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ORIGINAL CONTRIBUTION

P.-L. Kuo
C.-L. Liao
S. K. Ghosh

Superior chemical–mechanical polishing performance of silica slurries made of surface-active siloxane/acrylic polymers

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P.-L Kuo (✉) · C.-L. Liao · S. K. Ghosh
Department of Chemical Engineering
National Cheng Kung University
Tainan 70101, Taiwan
e-mail: plkuo@mail.ncku.edu.tw
Tel.: +886-6-2757575 ext. 62658
Fax: +886-6-276-2331

Abstract Two new anionic polymers have been synthesized by opening the epoxide ring attached with polysiloxane (SHS) and polyacrylic (CHS) backbones with a comparable weight-average molecular weight range. The colloidal stability of the experimental silica slurries made of these polymers has been investigated and their performance in the chemical–mechanical polishing (CMP) has been studied. A nonionic polysiloxane copolymer (SHE) was also used as a cosurfactant. It was observed from surface tension and fluorescence studies that all these polymers transfer to the air–water interface before forming any aggregates and the ease of forming the aggregates is in the order CHS–SHE (1:1 blend) ≈ SHS–SHE (1:1 blend) > SHE > SHS > CHS. The apparent viscosity data and the scanning electron microscopy micrographs of the silica slurries indicate that all the polymers provide good colloidal stability over a wide range of concentrations. The superior CMP performance over commercial slurry is reported and the order of performance due to the presence of these polymers was also determined and is correlated with the dynamic contact-angle values. The results were interpreted in view of the hydrophilicity of the copolymers and their surface wetting ability.

Key words Copolymer · Anionic · Nonionic · Colloid · Silica

Introduction

With the increased scale of integration and reduced feature size, integrated circuit interconnect structures need global planarization to minimize the depth of focus constraints in the photolithographic process. Chemical–mechanical polishing (CMP) is the preferred method by which the global planarization can be achieved simply and economically, and has now become the standard ultra-large-scale integrated circuit manufacturing process for sub-0.25-μm Si devices [1, 2].

The CMP process is a complex phenomenon that involves chemical as well as mechanical interactions between the wafer materials, the slurry and the polishing pad. The polishing slurry provides the means by which both chemical and mechanical actions are used to planarize the wafer surface. The effect of the physical properties of the slurry on the polishing rate is not very well understood; however, the rheological properties of the slurries affect the material removal rate and the surface quality of the semiconductor wafers a lot [3]. The compositions of the polishing slurry play a major role in CMP as it modifies the film surface in such a way so that the materials can be removed in a smooth and uniform manner [2–5]. A typical CMP slurry always consists of an abrasive (silica or alumina) as a major component for mechanical working and remains in dispersion form generally in aqueous media. Thus, the dispersion technology of abrasives in CMP is of considerable interest to researchers in industry as well as in academia [6, 7]. The dispersants are commonly used as surface-active agents, which provide either an electrostatic barrier between the particles [8] or steric stabilization [9] against coagulation of the particles. Different commercial surfactants have been tried to manufacture CMP slurries by different manufacturers [10]; however, the dispersion agents are generally considered proprietary and are not disclosed to the users.

In our present investigation, two anionic surface-active polymers were synthesized to study their performance in CMP. A nonionic siloxane copolymer was used as a cosurfactant and the colloidal behavior of these polymers in aqueous solution and their dispersing ability towards fumed silica were studied before using these experimental silica slurries in CMP. Finally the CMP performance of the silica slurries made of these anionic surface-active polymers and the blend of anionic–nonionic surface-active polymers was investigated and compared with a commercial CMP slurry.

Experimental

Materials

The starting polymers for our present synthesis, siloxane copolymer (SG, with backbone composition,x:y = 60:40) and acrylic copolymer (PMGMMA, with backbone composition m:n = 70:30), with pendent epoxide rings were synthesized in our laboratory following methods described elsewhere [11, 12]. The nonionic siloxane copolymer (SHE) was also synthesized in our laboratory by epoxide ring-opening of SG copolymer with diethanol amine [13]. For this research SG having $\bar{M}_w = 2.98 \times 10^3$ with polydispersity ($\bar{M}_w/\bar{M}_n$) = 2.1 and PMGMMA having $\bar{M}_w = 2.37 \times 10^3$ and $\bar{M}_w/\bar{M}_n = 1.33$ were used [13] to obtain the final hydrophilic polymers SHE, SHS and CHS with comparable molecular weights. However, the molecular weights of SHE and SHS would be the same as both polymers were derived from one SG copolymer.

