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Copper etching by water-in-oil microemulsions

Y.-J. Huang, M.Z. Yates *

Department of Chemical Engineering, University of Rochester, Rochester, NY 14627, United States

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Abstract

The etching of copper was studied using an aqueous chemical etchant dispersed in water-in-oil microemulsions droplets. The aqueous phase consisted of an equimolar copper chloride/potassium chloride solution, a typical copper etchant. The phase behavior, conductivity, copper etching rate, and surface roughness after etching were studied using microemulsions formed with ionic, nonionic, and mixed ionic/nonionic surfactants. The composition of each microemulsion was fixed, and the conductivities and copper etching rate were measured as a function of temperature. The conductivity offers a route to probe ion exchange between microemulsion droplets, while the etching rate is related to the interaction of the droplets with the solid surface. The data show a correlation between etching rate and electrical conductivity of the microemulsion. For a given microemulsion, the etching rate increases as the conductivity increases. Atomic force microscopy showed that the copper surface roughness after etching with the microemulsion was lower than the surface roughness when etched without the microemulsion. The results demonstrate that it is possible to adjust the etching rate and surface roughness by changing the microemulsion composition and temperature. Confinement in water-in-oil microemulsion droplets significantly alters the transport of the etchant to the surface and may find use in nanoscale metal patterning.
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Keywords: Microemulsion; Copper; Etching; Conductivity

1. Introduction

Microemulsions are thermodynamically stable self-assembled aggregates of surfactant surrounding small droplets of either oil-in-water or water-in-oil. The microemulsion droplets are typically only on the order of 10 nm in diameter [1]. Water-in-oil microemulsions have hydrophobic surfactant tails as an outer shell and hydrophilic head groups surrounding a water droplet in the core. A variety of water-soluble compounds may be dissolved in the core of the water-in-oil droplets, including salts, proteins, and reagents to nucleate nanoparticles [2,3]. Water-in-oil microemulsions have significantly higher electrical conductivity than the pure organic continuous phase because the microemulsion droplets are able to transport charges [4,5]. The conductivity of the water-in-oil microemulsion depends on the rate of ion transport between droplets. Ion transport through the microemulsion depends on the droplet collision frequency and film rigidity, or the ability of the surfactant layers to separate and expose the water cores of two droplets. The ion exchange of microemulsions may be affected by a variety of experimental parameters such as the surfactant structure, oil type, and droplet size [6,7]. Many microemulsions exhibit a sharp increase in conductivity above a certain threshold temperature or concentration, referred to as electrical percolation [8–11].

In addition to exchanging material between droplets, microemulsions can exchange material with solid surfaces, or bulk liquid phases that they are in contact with. Water-in-oil microemulsions have been used to extract proteins from aqueous solutions, and a mechanism of mass transfer between an aqueous phase and a water-in-oil microemulsion has been presented [12,13]. Microemulsions also interact with solid surfaces, as recent studies have shown water-in-oil microemulsions rapidly dissolve salts from solids [14,15]. It is the goal of the present study to examine the relationship between electrical conductivity through the microemulsion and the rate of removal of ions from solid surfaces. The results have implications in using microemulsions in cleaning, reactions, and extractions from solids. In this paper, water-in-oil microemulsions are used to chemically etch solid copper. The water droplets contain the etching solution and the etching rate provides an indirect measure of the interaction of the droplets with the solid surface. Confinement of the chemical etchant in water-in-oil

* Corresponding author. Tel.: +1 5852732335; fax: +1 5852731348.
E-mail address: myates@che.rochester.edu (M.Z. Yates).

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microemulsion droplets is expected to dramatically alter the transport of the etchant to the surface when compared to etching with a bulk aqueous solution. In addition, the microemulsion droplets will have restricted diffusion into nanometer-scale pores or cracks. If nanometer-scale features are filled with copper, microemulsion-based chemical etching has the potential to selectively remove metal from outside of these features. As a result, metal etching using water-in-oil microemulsions may find use in nano-scale metal patterning. The microemulsion may provide a self-limiting reaction to selectively remove metal from outside nanometer scale features. In addition, the kinetics of etching can be controlled with the microemulsion, allowing better control of the amount of metal removed from the surface when compared to traditional chemical etching.

