There is a limited number of papers which describe the use of ultrasound in various cases of electrochemical and electroless deposition of metals. The main studies are generally restricted to specific measurements, typically observed in dependence on a single variable parameter, which is, e. g. the ultrasonic frequency, ultrasonic intensity and power, temperature or pH. According to the literature data, depending on the selected conditions, the use of ultrasound can increase plating rates (up to 50%) for metal deposition processes. Coatings deposited in US milieu, comparing with that deposited without ultrasonic radiation, usually are evenly distributed on the surface, have more compact crystal structure and sometimes have higher electrical conductivity, are smoother, mechanically and chemically more resistant; US can expand the coating adhesion (up to 3-fold), increase mass transfer rates (up to 38 times) [3–14]. However, it is difficult to predict experimental results and often it is complicated to explain the data obtained on the basis of known patterns and mechanisms. Since data about the effects of ultrasonic radiation on the parameters of electroless copper deposition are very limited and sometimes rather contradictory, the aim of the study presented herein was to clarify the general effects of ultrasound on the process of electroless copper deposition.

EXPERIMENTAL

The solutions were prepared from “Fluka” analytical grade reagents. For preparation of solutions three times distilled water was used. Experiments were carried on a polished platinum electrode which was covered with a layer of electrolytic copper from an acidic electrolyte (1 mol/l CuSO_4 and 0.5 mol/l H_2SO_4 , current density – 15 mA/cm², plating time – 20 min using soluble copper anodes). Electrolytically

plated copper electrode was rinsed with distilled water, dried, weighted and for 30 seconds activated in an acidic solution of 1 g/l PdCl_2 in HCl (pH ~ 2). After rinsing the activated copper electrode was immersed into a solution of electroless copper deposition. Electroless copper plating was carried out in a thermostatted 50 ml beaker (loading – 0.4 dm²/l). Each electroless copper plating experiment was carried out from the newly prepared solution and repeated at least three times.

Chemical composition of electroless copper plating solutions containing formaldehyde as a reducing agent and EDTA or saccharose as ligands for Cu(II) ions was as follows: 0.05 mol/l CuSO_4 , ligand (0.1 mol/l saccharose or 0.055 mol/l EDTA), 0.15 mol/l CH_2O , NaOH up to the required pH value. The electroless copper plating system with Co(II) complexes with pentaethylenehexamine (Penta) as reducing agents was as follows: 0.05 mol/l CuCl_2 , 0.15 mol/l CoCl_2 , 0.4 mol/l pentaethylenehexamine (Penta), HCl up to the required pH value. Measurements of pH were achieved using an EV-74 pH-meter and an ESL 63–07 glass electrode.

Electroless copper deposition time was 1 hour at 20 °C temperature.

Electroless coated electrodes were rinsed, dried, weighed and transferred into the thermostatted cell for electrochemical measurements for further determination of the real surface area of the coatings obtained (see below).

The electroless copper deposition in the environment of ultrasound was carried out in an Elma D-78224 ultrasonic bath (Germany), using ultrasound with frequencies of 35 kHz and 130 kHz in *sweep*, *normal* and *degas* modes.

The copper surface determination method is based on the underpotential deposition of the thallium monolayer on the Cu electrode surface and is described elsewhere [15, 17, 19]. Briefly, the procedure was as follows. The measurements were carried out at 25 °C in 1 mol/l Na_2SO_4 solution containing 1 mmol/l TlNO_3 . Before the measurements copper oxides were removed from the surface: the working Cu electrode was kept at –0.80 V for 5 s. Then the electrode was kept at +0.15 V for 5 s (for dissolving the bulk Tl deposit). A Tl monolayer was formed at –0.49 V for 200 s. Then the Tl monolayer was dissolved using anodic scanning of the potential up to –0.1 V (scanning rate 50 mV s^{–1}). By integration of the potentiodynamic curve obtained (in the range from –0.39 to –0.10 V), the quantity of electricity (Q, μC) used for anodic dissolution of the Tl monolayer was calculated.

