

Preparations of Silica Slurry for Wafer Polishing via Controlled Growth of Commercial Silica Seeds

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Abstract—Silica slurry in aqueous medium for wafer polishing was prepared by sol-gel reaction of silicon alkoxide utilizing commercial silica particles as seeds that were grown stepwise through intermittent additions of tetraethylorthosilicate (TEOS) as a silica precursor. Before the growth reaction, the commercial silica particles were pre-treated in the vibratory mill partially filled with zirconia ball and the sonicator to ensure good dispersion. The alcohol left after growth reaction was removed by vacuum distillation and repeated washings with distilled water followed by centrifugations. Then, the alcohol-free silica particles were redispersed in water. The dispersion stability of the silica slurries was examined by measuring surface charge of silica particles and rheological properties. Finally, wafer-polishing performance of the prepared silica slurries was considered by measuring the polishing (or removal) rate, and RMS (root mean square) roughness of the polished wafer surface. For the polishing, MEA (monoethanolamine) and TMAH (tetramethylammonium hydroxide) were used as polishing accelerators. The polishing result showed that the removal rate was nearly independent of the concentrations of MEA and TMAH in the range of 0.3-0.5 wt% and 100-500 ppm, respectively. One of the most interesting features is that hydrothermal treatment of the prepared silica slurries in autoclave increased the removal rate as high as ten times. Although the removal rate was increased by the increased size of the abrasive particle, surface roughness of the polished wafer surface was deteriorated.

Key words: Silica Slurry, Particle Growth, Wafer Polishing, Surface Roughness

INTRODUCTION

Slurries of amorphous silica particles have numerous applications in catalysis, separation processes, composite materials, micro-electronics, and others. Aqueous silica slurries have been also used in the wafer polishing process, which is the final step in manufacturing the silicon wafer by crystal growth and wafer preparation steps [Sze, 1988]. For many years, the planarization of polysilicon wafers has employed polishing slurries containing abrasive colloidal silica particles dispersed in aqueous alkaline solution. Generally, a wafer is polished by simultaneous chemical and mechanical actions [Sze, 1988; Sasaki and Maeda, 1994; Chang and Sze, 1996]. Rapid oxidation of silicon-to-silicon oxide by the solution softens the surface of silicon wafer. In aqueous medium, hydroxyl groups are formed on the silicon oxide surface, and hydroxyl-hydrogen bonding or molecular bonding occurs between slurry particles and wafer surface. Then, Si-Si bonds on the wafer surface are broken when the slurry particles move away. Thus, the silica particles in the polishing slurry serve as fine abrasives removing micro peaks from the wafer surface caused by earlier processing steps [Sze, 1988; Chang and Sze, 1996; So et al., 2001a]. This simultaneous chemical and mechanical polishing (so-called CMP process) is also applied to global planarization of multilevel interconnections of ultra large-scale integrated circuits [Yasseen et al., 1997].

Amorphous silica particles have been synthesized by various ways and can be classified by synthetic route in liquid or vapor phase.

Amorphous silica prepared in liquid phase is called precipitated or colloidal silica, and vapor-phase synthesis produces fumed silica. These amorphous silica particles are commercially available from such companies as DuPont and Degussa. Most precipitated or colloidal silica particles are synthesized by the formation of active silicic acid from a dilute aqueous solution of water glass followed by nucleation and polymerization [Iler, 1979]. In those processes, purification of silica sol or removal of impurities is important. In particular, low sodium content is preferred and extensive studies have been reported on the preparation of colloidal silica with low-sodium content [Romberger and Payne, 1991; Deal and Werho, 1992]. Fumed silica particles synthesized by pyrolysis of SiCl_4 have irregular shape and wide size distribution relative to colloidal silica. During the synthesis, fumed silica particles tend to fuse together to form larger aggregates irreversibly [Eisenlauer and Killmann, 1980].

Stöber et al. [1968] synthesized monodisperse silica particles by hydrolysis and condensation of silicon alkoxide and showed that the control parameters in determining final size were the concentration of ammonia, water, and types of alkoxide precursor and alcohol solvent. Since this sol-gel method has many advantages in preparing advanced materials at low temperature with high purity, much work has been done, especially in the development of the fine ceramics and the advanced functional materials [Stöber et al., 1968; Brinker and Scherer, 1990; Pierre, 1998]. Many related studies have focused on the control of final particle shape, size and its distribution [Tan et al., 1987; Blaaderen and Kentgens, 1992; Konno et al., 1994; Buining et al., 1996; Satoh et al., 1997; Oh et al., 1999]. In spite of numerous studies, silica particles formation through the hydrolysis and condensation of silicon alkoxide have not been