2. Methods

Sodium bis(2-ethylhexyl)sulfosuccinate (AOT, 99%), polyoxyethylene 20 stearyl ether (Brij78), *para*-xylene (99%), 2,2,4-trimethylpentane (iso-octane, 99%), *n*-propanol (99%), copper chloride ($CuCl_2$, 99%), and potassium chloride (KCl, 99.9+%) were purchased from Sigma–Aldrich and used as received. Poly(ethylene oxide)-*block*-poly(propylene oxide)-*block*-poly(ethylene oxide) (EO_6–PO_{34}–EO_6, Pluronic L62) was kindly donated by BASF and used as received. Deionized water was used for all experiments. Copper foil with the purity of 99.999% was purchased from Alfa Aesar.

Three microemulsion systems were studied: one formed with an ionic surfactant (AOT), one with a nonionic surfactant (Pluronic L62), and one containing an ionic/nonionic surfactant mixture (AOT/Brij78). The microemulsions were treated as pseudo-ternary systems with oil, aqueous, and surfactant components. The “oil” component was an organic solvent. The “aqueous” component consisted of either a copper etchant solution or pure water. The “surfactant” component was either pure surfactant or mixture of surfactant and cosurfactant held in a fixed mass ratio. Microemulsion phase behavior was determined by visual observation of an optically transparent single phase microemulsion. Ternary phase diagrams were constructed by varying the compositions of the three pseudo-components.

For each microemulsion system, electrical conductivity was measured at a water-in-oil microemulsion composition containing nearly the maximum amount of aqueous phase. For ionic and mixed surfactant microemulsions, the electrical conductivity was measured using a Brookhaven Instruments 90 Plus phase analysis light scattering instrument. For the non-ionic microemulsion, the conductivity was too low for the Brookhaven instrument so it was measured by using an Electro Scientific Industries impedance meter 251. Both instruments were calibrated using a 1000 mS/cm KCl standard solution.

The same microemulsion compositions used for conductivity measurement were used for copper etching to examine the relationship between etching rate and conductivity. Copper etching rates were measured by the weight change versus time of copper foils fully submerged in microemulsions containing a chemical etchant. The copper foils each weighed 2.79 g and were 1 mm thick. The foil was removed periodically, washed with water and acetone alternately, and then weighed. Etching rates with the microemulsions were measured at several different temperatures, depending on the phase behavior or the microemulsion. The etching rate was also measured in a similar manner using only the aqueous etchant solution for comparison to the microemulsion results. Every etching rate was the average of three measurements. The weight change was converted into a thickness change assuming a copper density of 8.94 g/cm^3.

For surface roughness analysis, copper foils of 0.025 mm thickness were used. In each case, the same size of copper foil was used (87 mg) and the etching time was adjusted so that the same amount of copper was removed (5 mg). Prior to roughness analysis, the copper samples were rinsed alternately with water and acetone, then submerged in water and placed in an ultrasonic cleaner for 20 min. The average surface roughness was determined before and after etching using a Nanoscope IIIa atomic force microscope in contact mode. A silicon tip having a nominal spring constant of 42 N/m and a nominal radius of 7–10 nm was used to scan 8 μm × 8 μm surface areas. The roughness is reported as both Ra and Rq, defined by:

$$\mathrm{Ra} = \frac{1}{L}\int_0^L |r(x)|\,\mathrm{d}x \qquad \mathrm{Rq} = \sqrt{\frac{1}{L}\int_0^L r2(x)\,\mathrm{d}x}$$

Ra is the average roughness, which is the integral of absolute value of the roughness profile over the evaluation length. Rq is the root-mean-square (RMS) average roughness.