Anhydrous sodium bisulfite, sodium hydrogen carbonate and sodium dihydrogen phosphate were purchased from Tedia and were used without further purification. Disodium hydrogen phosphate and pyrene from Merck were also employed as received. The solvents used in this study were purified following standard methods.

The fumed silica Aerosil 90G with an average primary particle size of 20 nm and a surface area of 90 m^2/g was kindly supplied by Nippon Aerosil Co. (Japan).

Synthesis

Synthesis of siloxane copolymer SHS

SG copolymer (20 g) and ethanol (80 g) were charged in a 500-ml three-necked flask and stirried 70 °C. An aqueous solution of sodium bisulfite (14.34 g in 120 g water) was then added dropwise (about 1.0 g/min) into the reactor. After completion of this addition, stirring was continued for a further 96 h with a mild reflux at 70 °C. The reaction mixture was then concentrated by evaporating about 70% of solvent with a rotary evaporator and the residual mass was taken for purification by dialysis (cutoff molecular weight of 1,000) for 24 h. The solvent was then completely distilled off under reduced pressure to obtain the SHS copolymer and finally the polymer was dried in a vacuum oven for 48 h at 60 °C.

Synthesis of acrylic copolymer CHS

PMGMMA copolymer (20 g) and 1,4-dioxane (80 g) was added to a 500-ml three-necked flask and stirred at 70 °C. An aqueous solution of sodium bisulfite (14.34 g in 120 g water) was then added dropwise (about 1.0 g/min) and stirring was continued for a further 96 h under a gentle reflux at the same reaction temperature. Then, the CHS copolymer was extracted, purified and dried by following the same methods as described for the SHS copolymer.

Characterization of SHS and CHS

The structures of the SHS and CHS copolymers (Fig. 1) were characterized by ^{1}H NMR and ^{13}C NMR spectra (Bucker AMX-400).

SHS: ^{1}H NMR (D_2O, δ): 0.1–0.3 (–Si–CH$_3$), 0.35–0.6 (–Si–CH$_2$–), 1.4–1.7 (-Si–CH$_2$CH$_2$.-), 3.0–3.15 (–CH$_2$–SO$_3$Na), 3.4–3.6 (-CH$_2$OCH$_2$).

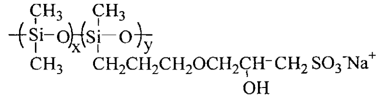
^{13}C NMR (D_2O, δ): 0.9–2.5 (–Si–CH$_3$), 14.4 (–Si–CH$_2$–), 24.0 (–Si–CH$_2$CH$_2$–), 55.5 (–CH$_2$SO$_3$Na), 68.0 [–CH(OH)–], 74.9 [–CH2-CH(OH)–], 75.3 (–Si–CH$_2$–CH$_2$–CH$_2$–).

CHS: ^{1}H NMR (D_2O, δ): 0.6–2.3 [–CH$_2$–C(CH$_3$)–], 3.12 (–CH$_2$SO$_3$Na), 3.5–3.66 (–OCH$_3$), 3.99 [–CH(OH)–], 4.33–4.43 (–OCH$_2$–).

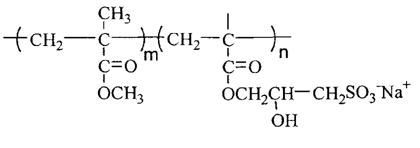
^{13}C NMR (D_2O, δ): 17.46–18.41 (–C-CH$_3$–), 44.8 (–C–), 53.0 (–OCH$_3$–), 53.8 (–CH$_2$–C–), 62.7 (–CH$_2$SO$_3$Na), 65.6 [–CH(OH)–], 69.4 (–OCH$_2$–), 178.5–179.2 (–C=O).