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used commercially because this route can produce monodisperse silica dispersions only with very low particle loadings [Bogush et al., 1988]. Also, the relatively high cost of silicon alkoxide makes this synthetic route less promising. To overcome these drawbacks, a seed growth technique has been conducted by adding reactants stepwise [Bogush et al., 1988; Han et al., 1999; So et al., 2001b, c]. The seed is either commercial Ludox silica [Coenen and de Kruif, 1988] or alcohol that is made by dilution of an aqueous sodium-silicate solution with excess ethanol [Buining et al., 1996]. Although a number of studies have been conducted, the Stöber silica particles have not been commercialized yet because of embedded micropores and poor hardness compared to fumed or precipitated silica particles.

In this study, amorphous colloidal silica suspensions were prepared by controlled growth of commercial seeds such as colloidal silica and fumed silica. To do this, the silica precursor of TEOS (tetraethylorthosilicate) was added sequentially. The stepwise growth method was promising in overcoming some drawbacks of Stöber's method mentioned above. We also considered applicability of the prepared silica slurries to the wafer polishing process by measuring the silica surface potential as a function of pH and the slurry viscosity as a function of shear rate. These material properties were used to determine the level of phase stability required by wafer polishing slurries. In addition, the polishing performance of the prepared slurries was examined by measuring the removal rate of polysilicon wafer and the surface roughness of polished wafer surface using AFM (atomic force microscopy). Finally, the silica abrasives were treated hydrothermally in an autoclave to improve the hardness of abrasive silica particles before polishing.

EXPERIMENTAL

1. Preparations of Silica Particles

The silica particles were prepared in water-ethanol mixture at 25 °C using stepwise growth reaction [Bogush et al., 1988; Coenen and de Kruif, 1988; Oh et al., 1999]. Fumed silica (Aerosil OX-50, Degussa) and colloidal silica (Ludox AS-40, DuPont) were used as seeds in the growth reaction. The seed particles grew through the hydrolysis and condensation of TEOS ($\text{Si}(\text{OC}_2\text{H}_5)_4$, tetraethylorthosilicate, Aldrich), which was added sequentially with a time interval of 12 hr. To obtain spherical silica particles, the growth reaction proceeded under basic condition using ammonium hydroxide (Aldrich) as a morphological catalyst. Detailed reactant compositions and particle size determined by TEM are given in Table 1. All re-

agents were used as received, except twice distilled ethanol (Oriental Chem.). Three types of silica suspensions were prepared by using different seed particles, which are conventional Stöber silica, Aerosil OX-50 and Ludox AS-40 (hereafter designated by PT, AG and LG, respectively). In Table 1, the number right next to the abbreviation indicates the number of the growth steps.

After the growth reaction was completed, the prepared particles were dispersed in organic ethanol medium. The organic ethanol medium must be substituted by aqueous medium for the purpose of wafer polishing. Therefore, ethanol was removed by vacuum distillation and the particles were redispersed in distilled water. The silica loading was adjusted to 12 wt% and the silica slurry was formulated by adding polishing accelerator such as MEA (monoethanolamine) and TMAH (tetramethylammonium hydroxide). Finally, the silica slurry was diluted to 10 wt% of silica content.

The particle size and its distribution were measured by using transmission electron microscopy (TEM, EM912, Carl Zeiss) in combination with electrophoretic light scattering (Zetaplus, Brookhaven). Moreover, the ζ -potential of silica surfaces at various pH and the effective diameter under the action of electrostatic repulsion were also measured. The pH of silica suspension was adjusted by 1 N hydrochloric acid and 1N sodium hydroxide (Yakuri).

2. Wafer Polishing

In the wafer polishing test, lapped silicon wafers of 4 inch in diameter (LG Siltron) were used. Wafer polishing was conducted by a wafer polisher (Logitech; PM5) with polyurethane polishing pads (Rodel; Suba 600). Removal rate of a wafer surface during polishing was determined from the weight loss during wafer polishing. Each silicon wafer was cleaned with SC1 standard cleaning solution with the composition of $\text{NH}_4\text{OH} : \text{H}_2\text{O}_2 : \text{H}_2\text{O} = 1 : 1 : 10$, and subsequently with deionized water. Before a wafer was loaded into the polisher, the pad temperature was stabilized at around 30 °C. Seasoning of the pad was conducted with pad conditioner so that the pad was sufficiently wetted. Usually, 30-min pre-rotation was required to stabilize the rotations of platen and pad temperature before a wafer was loaded. The polishing slurry was diluted with deionized water to 2 wt% of silica content. After the polishing was completed, the polished wafer surface was washed with deionized water, and the pad conditioning was conducted for 1 minute. The morphology of the polished wafer surface and its roughness were examined by AFM (atomic force microscopy) with a scan area of $5 \mu\text{m} \times 5 \mu\text{m}$.