3. Results and discussion

The basic etching reaction that was employed is: $Cu + CuCl_2 \rightarrow Cu_2Cl_2$. In this reaction, the cuprous chloride has lower solubility than that of cupric chloride, so excess chloride is needed to help dissolve the cuprous form [16]. A chemical copper etching solution consists of a mixture of $CuCl_2$ and a source for the additional chloride ions such as NaCl, HCl, or KCl. We chose KCl for all etching experiments. More favorable copper etching kinetics are obtained with high concentration of etchant, and a typical copper etching bath has a concentration of at least 1 M $CuCl_2$ and 1 M KCl (ionic strength = 4) [17]. Therefore, we investigated microemulsion phase behavior with equimolar mixtures of $CuCl_2$ and KCl. The salts do not react with the surfactants, but it is possible for the added salt to form complexes with ionic surfactants.

Fig. 1 shows the phase diagram of the Pluronic L62 microemulsion using *p*-xylene as the oil phase, and an aqueous phase consisting of either pure water or 1 M $CuCl_2$ and 1 M KCl (referred to as a 1 M etching solution). The closed circles are measured cloud points with pure water, and the open circles are cloud points with 1 M etching solution. Compositions below the line connecting the points results in an optically transparent single phase microemulsion. The phase behavior of the microemulsion containing pure water is similar to that reported in the literature [18]. When the 1 M etching solution is used as the aqueous phase, the single phase region increases slightly. With higher surfactant concentration, the microemulsion can solubilize more of the aqueous component. As a result, the curve goes

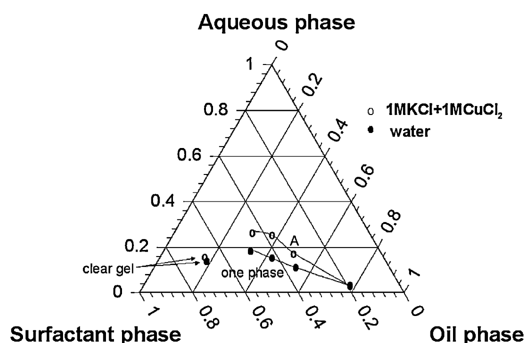

Fig. 1. Phase behavior of Pluronic L62 microemulsion with deionized water and with 1 M KCl/1 M $CuCl_2$ as the aqueous phase. The oil phase is *para*-xylene. Compositions below the curves result in a single phase microemulsion. A microemulsion composition near the point marked "A" was chosen for copper etching.

up from the right to left upon moving towards the surfactant-rich end of the phase diagram. The viscosity of the microemulsion increases as the surfactant concentration increases. At the highest surfactant concentration studied, the microemulsion forms a clear gel both with pure water and with the 1 M etching solution. The gelation points are marked with circles on the diagram. Once the microemulsion gelled, it could not be stirred and cloud point identification was difficult and not determined.

For the ionic surfactant AOT, a single phase microemulsion did not form with the 1 M etching solution because of the adverse effect that salt has on microemulsion formation. Salt screens the electrostatic repulsion between ionic surfactant head groups and can change the preferred surfactant curvature. Other authors have shown that salt reduces the single phase region of AOT/isooctane microemulsions [19]. In addition, the copper cations can replace sodium to form an ionic complex with AOT. The AOT–Cu will have markedly different surfactant properties and surfactant complex formation will reduce the amount of etchant available. The concentration of the etching solution was lowered to 0.05 M (0.05 M $CuCl_2$ + 0.05 M KCl) to allow microemulsion formation, and the ternary microemulsion phase diagram was determined as shown in Fig. 2. The open circles are visually measured cloud points with 0.05 M etching solution, and the closed circles are for the microemulsion formed with pure water. Compositions below the curves shown on the figure result in an optically transparent, single-phase microemulsion. As with the Pluronic L62 surfactant, the viscosity of the microemulsion increases as the surfactant concentration is increased. Also similar to the Pluronic L62 phase behavior, the microemulsion forms a clear gel at the highest surfactant concentrations examined. Gelation is likely due to a structural change in the microemulsion droplets from spherical to entangled cylindrical shapes [20]. The curves for the microemulsion with pure water and that with the 0.05 M etching solution appear similar, except that the single phase region is smaller with the etchant.