Methods and measurements

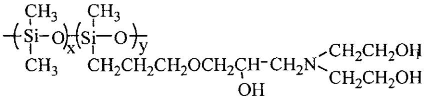
The surface tensions of all the polymers were measured by using a commercial ring tensionmeter (model TE1C, Lauda, Germany) as a function of concentration and the equilibrium surface tensions were then obtained from the long-time asymptotes at 25 °C. Fluorescence measurements were conducted by using a fluorescence spectrophotometer (model F-4010, Hitachi, Japan) using pyrene as a probe and at a fixed pH of 7.0. The intensity



SHS (x : y ≈ 60 : 40)



CHS (m : n ≈ 70 : 30)



SHE (x : y ≈ 60 : 40)

Fig. 1 Structures of the surface-active polymers ratio of the emission spectrum of pyrene at 374 and 385 nm is defined as I_1/I_3 and was measured as a function of concentration. The dispersions of fumed silica in water with different polymers were prepared by the following method. Polymeric dispersant (0.125 wt%) was dissolved in deionized water and 12.5 wt% of fumed silica was subsequently added into it and milled mechanically for 60 min at room temperature with the help of zirconium oxide beads (diameter 2 mm) at 1,000 rpm. The apparent viscosities (under a fixed shear rate) of these dispersed silica slurries were measured by using a rotating cylindrical viscometer (Brookfield DVII LVT, spindle no. 1) at 25 °C. The dry films of the slurries were deposited with a layer of platinum and the scanning electron microscopy (SEM) was carried out using an electron probe microanalyzer (Hitachi S-4200). For CMP tests the oxide thin films were prepared on 150 mm p-type Si wafers employing plasma-enhanced chemical vapour deposition. The CMP experiments were carried out on a Westech 372 M CMP polisher with a Rodel IC1400 polish pad on the primary platen, a Rodel Politex regular postpolish buffing pad on the second platen, and a Rodel R200-T3 carrier film to provide a buffer between the carrier and the wafer. The polishing parameters were down force 7 psi, platen speed 20 rpm, carrier speed 25 rpm, back pressure 3 psi and slurry flow rate 150 ml/min.

The substrate film thickness of pre- and post-CMP operation was measured with a Nanospec 210XP instrument and the dynamic contact angle between the slurry and the wafer surface was measured by using a DCA-315 (Cahn, USA).

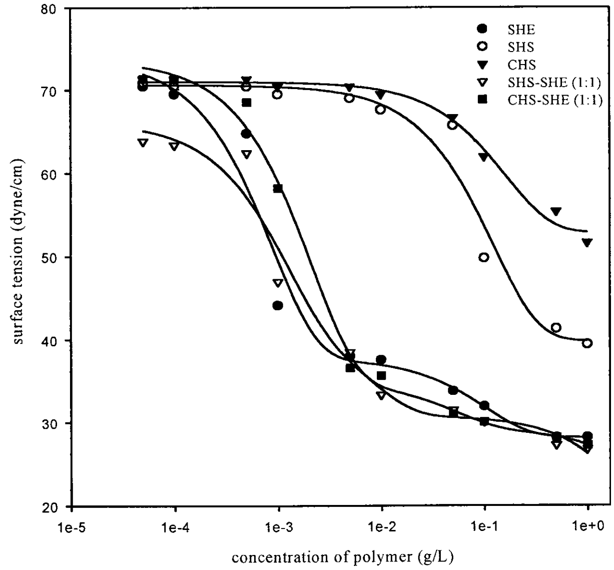


Fig. 2 The equilibrium surface tension as a function of concentration of surface-active polymers

Results and discussion

The NMR data indicate the successful synthesis of the anionic surface-active polymers. A nonionic surface-active siloxane, SHE (Fig. 1), with same weight-average molecular weight as SHS and CHS, was used as cosurfactant in the present study.