RESULTS AND DISCUSSIONS

Table 1. Compositions of the controlled growth reaction and particle size distributions

	TEOS [M]	H ₂ O [M]	NH ₃ [M]	Seed particle	Average radius
PT-1	0.38	10.46	0.18	-	105.0±0.7
PT-3	0.38+0.25+0.13	4.09	0.13	-	62±2.2
AG-1	0.38	10.46	0.18	Aerosil OX50 0.07 M	124.0±40.4
AG-2	0.38+0.25	10.46	0.18	Aerosil OX50 0.07 M	-
AG-3	0.38+0.25+0.13	10.46	0.18	Aerosil OX50 0.07 M	-
LG-1	0.38	4.09	0.13	Ludox AS40 0.24 M	-
LG-2	0.38+0.25	4.09	0.13	Ludox AS40 0.24 M	-
LG-3	0.38+0.25+0.13	4.09	0.13	Ludox AS40 0.24 M	47.3±8.6

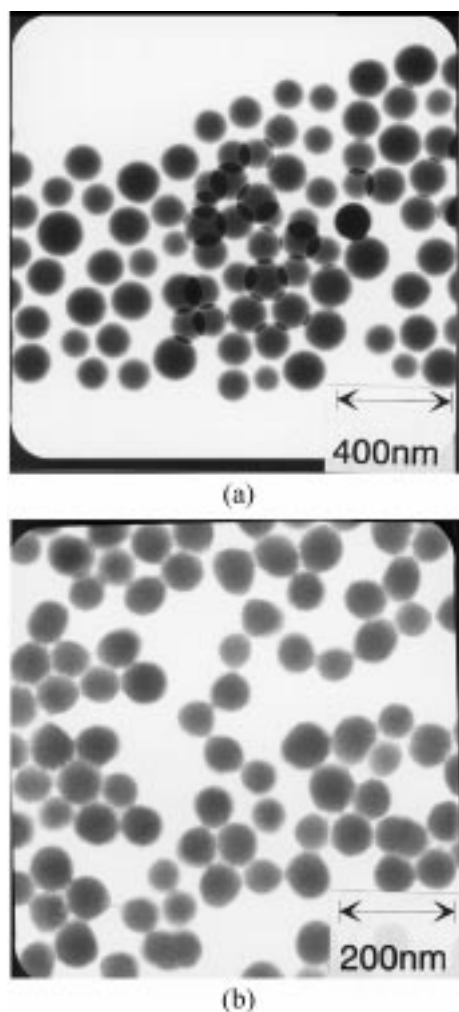


Fig. 1. Silica particles prepared (a) by conventional Stober method (PT-1); (b) by three-step addition of TEOS (PT-3).

1. Preparations of Aqueous Silica Suspensions

In the controlled growth of silica particles from seeds, the particle size was controlled by the sequential additions of TEOS. First, in order to check this size tailoring with the concentration of the added TEOS, silica suspensions, PT-1 and PT-3, were prepared by intermittent addition of TEOS without seeds (see Table 1 for the reactant compositions). The transmission electron microscope images of the prepared particles are shown in Fig. 1. It can be noted that spherical silica particles were produced successfully as reported by Bogush et al. [1988]. As expected, however, the stepwise sol-gel reaction produced silica slurries with very low mass fractions of silica particles, which displayed somewhat broad size distribution. Moreover, when silica particles are grown directly without seeds, there is a certain critical concentration of silicon alkoxide in the stepwise growth reactions above which bidisperse silica particles are formed [Bogush et al., 1988]. Therefore, it was necessary to synthesize silica particles by stepwise growth of seeds. We used commercially available fumed and colloidal silica particles. Although cheap, fumed silica particles are irregular in shape and have a broad size distribution. Meanwhile, colloidal particles are spherical and monodisperse [Coenen and de Kruif, 1988]. In the present study,

we compared morphological features of the prepared silica particles grown from seeds of fumed and colloidal silica particles.

