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Facile Synthesis Monodisperse SiO_2 Sphere and Their Application Performances in Chemical Mechanical Polishing

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Abstract: The chemical mechanical polishing (CMP) has been widely used for surface modification of critical materials and components and provided global planarization of topography with a low post-planarization slope high quality and efficiency. The colloidal silica slurries play an important role in the finishing processes of optical components. Monodisperse mesoporous silica nanospheres were synthesized with different surfactant by Stöber method. Their architectural features and texture parameters were characterized by XRD, FTIR, N_2 adsorption–desorption isotherms, SEM, XPS, and TGA techniques. The spherical SiO_2 products presented with the controllable 50-150 nm particle size and distribution. The AFM micrographs reveal that flat and smooth surfaces without distinct scratches residual particles are achieved by using the as-obtained composite particles as abrasives[The particle size of SiO_2 slurry plays a critical role in the chemical-mechanical polishing process.

Keywords: Stöber method ;SiO_2 sphere; chemical mechanical polishing; planarization; SiO_2 slurry

1. Introduction

Chemical mechanical polishing (CMP) is an essential technology to improve the atomic-level planarization of a diverse variety of materials, such as silicon wafers, silicon carbide, sapphire, copper, gallium nitride, and glass[1–3].In a typical CMP process, there are many influencing factors involved in polishing planarization process including pad properties, slurry characteristics and processing conditions. It is well known that chemical and mechanical mechanisms are two important mechanisms in polishing process[4–6].Investigations of the variables in the CMP process , solid loading, particle size and distribution, modulus, modulus, hardness, asperity sizes and distribution, down-pressure, velocity are believed to be responsible for the material removal[7–9].It is essential to develop a novel green slurry to perform CMP for sapphire wafers used for high performance devices, eliminating the environmental contamination and reducing potential harmful impact on operators[10]. The physicochemical properties of abrasives play a critically important role in the performance of silicon wafers[11–13].

Currently, the mechanically active silica (SiO_2), ceria (CeO_2), alumina (Al_2O_3) [14], zirconia (ZrO_2) are widely used in preparation of abrasives[15]. Novel environment-friendly silica slurries have been developed[16]. The silica slurries provide high polishing rate, good planarity, and high selectivity, which have greatest influence and play an important role in the optimization of the CMP process. The regular ball like shapes SiO_2 abrasives have less scratches damage or defects during CMP[17–19].

Substantial research efforts have been made to improve the MRR of The silicon wafers with minimally damaged surfaces during the CMP process using silica abrasives[20–22]. Chen et al. estimated the interaction forces between the substrate surfaces and the abrasive nanoparticles, the ceria particle processed a chemical tooth and formed Si-O-Ce bonds between ceria particles and the

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sample surface[23]. Chen et al. prepared parallel channels hexagonal mesoporous silica (H-mSiO_2) particles, which attached with CeO_2 nanoparticles. The H-mSiO_2-CeO_2 composite particles as abrasives revealed a reduced surface roughness, a low topographical variation, and an improved removal rate. Shi et al. reported on a novel acid based SiO_2 slurry which can simultaneously realize an ultralow Rq of only ~0.193 nm and a high MRR of ~10.9 μm h^{-1} on the FS substrate. Chen et al. used the good uniformity and dispersity 30-140 nm D-mSiO_2 nanospheres as functionalized abrasives[24].The abrasives played a key role and achieve nearly non-damage surfaces with atomic level roughness, which can avoid surface scratches commonly caused by particle agglomerations in slurries[25].

In this work, we successfully synthesized monodispersed SiO_2 nanospheres by Stöber method with the controllable 50-150 nm particle size and distribution. Furthermore, the CMP performance using silica colloid with different sizes have been studied. In addition, the possible CMP mechanism of the developed polishing slurry containing SiO_2 with different sizes abrasives wafers was proposed.