Studies of colloidal behavior

The equilibrium surface tensions versus the concentration for different polymers are plotted in Fig. 2. Since surface tension is inversely proportional to the surface-excess concentration at the air/water interface, the phenomenon where the surface tension keeps on decreasing means that the surface-excess concentration of the polymer in that solution increases proportionally [14]. It is evident from the curves that the surface tension of the CHS and SHS polymer solutions is 70 dynes/cm at very low polymer concentrations (below 10^{-3} g/l), which is close to that of pure water. However, the surface tension clearly starts to decrease at concentrations of around 10^{-3} and 5×10^{-3} g/l for SHS and CHS, respectively. This indicates that these polymers begin to transfer to the air/water interface at those concentrations. From Fig. 2 it is observed that the nonionic polymer SHE and the blends with ionic one start transferring towards the air/water interface at comparatively lower concentration (about 5×10^{-5} g/l) and the surface tension decreases gradually for all these polymer systems when the concentration increases, which indicates that the polymer concentration at the air/water interface increases gradually with polymer concentration. A surfactant with higher hydrophilicity can interact more with water molecules and has less tendency to transfer to the air/water interface. Thus, surfactants with higher hydrophilicity have less efficiency to reduce the surface tension. It is evident from Fig. 2 that the ability of reducing surface tension by the copolymers in aqueous solution is in the order SHS–SH ≈CHS–SHE≈ SHE > SHS > CHS. So the hydrophilic order of the polymers is just the reverse (CHS > SHS > SHE≈CHS–SHE≈SHS–SHE). Because the molecular weights of these polymers are nearly same, the observed order can be well explained by the presence of siloxane moieties, which make the siloxane copolymers more hydrophobic than the acrylic homologue (CHS). However, the ionic nature of SHS makes it more hydrophilic than its nonionic counterpart (SHE). The improved reduction ability of the surface tension by the blend systems (CHS–SHE and SHS–SHE) may be due to the presence of higher surface-active SHE polymer, which dominates the surface activity in the physically blended system, even when the concentration of this component is too low. A steady value of the surface tensions at high polymeric concentrations hwas also observed for all the systems. The reason behind this may be the saturation of the corresponding polymers in the air/water interface.

One of the most useful techniques to study the aggregation behavior of surface-active polymers in water involves fluorescent probes [15–18]. Recently Vorobyova and coworkers [19] extensively studied the solution properties of hydrophobically modified poly(ethylene oxide) using pyrene as a fluorescent probe and also fluorescently labeled amiphilic polyelectrolytes [20]. In this research we also employed the fluorescent probe technique to study the fundamental solution properties of the synthesized polymers and to determine the concentrations at which those polymers start forming aggregates. The plots of the I_1/I_3 values versus the corresponding polymer concentrations are summarized in Fig. 3. At low polymer concentrations, the I_1/I_3 ratios are very close to 1.76, which means there is no aggregate formation by the polymers at those low concentrations. However, the I_1/I_3 value begins to decrease at concentrations of about 10^{-2}, 5×10^{-2} and 10^{-1} g/l for SHE, SHS and CHS, respectively, and at 5×10^{-2} and 10^{-2} for the CHS–SHE and SHS–SHE blend systems, respectively, which indicates that above these corresponding concentrations the polymers begin to form aggregates. The gradual decrease of the I_1/I_3 values with increasing polymer concentration for all the polymer systems demonstrates the decreasing trend of micropolarity of the inner cores of the aggregates. This continual decrease in micropolarity can be interpreted by the increase in the aggregate size and the hydrophobic character of the inner cores of the aggregates with polymer concentration [16]. The value of I_1/I_3 at a polymer concentration of 1.0 g/l indicates the ease of forming aggregates by these polymer systems in aqueous solution and is in the order CHS–SHE ≈ SHS-SHE > SHE > SHS > CHS. This trend is again an indication of the hydrophobic order of the polymers, as we know that surfactants with higher hydrophobic character form aggregates more readily [15]. Thus, like surface tension the fluorescence spectroscopic data also coincided well with and are correlated with the construction of the polymers. These data also help us to select the polymeric dispersant concentrations for making silica slurries and the value chosen is always well above the aggregate formation concentration of any of the dispersants used [6, 21]. The aggregate formation concentration is quite similar to the critical micelle concentration (cmc) of conventional surfactants. Conventional surfactants form micelles suddenly when the concentration reaches the cmc. For polymeric surfactants, however, the polymers start to form aggregate above a certain concentration and the aggregates become closely packed gradually over a wide range of polymer concentrations [16].