During the growth reaction, the initial dispersion stability of the commercial seeds was extremely important in order to prepare mo-

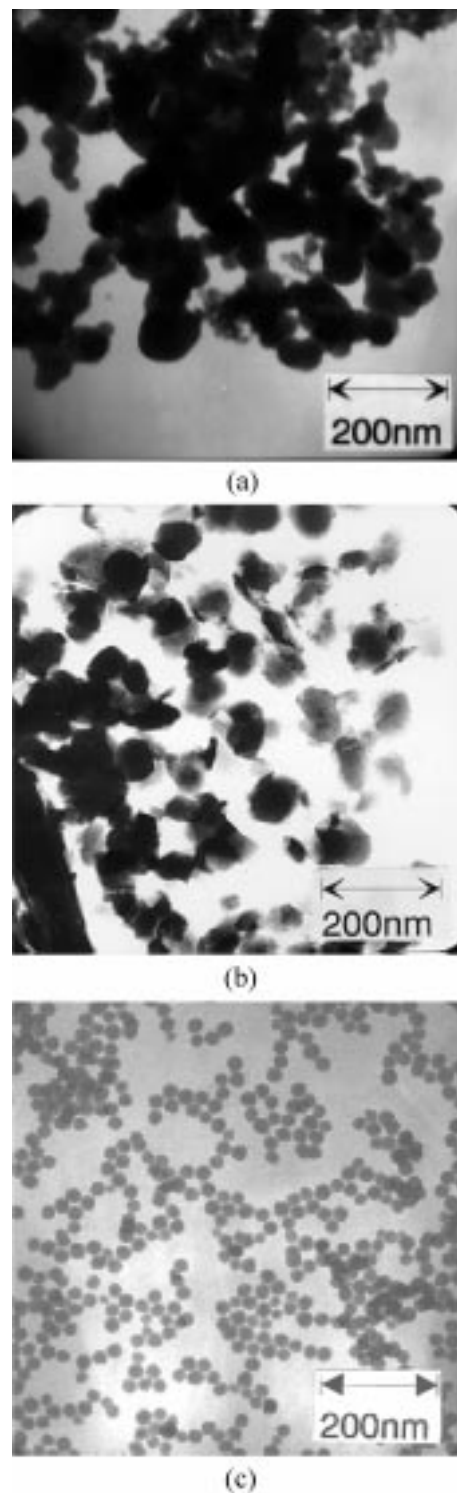


Fig. 2. Commercial silica particles used as seeds in the growth reactions: (a) Aerosil OX-50 without pretreatment; (b) Aerosil OX-50 pretreated with paint shaker using zirconia ball; (c) Ludox AS-40.