Table 1. Results of determination of ultrasonic power transmitted to 50 ml of water in ultrasonic bath

| Ultrasound frequency, kHz | 130* | 35* |
|--|----------|----------|
| The solution temperature increase per 1 h in 50 ml vessel, °C | +20.5 | +16.5 |
| Energy necessary to warm 50 ml by 1 °C, J | 208.5 | 208.5 |
| Total energy consumed by solution for increase of temperature, J | 4 274.25 | 3 440.25 |
| Ultrasonic power transmitted to solution (50 ml), W | 1.19 | 0.96 |

* Sweep mode.

The real surface area (in nano-scale dimensions) of the Cu electrode (S_R , cm²) was calculated using the Tl monolayer capacity Q_{Tl} (the quantity of electricity necessary to form a monolayer on 1 cm² of electrode) equal in this case to 112 $\mu\text{C cm}^{-2}$ [19]:

$$S_R = Q/Q_{Tl} \quad (1)$$

The surface nano-scale roughness factor R_f is calculated as a ratio of real and geometric surface areas:

$$R_f = S_R/S_G \quad (2)$$

All represented data are average, calculated from three experiments.

RESULTS AND DISCUSSION

Calorimetric measurements of ultrasonic power

In order to compare data obtained at frequencies of ultrasound the ultrasonic power was measured calorimetrically per 50 ml of water situated in an ultrasonic bath. The data of performed experiments are presented in Table 1.

The obtained results show that under the same operating mode the ultrasonic power transmitted to the solution under calorimetric investigation (in W) depends on the selected frequency of the ultrasonic bath and is higher in the case of higher frequency (Table 1).

Electroless copper deposition using EDTA as ligand and formaldehyde as reducing agent

Conventional electroless copper plating system with ligand EDTA [17, 18] was selected for primary investigation of the effects of ultrasound. Since ultrasonic cavitation exhibits also the mixing effects, additionally experiments were carried out with the same solutions with and without ultrasonic irradiation but in the latter case using mixing by a magnetic stirrer or agitation by argon bubbles. The results obtained are presented in Figs. 2–4.

Without additional agitation of the solution, i. e. under conditions of natural convection, the rate of electroless copper deposition is the lowest and does not exceed ca. 2.1 $\mu\text{m/h}$ (Fig. 2). The use of ultrasound or other agitation methods accelerate the plating rate v_G (according to the geometrical surface area of electrode) to some extent. The acceleration of the electroless copper plating rate is observed in the following