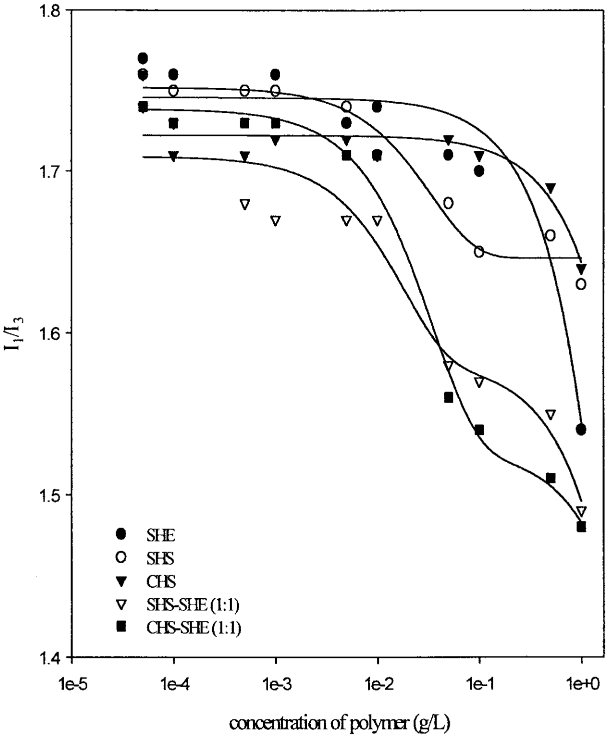


Fig. 3 I_1/I_3 as a function of concentration of surface-active polymers

Assessment of dispersing ability

For industrial applications, a good dispersant is expected to reach a low minimum viscosity at a low optimum concentration [22]. The change in the apparent viscosities of the silica dispersion in water as a function of concentration of the polymeric dispersants at a fixed pH of 10.5 ± 0.1 is shown in Fig. 4. At low concentration of the dispersants, the viscosity decreases as a function of the increasing concentration of the dispersants for each plot; however, the dispersions show a minimum in their viscosity (called the minimum

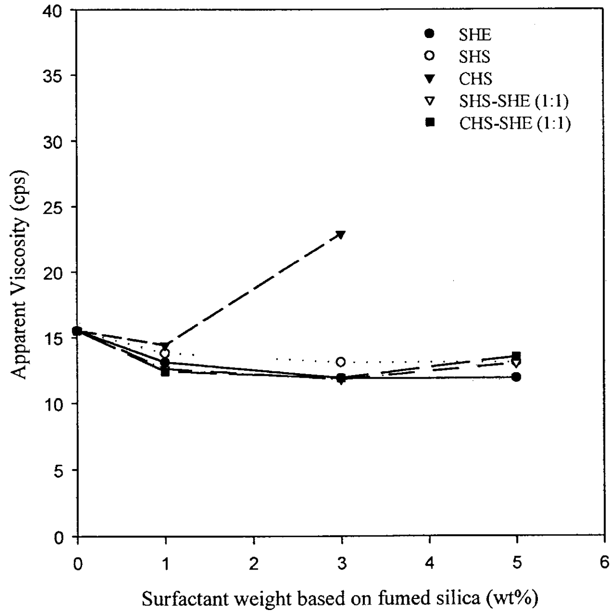


Fig. 4 Apparent viscosities of the silica slurries as a function of concentration of surface-active polymers

viscosity) at different optimum dispersant concentration. The optimum concentrations for each dispersant with respect to silica to reach its minimum viscosity are about 3.0 wt% for SHS, SHE, SHS–SHE and CHS–SHE and 1.0 wt% for CHS.

The dispersion made of CHS polymer become unstable beyond a concentration of 1.0 wt% with respect to fumed silica, indicating a sharp increase in viscosity at this concentration. The anionic polymer CHS adsorbs on the negative sites of silica particles through Na^+ counterions [23] and with increased concentration of highly hydrophilic CHS copolymer in the water phase, the ionic strength of the slurry may also increase. One possibility for the flocculation of silica slurry is due to the presence of a large number of ions in the solution phase, which enhances the charge shielding to compress the electrical double layer around the particles [6]. A further possibility is the bridging effect [24] among the dispersed particles at higher concentration, inducing the attractions between the approaching surfaces [25]. For the slurries made of dispersants containing copolymers with a more hydrophobic siloxane backbone (SHS and SHE) the viscosity remains constant after reaching the minimum. These results suggest that the increasing hydrophobicity due to the presence of the siloxane backbone helps these polymers to partition out of the aqueous solution and to adsorb onto the particle surface effectively. The stability of the slurries made of SHS polymer is the combined effect of electrostatic repulsion of bilayers on adjacent particles and the steric repulsive barrier owing to the presence of the bulky polysiloxane moiety. However for the nonionic SHE polymer the stability is purely from the steric component [6]. It is also observed from Fig. 4 that the apparent viscosity of the slurries made of the SHS–SHE and CHS–SHE blend systems reaches a constant minimum viscosity. From the plots it is clear that the presence of SHE effectively improves the stability of the slurries made of CHS as a dispersant at blend concentrations higher than 3 wt%.