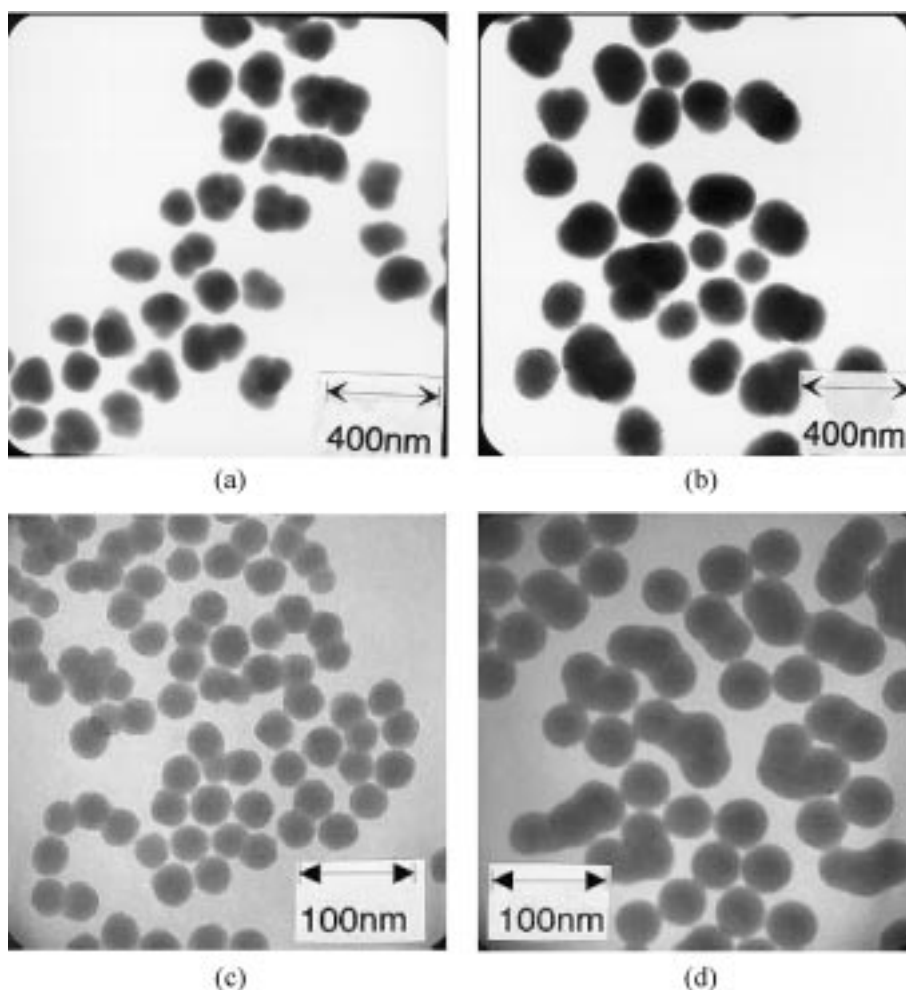


Fig. 3. Silica particles grown from the pretreated seeds by stepwise addition of TEOS.

(a) AG-1, (b) AG-3, (c) LG-1, (d) LG-3

nodisperse particle dispersions. If the seed particles had aggregated or flocculated structure, they were not able to grow uniformly and formed highly aggregated and unstable particle dispersions. In this case, rapid formation of silica sediment was observed during the growth reaction. In particular, fumed silica particles synthesized by flame hydrolysis displayed a fractal structure composed of fused primary particles in the dispersion, unlike colloidal silica. Therefore, the seeds of fumed silica were treated before growth by using proper methods such as precipitation, filtration, sonication, milling and others. In this study, the fumed silica (Aerosil OX-50) seed dispersion of 15 wt% was pre-treated with a vibratory mill (Paint shaker, ASADA) containing zirconia balls for 6 hr. After the milling process, the aqueous seed particle suspension was sonicated vigorously for 30 min to ensure its dispersion stability. TEM images of the commercial seed particles used in this work are shown in Fig. 2. It can be easily seen from Fig. 2(a) that the fumed silica particles (Aerosil OX-50) dispersed in water with no pretreatment had a broad size distribution and formed large aggregates. As expected, the size of aggregates was much larger than the primary particle size of 44 nm in diameter. Fumed silica seed particles pretreated with the vibratory mill for 6 hr are also shown in Fig. 2(b). Indeed, the pretreatment enhanced the dispersion stability greatly. In addition, un-

treated Ludox silica of 20 nm in diameter formed loose aggregates as noted from Fig. 2(c). These colloidal silica seeds were also sonicated for 30 min beforehand to improve dispersion stability. With the pre-treated commercial silica seeds, silica suspension was prepared through the controlled hydrolysis and condensation of TEOS under basic condition. Each reactant composition in the growth reaction is shown in Table 1. The growth reaction at each step proceeded as TEOS was added intermittently with a time interval of 12 hr. In order to confirm the controlled growth reaction, the silica particles prepared by a single-step growth and a three-step growth were observed by TEM (see Fig. 3). The effective diameters and particle size distributions measured by electrophoretic light scattering were also plotted as a function of the TEOS concentration in Fig. 4(a) and (b), respectively. It can be easily seen that the seed particles become larger as the hydrolysis and condensation reactions of TEOS proceed. Consequently, the final weight fractions of solid content were also increased. Moreover, as the growth steps proceed, the particles grow and size distribution becomes somewhat narrow. However, AG-3 contains too large particles for wafer polishing. Therefore, wafer polishing was conducted only using AG-1 & LG-3 silica abrasives. Average radius and standard deviation of AG-1 & LG-3 measured from TEM images are included in