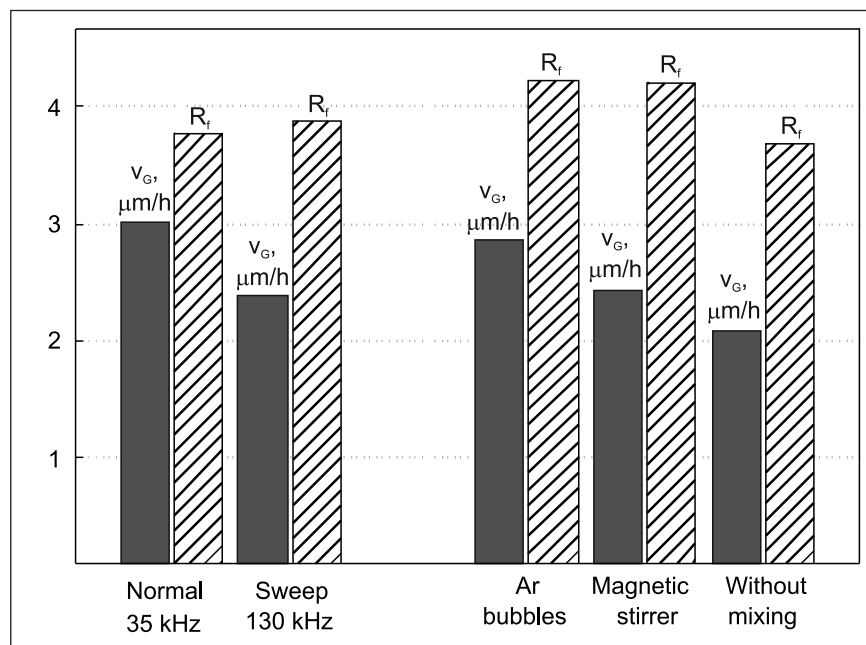


Fig. 2. Dependence of characteristics of electroless copper plating on different solution agitation methods. Solution composition (mol/l): $[\text{Cu(II)}]_0 = 0.05$, $[\text{EDTA}]_0 = 0.055$, $[\text{CH}_2\text{O}]_0 = 0.15$, pH 12.6; plating time 1 hour, 20 °C, ultrasonic bath power 100%

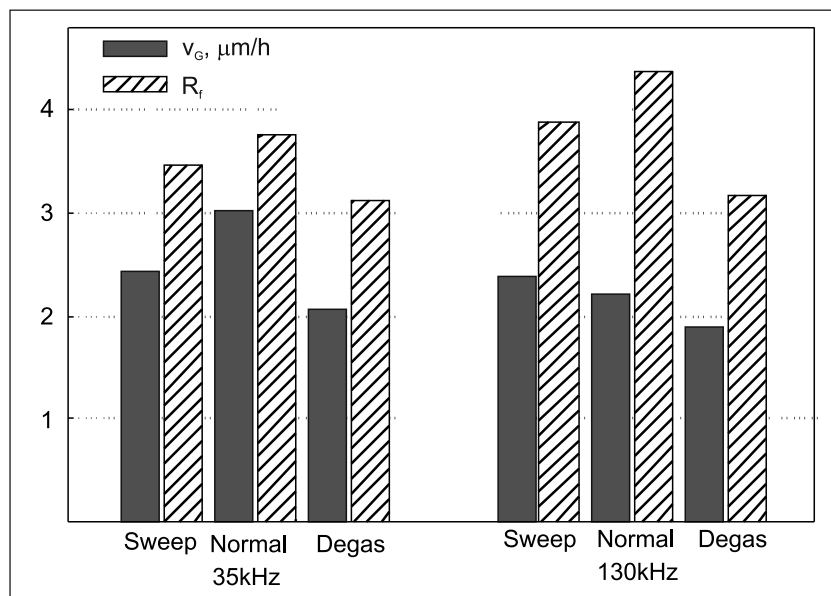
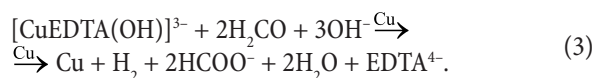


Fig. 3. Dependence of characteristics of electroless copper plating on frequencies and modes of ultrasound. Solution composition (mol/l): $[\text{Cu(II)}]_0 = 0.05$, $[\text{EDTA}]_0 = 0.055$, $[\text{CH}_2\text{O}]_0 = 0.15$, pH 12.6; plating time 1 hour, 20 °C, ultrasonic bath power 100%

sequence: magnetic stirring < US (*sweep* mode, 130 kHz) < bubbling of Ar < US (*normal* mode, 35 kHz). The highest acceleration is observed using 35 kHz ultrasound and bubbling of Ar through the solution. In our opinion, both methods create the best conditions for removal from the surface to be plated of hydrogen formed during the autocatalytic reaction of electroless copper deposition using formaldehyde as a reducing agent (3):



Since small hydrogen bubbles formed on the surface to be plated during the electroless copper deposition process block the part of the catalytic surface, their efficient removal determines the highest rates of electroless copper deposition.