Mixed nonionic/ionic surfactant microemulsions have properties, such as electrical conductivity, that are intermediate between those of ionic and non-ionic microemulsions [21]. We chose to investigate microemulsion formation with a mixture of the ionic surfactant AOT, the nonionic surfactant Brij78, and the cosurfactant *n*-propanol. Short chain alcohols are often used as cosurfactants to facilitate single phase microemulsion formation [22]. The sensitivity of the microemulsion formation to ionic strength can be adjusted by adjusting the ratio of ionic surfactant to nonionic surfactant. The higher the fraction of nonionic surfactant, the less sensitive the microemulsion is to ionic strength. A surfactant mixture consisting of a 3:1:2 weight ratio of AOT:Brij78:propanol was incapable of microemulsion formation with a 0.5 M etchant solution. When the AOT fraction was lowered to 1:1:2 AOT:Brij78:propanol, the microemulsion only formed in a very small region. However, if the AOT fraction ratio was further reduced to 1:3:6 AOT:Brij78:propanol, the microemulsion formed with up to 1 M etching solution (1 M KCl + 1 M $CuCl_2$). Fig. 3 shows the phase behavior of a microemulsion formed with deionized water using an AOT:Brij78:cosurfactant ratio of 1:3:6. Fig. 4 is the microemulsion phase behavior with the same surfactant mixture using a 1 M etching solution. The microemulsion can just stabilize the etching solution and has a very small single phase region at low surfactant concentrations. A small amount of the aqueous phase is required to form the microemulsion, so it has a lower phase boundary line that is not present in Figs. 1 and 2. Figs. 3 and 4 have similar trends, with the single phase region

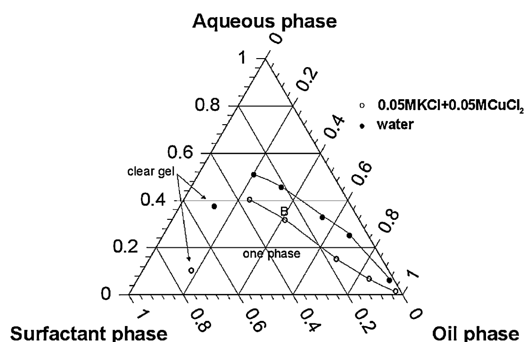

Fig. 2. Phase behavior of AOT microemulsion with deionized water and with 0.05 M KCl/0.05 M $CuCl_2$ as the aqueous phase. The oil phase is isooctane. A microemulsion composition near the point marked "B" was chosen for copper etching.

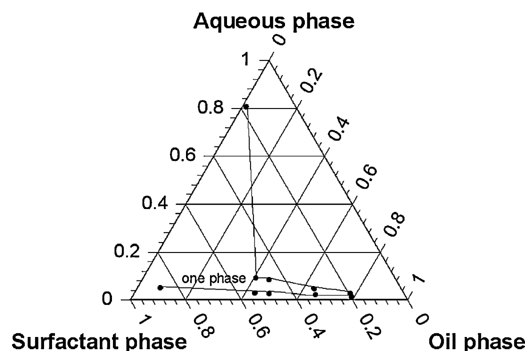

Fig. 3. Phase behavior of the microemulsion formed with a nonionic/ionic surfactant mixture consisting of AOT:Brij78:*n*-propanol in a weight ratio of 1:3:6. Deionized water is the aqueous phase and isooctane is the oil phase. Compositions between the two curves form single phase microemulsions.

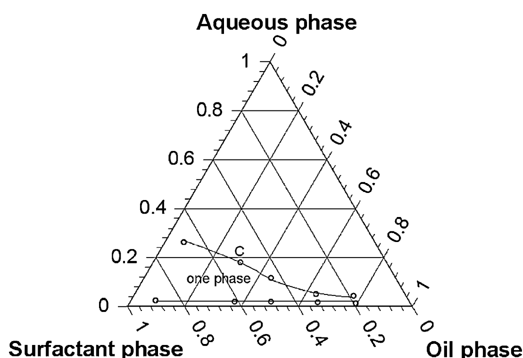

Fig. 4. Phase behavior of the microemulsion formed with a nonionic/ionic surfactant mixture consisting of AOT:Brij78:*n*-propanol in a weight ratio of 1:3:6. The aqueous phase is 1 M KCl and 1 M $CuCl_2$. Isooctane is the oil phase. Compositions between the two curves form single phase microemulsions. A composition near the point marked "C" was chosen for copper etching.