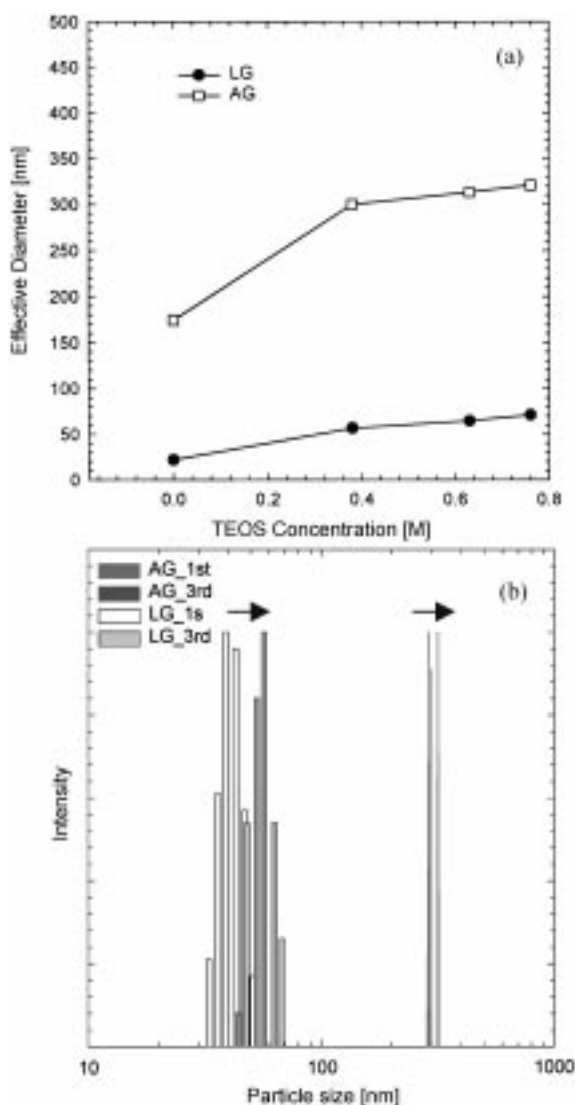


Fig. 4. Particle size and size distribution measured by electrophoretic light scattering (a) Particle size change during the growth reaction, (b) Particle size distribution changes during growth reaction.

Table 1. The particle size measured by light scattering often reflects the effective diameter when the primary particles form aggregates. Therefore, the particle size measured by light scattering is somewhat larger than that from TEM images, which reflect the primary particle size. This is because the particles tended to form loose aggregates as the amount of added TEOS increased. The electrophoretic light scattering cannot distinguish a single particle from a loose aggregate, which is counted as a 'large' particle. Thus, the electrophoretic light scattering reflects only the effective particle size.

Meanwhile, silica particles grown from the fumed silica, which were irregular in shape, showed rather broad size distribution compared with silica particles grown from colloidal silica. This can be expected from the fact that the fumed silica seeds used in the growth reaction were still nonspherical with a broad size distribution in spite of several steps of the pretreatment. As the growth step proceeded, the particle size clearly increased. In the subsequent polishing test, one-step growth fumed silica (slurry AG-1) and three-step growth

colloidal silica (noted as LG-3) were used as polishing abrasives.

As mentioned earlier, the solvent ethanol used in the growth step was replaced by water, for the purpose of wafer polishing, through distillation at 40 °C and 13–40 kPa using a vacuum evaporator (Heidolph). To ensure no residual ethanol, the supernatant separated from the silica dispersion by centrifugation was analyzed with a gas chromatograph after the solvent was exchanged. In addition, the particle size was measured again by electrophoretic light scattering. These analyses confirmed that no measurable residual ethanol was present and the particle size distribution remained unchanged.

2. Dispersion Stability

The colloidal dispersion stability is very important for practical applications. Particularly, the dispersion stability is required for long-time shelf storage of the silica slurry. In order to enhance the dispersion stability of the prepared silica slurry, either electrostatic or steric stabilization can be used [Tadros, 1978; Hunter, 1989; So et al., 2001a]. In this study, the electrostatic interaction was checked by monitoring the ζ -potential of aqueous silica particles in the dilute regime. The ζ -potentials of AG-1 and LG-3 silica particles at various pH's are shown in Fig. 5. The isoelectric point of silica was about pH=2–3, which coincided with other previous reports [Tadros, 1978; So et al., 2001a]. For both the silica particles, the surface potential decreased from positive to negative as pH increased. In the particle preparation, ammonium hydroxide was used as a morphological catalyst. Therefore, the particle suspension remained at high pH around 10–11 during the synthetic step, which produced strong electrostatic repulsion. Moreover, pH was kept at relatively high value of about 9 during the solvent exchange step to prevent aggregation.

The dispersion stability of AG-1 aqueous silica suspension was also examined by measuring the shear viscosity as a function of the shear rate (Fig. 6). The shear viscosities of slurry AG-1 with 10 wt% silica content were given as a function of the shear rate at two different pH=3 and 9. At pH=3, which is close to the isoelectric point, slurry AG-1 showed a very rapid shear thinning behavior, and the slope of shear viscosity versus shear rate in log-log scale was -1 at low shear rates. The slope of -1 means that yield stress developed, which was indicative of the formation of flocculated