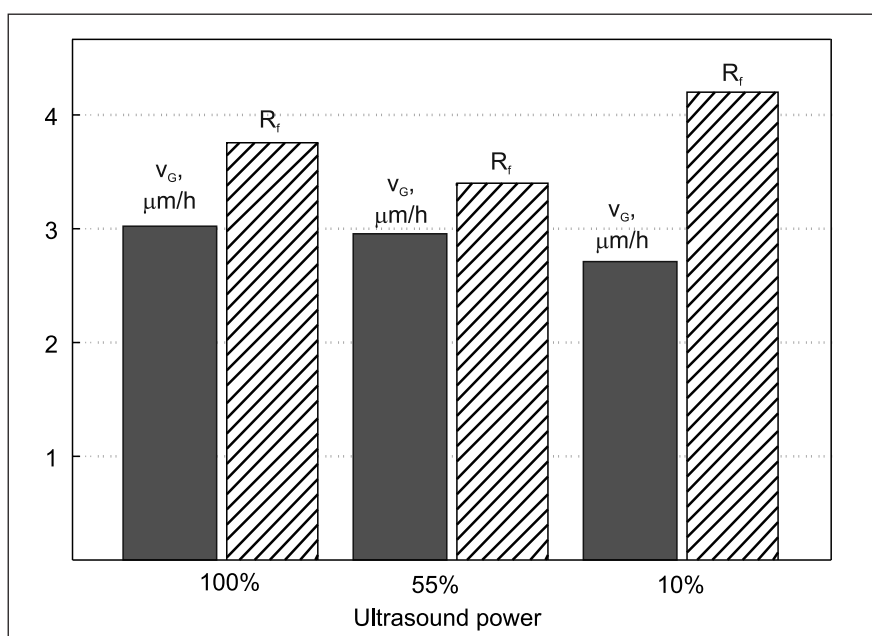


Fig. 4. Dependence of characteristics of electroless copper plating on the ultrasonic power (35 kHz, normal). Solution composition (mol/l): $[\text{Cu(II)}]_0 = 0.05$, $[\text{EDTA}]_0 = 0.055$, $[\text{CH}_2\text{O}]_0 = 0.15$, pH 12.6; plating time 1 hour, 20 °C

As it is seen from Fig. 3, the deposition rate of electroless copper is higher under ultrasonic irradiation with lower frequency (35 kHz), i. e. with lower power (see Table 1), than that under ultrasonic irradiation with higher frequency (130 kHz).

Results obtained under conditions of different ultrasonic power are presented in Fig. 4.

As can be seen from the results, the ultrasonic power reduction of up to ten times, the plating rate reduces only by 10%. Meanwhile, the effect of the ultrasonic frequency can be seen to have a greater impact on the copper deposition rate. Higher influence of changes in frequencies and lower effects of power changes were obtained also when investigating the process of electroless nickel deposition [14].

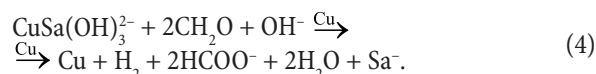
Measurements of the real surface area of the electrode allowed calculations of the surface roughness factor R_f (the ratio of real and geometric surface areas) of copper coatings obtained. During the investigation of kinetics of electroless copper deposition under impact of different factors the real surface area of the coatings obtained was also characterized. Different solution agitation methods have comparatively low influence on R_f of coatings obtained – under the conditions of ultrasonic irradiation the values of R_f increase by ca. 2–3%, whereas Ar bubbling and magnetic stirring enhance the surface development by 8–11% compared with the data obtained without mixing (Fig. 2).

When comparing the influence of the frequency of ultrasound on the surface roughness of electrolessly deposited copper coatings it can be stated that the higher frequency of ultrasound determines higher surface roughness. The mode of ultrasound also influences the surface roughness – for both used ultrasound frequencies (35 and 130 kHz) the most developed surfaces were obtained using the *normal* mode, R_f being lower at the *sweep* mode and the smoothest copper

coatings were plated at the *degas* mode (Fig. 3). It is worth noting that the roughness of copper coatings deposited at the *degas* mode is ca. 10% lower compared with the coating deposited without mixing (Figs. 2, 3).