The SEM technique was used to observe the surface of a film of dispersed particles directly. The silica slurries made of different individual concentrations of SHE, SHS, CHS, SHS–SHE and CHS–SHE at pH 10.5 ± 0.1 were studied by SEM micrographs under the same preparative conditions and with a magnification of 10,000×. The SEM micrographs of silica slurries made of CHS, SHS and SHE separately at their corresponding minimum viscosity concentrations and also with the CHS–SHE and SHS–SHE blend systems are shown in Fig. 5. A SEM micrograph of the blank (without any dispersant) is also shown in the figure. CHS shows the best silica dispersion as far as the SEM images are concerned; however, SEM does not provide any direct evidence for the assessment of the dispersing ability of the polymers.

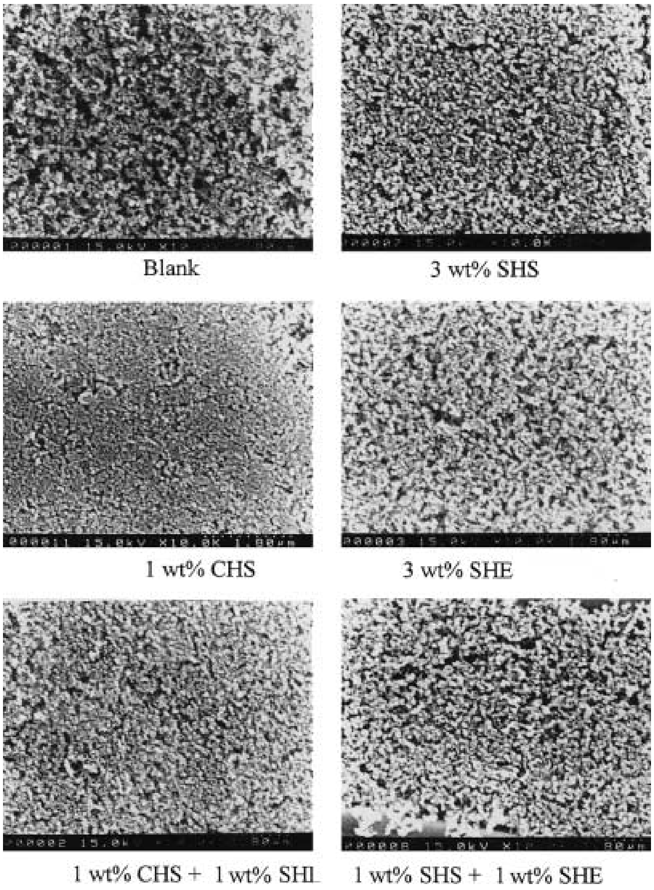

Blank
3 wt% SHS
1 wt% CHS
3 wt% SHE
1 wt% CHS + 1 wt% SHE
1 wt% SHS + 1 wt% SHE

Fig. 5 SEM micrographs of silica slurries using various surface-active polymers at pH 10.5 ± 0.1

CMP performance assessment

After a careful study of all the characteristic phenomena of the polymeric dispersants in solution as well as in the silica dispersion, we prepared silica slurries for CMP studies by dispersing 12.5 wt% of fumed silica (solid loading of most of the commercial CMP slurries varies from 5–20 wt%) in deionized water using 1.0 wt% (with respect to the solid loading) of individual or blended (1:1 by weight) polymeric dispersants. The concentration of the polymeric surfactant was chosen in such a way that it should be well above the aggregate formation concentration (Figs. 2, 3) and their adsorption on the fumed silica surface should reach a saturation level, and the dispersion is also stable.