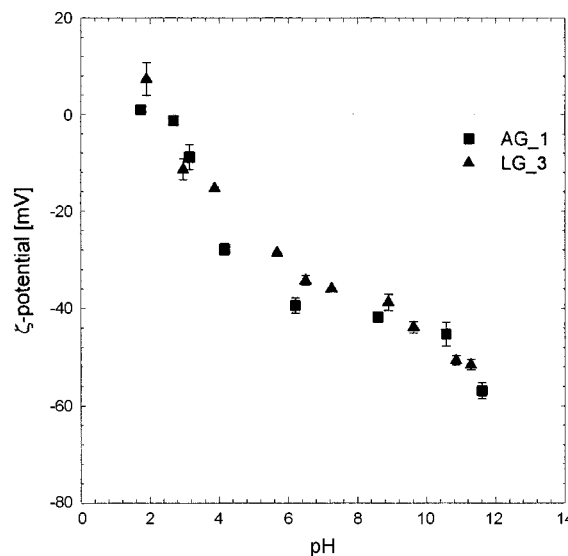


Fig. 5. ζ -potential of silica particles as a function of pH.

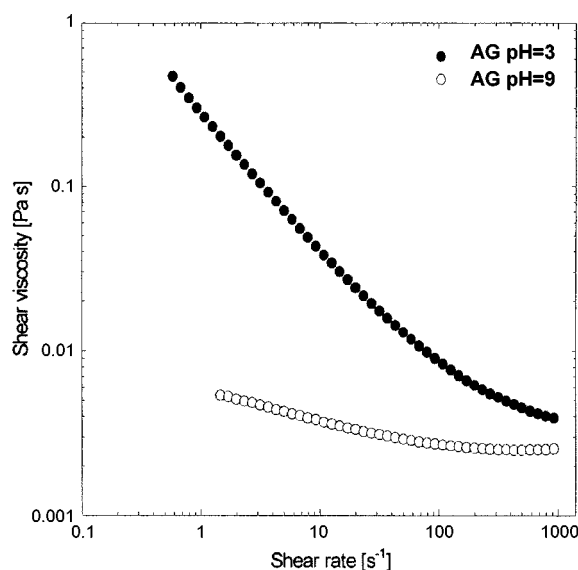


Fig. 6. Shear viscosity as a function of the shear rate for slurry AG-1 at pH 3 and 9.

structure due to weak stabilizing electrostatic repulsion. At pH 9, however, the viscosity was a very weak function of the shear rate and shear thinned slowly and smoothly. In fact, the suspension of 10 wt% behaved like Newtonian liquid. This implies that the silica particles underwent a strong electrostatic repulsion and remained stable under flow. The rheological behavior was clearly coincident with the zeta potential data.

3. Polishing of Silicon Wafer

Polysilicon wafers were polished with the silica slurries of AG-1, LG-3 and their mixtures. All wafers were p(100) type and 4 inches in diameter. Silica slurries for wafer polishing were composed of silica particles, polishing accelerator, dispersion stabilizer and bactericide [Romberger and Payne, 1991; Deal and Werho, 1992]. In this study, MEA and TMAH were used as both polishing accelerator and bactericide. In polishing tests, the aqueous silica slurries of AG-1 and LG-3 containing 200 ppm of TMAH and 0.5 wt% of MEA showed removal rate of $2\text{--}3 \times 10^{-2} \mu\text{m/min}$, which was lower than that of fumed silica slurry with the same accelerator compositions. This is because the untreated bare silica particles were too soft to polish silicon wafer. Therefore, the bare silica slurries were treated hydrothermally in an autoclave at 120 °C for 90 min to increase the hardness of silica particles and modify the surface characteristics of silica abrasives [Seo and Kong, 2000]. In these tests, MEA concentration was fixed at 0.5 wt% and content of TMAH was varied from 100 to 500 ppm. The applied pressure and slurry flow rate were 2.18 kPa and 200 ml/min, respectively.

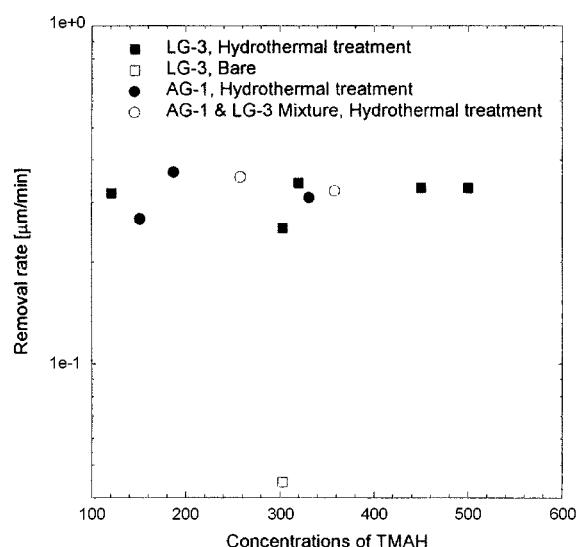


Fig. 7. Removal rates of the polysilicon wafer as a function of the additive concentration. The abrasive was the hydrothermally treated silica.