Electroless copper deposition using saccharose as ligand and formaldehyde as reducing agent

The effects of ultrasound during autocatalytic Cu(II) reduction by formaldehyde were studied also by using other than EDTA Cu(II) ligand, namely saccharose. This ligand for electroless copper plating systems was introduced recently [15]. Like in the system with ligand EDTA, hydrogen is formed during the autocatalytic reaction using formaldehyde as a reducing agent and saccharose as a ligand (4):



Experiments using saccharose as a ligand were carried out under conditions of exposure in ultrasonic irradiation (two frequencies and *sweep* mode were applied), as well as under normal conditions, i. e. without additional impact.

Rather unexpected results were obtained in the system under investigation. The literature data indicates that under conditions of ultrasonic irradiation the increase in plating rate does not exceed 50% for electroless deposition test systems of different deposited metals [3–14], whereas in our case this augmentation was much higher.

The highest electroless copper deposition rates derived using ultrasonic irradiation were more than five times higher than that obtained under conditions without ultrasound (Fig. 5 and Table 2). Without additional impact ca. 2 μm thick copper deposits were obtained in one hour, whereas at ultrasound frequency of 35 kHz the plating rate reached 10.9 $\mu\text{m}/\text{h}$

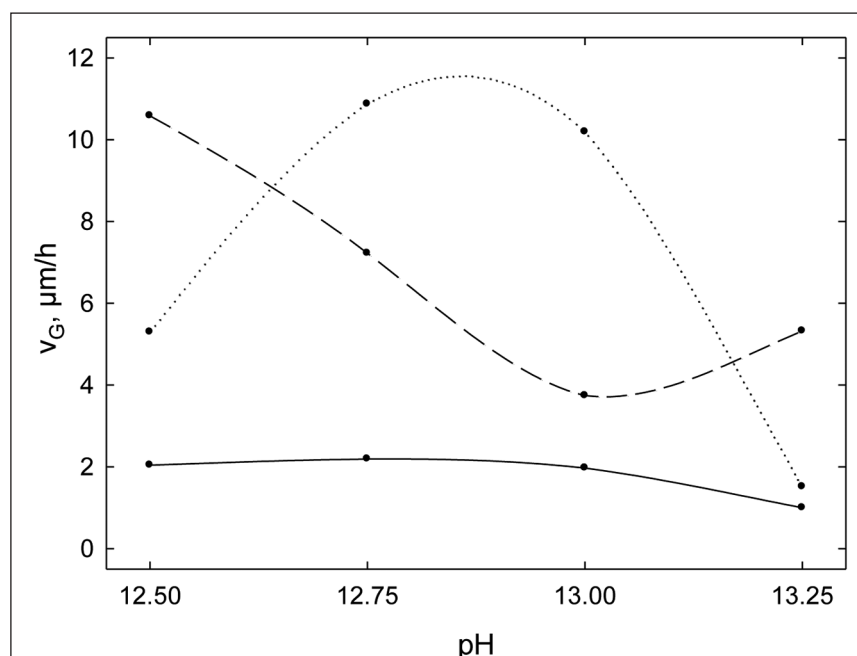


Fig. 5. Dependence of the electroless copper plating rate on solution pH. Solid line – without US, dashed one – 130 kHz US, dotted line – 35 kHz US, *sweep* mode. Solution composition (mol/l): $[\text{Cu(II)}]_0 = 0.05$, $[\text{Sa}]_0 = 0.1$, $[\text{CH}_2\text{O}]_0 = 0.15$; plating time 1 hour, 20 °C