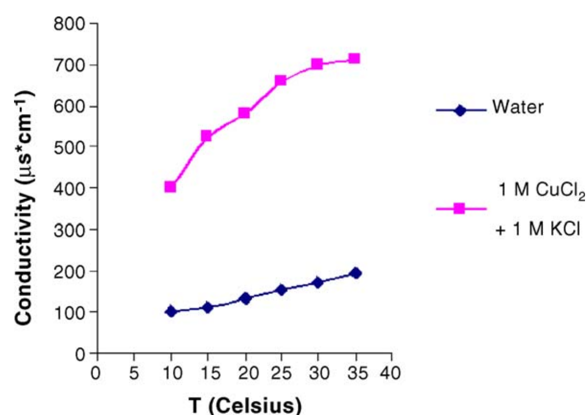

Fig. 6. Conductivity vs. temperature for the mixed nonionic/ionic surfactant microemulsion formed near composition "C" on Fig. 4, both with deionized water and with 1 M KCl/1 M $CuCl_2$ as the aqueous phase.

increasing with increasing surfactant, except that the microemulsion with deionized water has a dramatic increase a the surfactant rich end of the phase diagram.

Since the oil phase is nonconductive, electrical conductivity in water in oil microemulsions is due to the ion transport between aqueous droplets. Ion transport through the microemulsion depends on the rate of exchange of materials between droplets, and there are many factors that may affect it, such as droplet size, droplet concentration, and film rigidity [7,8]. As the volume fraction of water increases, the droplets become larger and more closely spaced, facilitating the exchange of ions between droplets. Larger droplets also experience stronger interactions that aid in exchange of material. Droplet size can be altered by many factors, such as surfactant type, solvent type, aqueous amount, and temperature. Lower rigidity of the film surrounding the droplets makes it easier for the film to open, allowing transient fusion of droplets to exchange material. The film rigidity depends on surfactant chain length and the penetration of the surfactant layer by the oil [7].

The conductivities of the three microemulsions examined are shown in Figs. 5–7. The microemulsion compositions used for conductivity are near the points marked A, B, and C on Figs. 1, 2 and 4, respectively. In each case, the conductivity goes up with increasing temperature. The increase in temperature increases droplet collision frequency and can change droplet size to increase droplet interaction. The AOT/isooctane microemulsion exhibits percolation, where the conductivity increases sharply above a certain temperature. The other microemulsions only have a gradual increase in conductivity with temperature. Increasing salt in the AOT microemulsion causes the percolation temperature to increase. Other studies have also shown that salt acts to increase the percolation temperature of AOT microemulsions [23]. The nonionic surfactant microemulsion with the surfactant Pluronic L62 has the lowest conductivity of the three microemulsions (Fig. 7). Salt increases the conductivity of the nonionic microemulsion slightly, but the conductivity is still low with the 1 M etchant solution. The mixed ionic/nonionic surfactant has moderate conductivity, intermediate between that of the AOT microemulsion above the percolation threshold and the Pluronic L62 microemulsion. The results demonstrate that the conductivity can be adjusted by changing the microemulsion structure through changes in temperature, salt concentration, and surfactant type.

For microemulsions to etch copper, the aqueous droplets must exchange material with the copper surface. It is expected that the etching rate with microemulsions will be dependent on

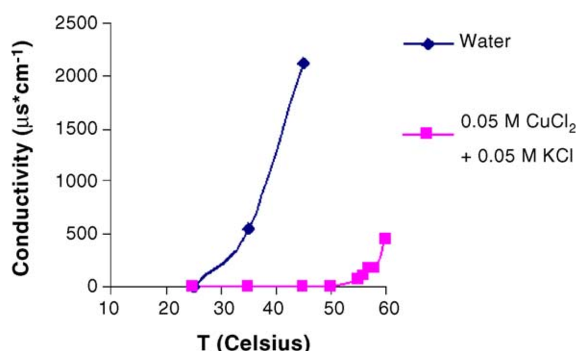

Fig. 5. Conductivity vs. temperature for the AOT microemulsion formed near the composition marked "B" on Fig. 2, both with deionized water and with 0.05 M KCl/0.05 M $CuCl_2$ as the aqueous phase. The microemulsion exhibits a sharp increase in conductivity with increasing temperature at the percolation threshold.