For a semiconductor wafer, the material removal rate, the surface roughness, the number of defects and the surface flatness are the benchmarks for CMP performance [4]. The defects are the primary killers of the submicron integrated circuits formed during the design (a systematic defect) or during the process (random defects), resulting in yield loss [26]. Hence,

Table 1 Studies of chemical–mechanical polishing performance of the experimental slurries made of various surface-active polymers as dispersants

Sample[a]	Dispersants	Removal rate	Nonuniformity (%)	Contact angle
1	SHE	1,364.2	11.07	37.78
2	SHS	1,363.6	10.08	31.17
3	CHS	3,562.4	5.47	19.08
4	SHS–SHE	1,615.6	8.76	26.91
5	CHS–SHE	3,306.6	2.66	16.90
6	Unknown[b]	1,597	8.00	–

[a] Silica 12.5 wt% and dispersant 0.125 wt%

[b] Commercial chemical–mechanical polishing slurry (SS-25, Cabot Corporation)

detects and monitoring of defect density is critical for maintaining an acceptable yield performance; however, a detailed study of the defects is beyond the scope of our research. In this study we measured the nonuniformity as the defect that formed on the silica wafer surface during the CMP process. Defects in the wafers can also appear owing to the formation of scratches, which may be due to the presence of large particles in the slurries. It is considered preferable to use soft abrasive grains having small diameters and uniformly dispersed in the solvent to prevent mechanical damage on products and to obtain a high-precision finished surface [27].

The polish rate and the nonuniformity were calculated from the film thickness difference between pre- and post-CMP operation and the removal rate defined as

$$\text{Removal rate} = \frac{(\text{pre-CMP thickness}) - (\text{post-CMP thickness})}{\text{Polishing time}} .$$

The wafer nonuniformity was defined as the difference between the maximum and minimum removal rates divided by twice the average removal rate [4]. The CMP removal rates and the nonuniformity with the experimental slurries are shown in Table 1. Under the same machine parameters, slurries made of CHS polymer as dispersant gives rise to higher removal rates followed by CHS–SHE blend $\gg$ SHS–SHE blend $>$ SHE $\approx$ SHS. The nonuniformity of the corresponding silica wafer surface after polishing with the slurries made of these polymers was measured and the performance order found is CHS–SHE < CHS < SHS–SHE < SHS < SHE. The same order was also observed for the contact angle, which suggests that the slurries with smaller contact angle can be spread more homogeneously on the wafer surface and provide better polishing [28]. The CMP performance largely depends on the chemical components that act isotropically on the wafer surface and the mechanical action of the abrasive particle, which is accomplished by the particle size in the dispersion, the concentration and also the slurry pH [29]. The contact-angle data imply that an anionic dispersant with a hydrocarbon backbone (more hydrophilic) helps to reduce the surface energy of the wafer substrate to a larger extent to provide better wetting than its siloxane counterparts. A low contact angle, i.e., a better surface wetting, promotes the chemical reaction between the slurries and wafer substrate to obtain a better removal rate; however, the presence of nonionic siloxane surfactant (SHE) may have a further effect to reduce the surface energy of the wafer substrate and the particle size of the silica dispersion to obtain the lowest contact angle and nonuniformity. These experimental slurries were also compared with the very popular commercial slurry SS-25 (Cabot Corporation) under the same experimental conditions and the results of experimental slurries 3 and 5 (Table 1) show better performance with respect to their removal rate and the nonuniformity of the wafer surface after CMP action.

Conclusion

Two surface-active anionic copolymers with polysiloxane and polyacrylic backbones (SHS and CHS) with comparable weight-average molecular weights were successfully synthesized. The study of the colloidal behavior of these polymers along with a nonionic polysiloxane copolymer (SHE) as a cosurfactant showed the order of the hydrophilic nature of the polymers to be CHS > SHS > SHE > CHS–SHE > SHS–SHE. The maximum stability of the silica dispersion for CMP use was obtained at a concentration of 3.0 wt% (with respect to the weight of the abrasive) for all the polymers except CHS, which shows the maximum stability when its concentration is only 1.0 wt%. However, the presence of the nonionic cosurfactant SHE system improved the performance of anionic–nonionic blend systems. The CMP slurries made of CHS and the CHS–SHE blend showed the best performance so far as the removal rate and the nonuniformity of the wafer substrates are concerned and were found to be much superior to commercial slurry SS-25 under the same experimental conditions. The performance order accorded well with the dynamic contact-angle values.

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