Table 2. Dependence of electroless copper deposition rate and surface roughness of the coatings obtained on solution pH and effect of ultrasound (*sweep mode*). (Solution composition (mol/l): $[\text{Cu(II)}]_0 = 0.05$, $[\text{Sa}]_0 = 0.1$, $[\text{CH}_2\text{O}]_0 = 0.15$, plating time 1 hour)

| pH | Without ultrasound | | Ultrasound frequency 130 kHz | | Ultrasound frequency 35 kHz | |
|-------|-----------------------------------|-------|-----------------------------------|-------|-----------------------------------|-------|
| | v_{cr} , $\mu\text{m/h}$ | R_f | v_{cr} , $\mu\text{m/h}$ | R_f | v_{cr} , $\mu\text{m/h}$ | R_f |
| 12.50 | 2.0 | 8.5 | 10.6 | 13.2 | 5.3 | 13.4 |
| 12.75 | 2.2 | 25.1 | 7.2 | 8.9 | 10.9 | 20.6 |
| 13.00 | 2.0 | 13.4 | 3.7 | 12.5 | 10.2 | 21.0 |
| 13.25 | 1.0 | 9.1 | 5.3 | 6.3 | 1.5 | 11.8 |

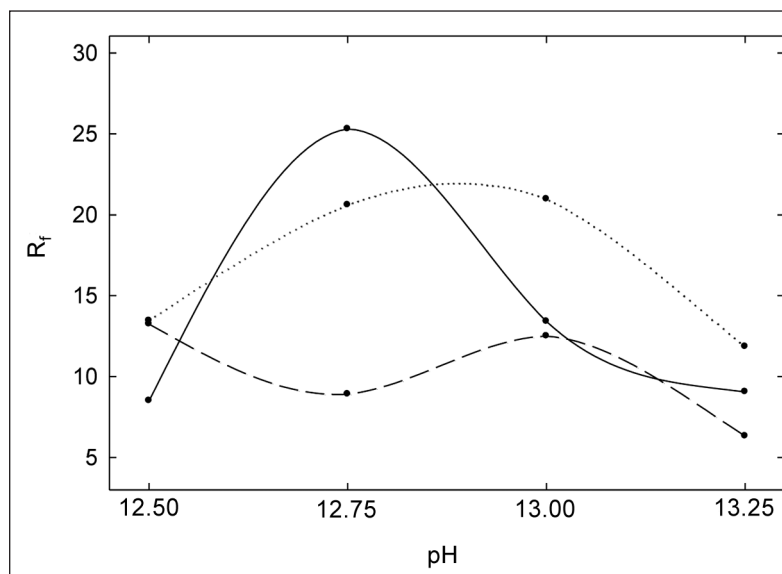


Fig. 6. Dependence of the surface roughness factor R_f of electrolessly deposited copper on solution pH: solid line – without US, dashed one – 130 kHz US, dotted line – 35 kHz US, *sweep mode*. Solution composition (mol/l): $[\text{Cu(II)}]_0 = 0.05$, $[\text{Sa}]_0 = 0.1$, $[\text{CH}_2\text{O}]_0 = 0.15$; plating time 1 hour, 20 °C

(pH = 12.75). The highest plating rate (10.6 $\mu\text{m/h}$) was observed using 130 kHz US (pH = 12.50). When using the ultrasonic irradiation the dependences of copper deposition rate on pH have extreme points, but the plating rate remains still higher than without using of ultrasound (Fig. 5). According to the data obtained there is no linear relationship between the copper deposition rate and ultrasonic power. Higher deposition rates were obtained at lower power (35 kHz frequency) than at higher power (130 kHz frequency) of ultrasound. In our opinion, this indicates that the copper coating deposition rate is determined not only by US power, but also it depends on the frequency of the ultrasound applied. Similar observation is also described in the literature [14].