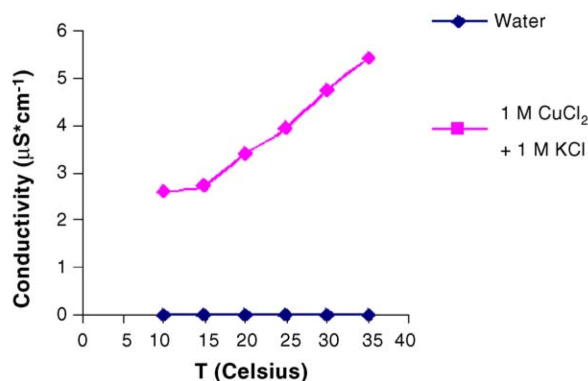

Fig. 7. Conductivity vs. temperature for the Pluronic L62 microemulsion formed near composition "A" on Fig. 1 with 1 M KCl/1 M $CuCl_2$ as the aqueous phase. For water, the black point below point A was chosen. A is in the two-phase region with water.

Table 1
Etching rates with 1 M KCl/1 M $CuCl_2$ in microemulsions

Temperature (°C)	Etching rate with 1 M KCl/1 M $CuCl_2$ alone (nm/min)	Pluronic L62 microemulsion etching rate (nm/min)	Mixed surfactant microemulsion etching rate (nm/min)
10	313	2	69
23	382	7	158

Table 2
Etching rates with 0.05 M KCl/0.05 M $CuCl_2$ in AOT microemulsion

Temperature (°C)	Etching rate with 0.05 M KCl/0.05 M $CuCl_2$ alone (nm/min)	AOT microemulsion etching rate (nm/min)
23	27	0.05
50	81	1.5
60	96	3.5

much the same things that the conductivity is dependent on; the droplet collision frequency, droplet size, droplet concentration, and film rigidity. The etching rate will also depend on the concentration of etchant used in the aqueous droplets because of its effect on the chemical reaction. With AOT, the maximum etchant concentration is much lower (0.05 M) than that obtainable with Pluronic L62 and the AOT/Brij78 surfactant mixture (1 M). Copper foils were submerged in the microemulsions, and the etching rate was determined by measuring the weight of the copper foil as a function of time. The etching rates with the Pluronic L62, and AOT/Brij78 microemulsions were compared to the rate obtained when copper foil was submerged in with a pure 1 M etching solution. Table 1 shows that the etching rate with Pluronic L62 and AOT/Brij78 microemulsions compared to the pure 1 M etching solution at room temperature and at 10 °C. The Pluronic L62 micromulsion has a much lower etching rate than the mixed ionic/nonionic surfactant microemulsion. The conductivity is also lower with Pluronic L62 than the mixed surfactant microemulsion, as shown in Figs. 6 and 7. Increasing the temperature of the microemulsion increases the etching rate for both the Pluronic L62 and mixed surfactant microemulsions. The increase in etching rate follows the trend of increasing conductivity with increasing temperature as shown in Figs. 6 and 7. For both microemulsions shown on Table 1, the etching rate is significantly lower than the control experiment with the pure 1 M etching solution. The parallel trends of microemulsion conductivity and etching rates indicate that droplet collision frequency in solution is strongly linked to the reaction and mass transport from the copper surface. In all cases, the etching was the result of diffusion only. Stirring of the microemulsion may enhance the etching rate by increasing the transport of microemulsion droplets to the surface.

The conductivity of the AOT micromemulsion shows percolation phenomena in Fig. 5 and the etching rate also follows a similar trend, as shown in Table 2. While the overall etching rates obtained with the AOT micromeulsion are very low, the rates increase strongly with temperature. In contrast, the pure 0.05 M etching solution has much lower temperature dependence. Table 2 shows the etching rate with the AOT microemulsion increases by more than a factor of two when the temperature is increased from 50 to 60 °C, while the pure etching solution has very little change in etching rate between these two temperatures. Percolation of the AOT microemulsion can result in the higher etching rates observed. Above percolation, microemulsion droplets have a very high droplet collision rate [6,7,11] so the etchant in the droplets is transported to the solid surface more readily. The correlation observed between the microemulsion conductivity and etching rate suggests that ion transport between droplets is similar to that between droplets and the solid surface. The same things that affect exchange of ions between droplets, such as interfacial film rigidity, also appear to play a determining role in the reaction kinetics with the solid surface.