In Fig. 7, the removal rates of polysilicon wafer surface were plotted for both the hydrothermally treated and untreated (bare) silica slurries. Indeed, the removal rate of the hydrothermally treated silica slurry was about ten times as high as that of the untreated bare silica slurry. This is due to the enhanced hardness of silica abrasives. As mentioned previously, the Stöber silica particles contain micropores and are softer than fumed silica particles. In addition, hydroxyl groups reside on the surface of amorphous silica as a vicinal hydrated form. However, when the slurry was hydrothermally treated at high temperature and pressure, the silica surfaces were dehydrated and the surface hydroxyl groups changed to the vicinal anhydrous form [Iler, 1979]. This modification resulted in enhancement of the hardness of silica particles. It is interesting to note that the removal rate was nearly independent of the TMAH concentration in the range of 100–500 ppm.

Polishing performance of the hydrothermally treated silica slurries of LG-3 and AG-1 was examined with the same polishing accelerator composition of 0.5 wt% MEA and 300 ppm TMAH. The surface morphologies of the polished polysilicon wafer were observed by AFM; the resulting removal rates, root mean square (RMS) roughness and average roughness are given in Table 2. In commercial wafer polishing processes, required removal rate is about 1 $\mu\text{m/min}$ and root mean surface roughness should be below 40 Å. However, polishing parameters such as rotational speeds of platen and head, pressure load of commercial processes are quite different from

Table 2. Removal rate and surface roughness of the polysilicon wafer polished by various silica slurries

Slurry	Removal rate [$\mu\text{m/min}$]	RMS roughness [Å]	Average roughness [Å]
Etched wafer surface	-	18.14	16.42
AG-1	0.3677	3.59	2.93
LG-3	0.3195	1.91	1.46
AG-1/LG-3 mixture (1 : 1 by weight)	0.3567	3.31	2.46
Nalco 2371 (Rodel Co.)	0.3500	3.39	-

those of this study. Therefore, it is not possible to compare our polishing results directly with the commercially required level of polishing performance. Instead, we compared the polishing performance of our silica slurries with those of commercialized wafer polishing slurry (Nalco 2371, Rodel Co.); the result is reproduced in Table 2. As noted, slurry AG-1 showed somewhat higher removal rate and slurry LG-3 displayed better roughness than the commercial slurry. Moreover, mixed AG-1/LG-3 (1 : 1 by weight) slurry showed better removal rate and surface roughness.

The results can be simply expected from the fact that the size distribution of silica particles prepared from fumed silica seeds was broad compared to that from colloidal silica seeds. Moreover, the silica particles from fumed silica were irregular in shape, whereas the silica particles from colloidal silica were nearly spherical. Therefore, the surface of polysilicon wafer polished by slurry LG-3 had lower roughness than it would by polishing with slurry AG-1. Also as noted from Figs. 3 and 4, the silica particles in slurry AG-1 were larger in average than those using slurry LG-3, which was also responsible for the high removal rate of slurry AG-1. The removal rate is usually increased with the average diameter of abrasives in polishing processes. However, the surface roughness is strongly affected by their size distribution, as observed in glass polishing [Cook, 1990].

SUMMARY

The silica particles were prepared from the controlled growth of commercial silica seeds of fumed silica and colloidal silica. These seeds were grown by sol-gel reaction of hydrolysis and condensation reaction induced by intermittent addition of TEOS with a time interval of 12 hr. Pretreatment of the commercial seeds was required for satisfactory dispersion stability. In the present work, milling with zirconia balls and sonication dispersed the silica seeds. Polishing performance was determined by measuring the removal rate, average roughness and RMS roughness of the polished wafer surface. The removal rate of hydrothermally treated silica slurry was ten times higher than that of the untreated slurry. However, the removal rate was nearly independent of MEA and TMAH concentrations in the range of 0.3-0.5 wt% and 100-500 ppm, respectively. In terms of the removal rate, the slurry prepared from fume silica seeds was better than the slurry from colloidal silica seeds. However, the latter showed much better polishing performance considering the polished surface roughness. The average roughness of polysilicon wafer surface was reduced substantially from 0.164 μm to 0.0146 μm by polishing with the slurry from colloidal silica seed.

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