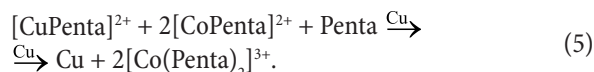
Surface roughness dependences on pH and US frequencies are represented in Fig. 6 and Table 2. At the lowest pH limit (pH 12.5) and the highest pH limit (pH 13.25) the roughness of the obtained copper coatings is very similar under conditions of ultrasonic irradiation as well as without using of US (Fig. 6, Table 2), i. e. no considerable effects of US are observed. Substantial effect was obtained at pH 12.75 – the ultrasonic irradiation diminished the surface roughness factor by ca. 20–60% (Fig. 6, Table 2). It can be also noted that in all cases the surface roughness of copper coatings deposited from saccharose containing solutions is higher than that obtained using EDTA as Cu(II) ligand (Figs. 2, 6).

It is worth noting that ultrasound affects the volume stability of the solutions for electroless copper deposition.

After 45 minutes of electroless copper plating the signs of copper(II) reduction in the volume of solutions are observed, whereas the solutions remained stable more than one day in experiments without ultrasound.

Electroless copper deposition using pentaethylenhexamine as ligand and Co(II)-pentaethylenhexamine complexes as reducing agent

The influence of ultrasound on the parameters of electroless copper plating process has been studied also in the system of new type, where the reducing agent was a Co(III)/Co(II) redox couple, and Cu(II), Co(II) and Co(III) ligand was pentaethylenhexamine (Penta). Experiments were performed under conditions described elsewhere [16]. According to the data on the stability constants of pentaethylenhexamine complexes given in the same work, the reaction of Cu(II) reduction by Co(II) in acidic solutions of pentaethylenhexamine can be expressed as follows:



As it is seen from Eq. (5), the reduction process occurs without formation of hydrogen, in opposite to the processes described above, where formaldehyde was used as a reducing agent. Therefore the effects of ultrasound could be different for systems with hydrogen-containing reducing agents and

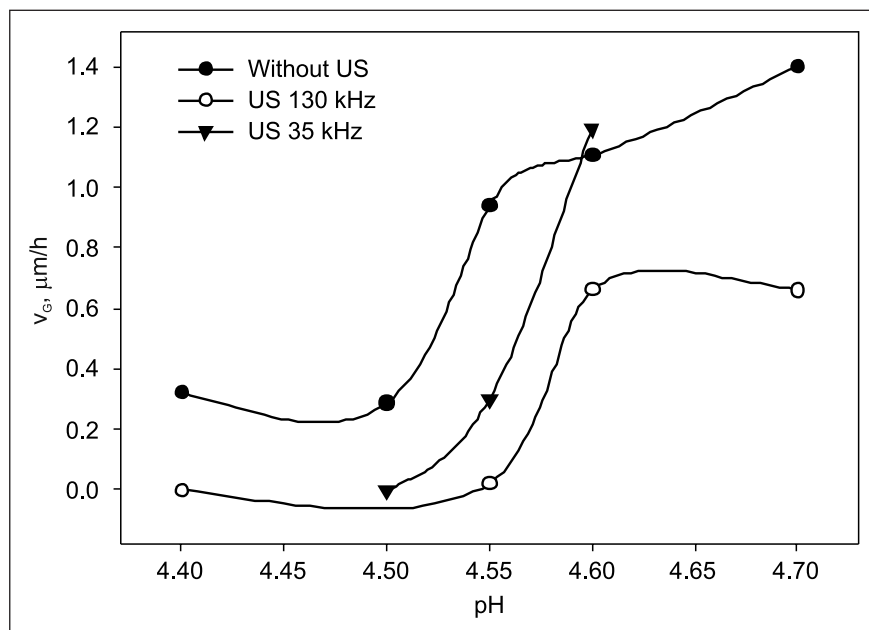


Fig. 7. Dependence of the electroless copper plating rate on solution pH. Solution composition (mol/l): $[Cu(II)]_0 = 0.05$, $[Co(II)]_0 = 0.15$, $[Penta]_0 = 0.4$; plating time 1 hour, 20 °C. Sweep mode of US

systems of metal ions with different oxidation states as reducing agents with charge transfer.