Table 3 shows the surface roughness of copper etched by the microemulsions compared to that etched by the aqueous solution only. These copper samples were all approximately the same size (87 mg) and the etching time for each sample was adjusted so that approximately the same amount of copper was removed by etching (5 mg). Before etching, the average surface roughness was approximately 4 nm, so in each case etching increases the surface roughness. However, the data show that etching by microemulsions results in smoother surfaces than the ordinary aqueous etching solution. For example, the average roughness goes from 84 to 70 nm when the 0.05 M aqueous etching solution is replaced by the AOT microemulsion. For the 1 M etching solution, the average roughness decreases from 88 nm with the aqueous solution only, to 44 nm when confined in the Pluronic L62 microemulsion and 45 nm when confined in the AOT/Brij78 microemulsion. It is postulated that the microemulsions introduce selective etching. Aqueous solutions are able to completely wet the surface and easily contact the copper even inside pores, cracks, or trenches that form. Microemulsion droplets, on the other hand, will have restricted diffusion inside features smaller than the droplet size, and slow diffusion rates inside large pores. The microemulsion would more easily interact with the surface or features protruding up from the surface than inside small cracks or pores. Therefore, the microemulsion would make the surface more even. There is a potential for exploiting microemulsions in nanoscale metal patterning. The etching rate would be much slower for metal inside features smaller than the microemulsion droplet diameter than metal on

Table 3
Roughness after etching

	0.05 M KCl + 0.05 M $CuCl_2$ alone	AOT	1 M KCl + 1 M $CuCl_2$ alone	Pluronic L62	Mixed surfactant
Ra (nm)	84	70	88	44	45
Rq (nm)	110	98	109	59	57

the surface. The etching reaction would thus be self-limiting and offer a purely chemical means for removing copper from the surface while leaving copper inside small trenches or pores.

4. Conclusions

Ionic, nonionic, and mixed nonionic/ionic surfactants are capable of forming water-in-oil microemulsions with an aqueous phase consisting of a chemical metal etching solution. As expected, the nonionic microemulsion is relatively insensitive to the ionic strength of the aqueous phase, allowing microemulsion formation with more concentrated etching solution that possible with an ionic microemulsion. The electrical conductivity is a probe of droplet–droplet exchange in the water-in-oil microemulsion. The nonionic microemulsion has very low electrical conductivity and therefore, poor droplet–droplet exchange of material, even with an aqueous phase of high ionic strength. The ionic microemulsion exhibited higher electrical conductivity, but the maximum ionic strength of the aqueous phase was low. By mixing nonionic and ionic surfactants, it was possible to form a microemulsion with concentrated chemical etchant while maintaining good electrical conductivity. All three types of microemulsions exhibited an increase in electrical conductivity with increasing temperature. The ionic surfactant displayed percolation phenomena, where the conductivity increased sharply over a small temperature range. The nonionic and mixed surfactant microemulsions exhibited a gradual increase in conductivity with increasing temperature.

The rate of removal of copper from a solid foil showed that the kinetics of reaction between the microemulsion droplets and the solid surface was correlated to the electrical conductivity of the microemulsion. The etching rate with the microemulsion followed the same trend with temperature as the electrical conductivity. The results show that it is possible to tune the etching rate by adjusting the microemulsion composition and temperature. It is assumed that many of the same factors that determine droplet–droplet interactions (related to conductivity) are also important in droplet–surface interactions (related to etching rate). AFM measurment of surface roughness after etching shows that the microemulsion produces a smoother surface than does the aqueous etchant alone. We postulate that the restricted diffusion of the microemulsion droplets into small pores or cracks causes the selectivity and results in a smoother surface.

Acknowledgements

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