Generally, ultrasound retards (at lower pH practically stops) the electroless copper plating process in the system with Co(II) as a reducing agent (Fig. 7). This retardation is more expressed under conditions of ultrasonic irradiation with higher frequency (Fig. 7). The data obtained is in good agreement with earlier (this work) obtained results when using formaldehyde as a reducing agent. In the latter case (Eq. (5)) no hydrogen bubbles are formed and therefore no acceleration effect is observed.

Generalization

The obtained experimental results of the effects of ultrasound on systems for electroless copper plating are rather different and it is difficult to uniquely explain all systems tested.

A significant positive effect of ultrasound on the plating rate in the systems with saccharose is explained by creation of favorable conditions for removal of the reaction products with further favorable mass transfer in the system and generation of active reaction centers on the surface.

In the system with EDTA the efficiency of ultrasonic irradiation is close to systems with mixing of solutions with argon bubbles or magnetic stirring. Mechanism of US action is similar to the system with sucrose, but the changes in copper deposition rate are much lower.

The adverse effects when using ultrasound in the system with pentaethylenhexamine can be explained by the absence of gaseous products, resulting in reduced cavitation influence on the surface and overall process.

The data obtained suggest that in the case of electroless copper plating it is difficult to predict and explain apparently

the effects and mechanisms of ultrasound actions, including impact of the characteristics of ultrasonic irradiation. Therefore it remains an area for further research.

CONCLUSIONS

1. When investigating electroless copper plating in the system of Cu(II)-EDTA-formaldehyde, it was established that the efficiency of ultrasonic irradiation is close to systems with mixing of solutions with argon bubbles or magnetic stirring. Different solution agitation methods have comparatively low influence on R_f of coatings obtained – under the conditions of ultrasonic irradiation the values of R_f increase by ca. 2–3%, whereas Ar bubbling and magnetic stirring enhance the surface development by 8–11% compared with the data obtained without mixing.

2. During the research of electroless copper plating in the system of Cu(II)-saccharose-formaldehyde, it was found that under optimum operating conditions ultrasonic irradiation can increase the electroless copper deposition rate up to 5 times. The roughness of the obtained copper coatings is very similar as under conditions of ultrasonic irradiation, as well as without using of US, i. e. no considerable effects of ultrasound are observed in the pH area investigated, except solutions with 12.75 pH, where the ultrasonic irradiation diminished the surface roughness factor by ca. 20–60%. The application of ultrasonic irradiation diminishes the volume stability of the above-mentioned solutions for electroless plating.

3. Ultrasound retards (practically stops at lower pH) the electroless copper plating process in the system with Co(II) as a reducing agent. This retardation is more expressed under

conditions of ultrasonic irradiation with higher frequency. The effects obtained can be explained by the absence of gaseous products, resulting in reduced cavitation influence on the surface and overall process.

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ULTRAGARSO PANAUDOJIMAS CHEMINIO VARIJAVIMO SISTEMOSE: ULTRAGARSINĖS SPINDULIUOTĖS ĮTAKA METALO NUSODINIMO GREIČIUI IR DANGŲ PAVIRŠIUI

Santrauka

Veikiant ultragarsu (UG) cheminio varijavimo sistemose reakcijoms vykti susidaro specifinės sąlygos. Chemiškai nusodinant varį sistemose su skirtingais ligandais (EDTA, sacharozė, pentaetilenheksaaminas) ir skirtingais reduktoriais (formaldehidai, Co(III)/Co(II) redokso pora) ultragarso įtaka pasireiškia skirtingai. Priklausomai nuo cheminio varijavimo sistemos, stebimasis dangų nusėdimo greitis varijuoja nuo nedidelio sumažėjimo (ar visiško proceso sustojimo) iki maždaug penkiagubo greičio padidėjimo, palyginti su sistemomis, kai ultragaras nebuvo naudojamas. Nustatytos ir aptartos gautųjų vario dangų paviršiaus šiurkštumo priklausomybės nuo įvairių veiksnių (ultragarsinės spinduliuotės, pH ligandų ir t. t.).