2. Experimental Section

2.1. Materials

Tetraethylorthosilicate (TEOS, A.R.), ammonia aqueous solution (28 wt%), Ethanol (A.R.), Cetyltrimethylammonium chloride(CTAC, A.R.), Cetyltrimethylammonium bromide (CTAB, A.R.) , Octadecytrimethyl ammonium bromide (STAB, A.R.) , Cetyldimethyl benzyl ammonium chloride (HDBAC, A.R.), Triethanolamine (TEA, A.R.), NH_4F,NaOH, Ethanol were purchased from Sinopharm Chemical Reagent Co., Ltd. All chemicals were used as received without further purification. Deionized water was used in all experiments.

2.2. Preparation of silica spheres

Silica spheres were synthesized by the Stöber method[26]. Typically, Solution A: a combination of 100 mg NH_4F (2.7 mmol), 21.7 g H_2O (1.12 mol) and 2.41 mL of a 25% aqueous CTAC solution (1.83 mmol) was stirred at 750 rpm in a 50 mL round bottom flask equipped with a stir bar and heated to 60℃.Solution B:14.35 g TEA (97 mmol) and 2.06 mL TEOS (9.3 mmol) were statically heated in a 20 mL capped polypropylene vial at 90℃ for 30 minutes. Both components remain unmixed after heating. Solution B was then added to solution A at once while stirring vigorously, followed by removal of the oil bath, leaving the reaction solution to slowly cool down to room temperature while continuing to stir overnight. On the next day after 12 hours, 50 mL ethanol was added to the solution, which was then transferred into two 50 mL centrifuge tubes and spun down at a speed of 20000 rpm for 20 minutes at room temperature. The supernatant of the solutions was decanted, and the samples were refilled with 30 mL ethanol each, redispersed mechanically with a spatula and by sonication for 10 minutes, and centrifuged again[27]. The sample was named CTAC-SiO_2.The CTAB-SiO_2, STAB-SiO_2, HDBAC-SiO_2 were synthesized nalogously by replacing CTAC with the equivalent molaramounts of CTAB,STAB, HDBAC ,respectively.

2.4. Polishing tests and evaluations

CMP experiments on silicon wafers were performed on a UNIPOL-300 CMP machine (Shenyang Kejing Instrument Co., Ltd., China) with a Rodel porous polyurethane pad. The force-volume images were recorded with the resolution typically of 10×10 pixels within a scanning area of 3×3 μm^2 area. The 1 wt% solid content SiO_2 abrasive particles were dispersed into deionized water and treated by sonication for 30 min before use. And the slurry pH values were adjusted to 8.5-8.6 using 0.1 M NaOH solution. The polishing parameters were: the feed rate of the polishing liquid was 180 ml/min, the pressure and table-platen speed are fixed at 6 psi and 70 rpm, and the polishing time was 2 h. After polishing, the substrates were cleaned with ultrasonic in DI water. Finally, they were dried with a stream of nitrogen prior to surface analyses.

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2.3. Characterization

Fourier transform infrared spectra (FT-IR) were measured using KBr pellets on a Nicolet iS10 analyzer using a scanned area of 4000-400 cm^{-1} and a 4 cm^{-1} resolution. The samples were treated using the potassium bromide pellet technique before testing.The crystal structures of the samples were characterized by powder X-ray diffraction on a Rigaku SmartLab SE with Cu Ka radiation (λ=1.54056A).The diffraction data were collected over the angle range of 5-80°with a step size of 0.02 at 35 kV and 20 mA. Thermogravimetric Analysis was performed on an in the tempteature range of 30-900°C under nitrogen at a heating rate of 10°Cmin^{-1} with a Netzsch STA 2500 Regulus analyzer. Nitrogen sorption desorption isotherms were obtained at 77 K using a Micromeritics ASAP 2020 sorptionmeter. The surface area based on the N_2 isotherm data was analyzed by BET (Brunauer–Emment–Teller).The elemental composition of the surfaces was determined with a Kratos Analytical Axis Ultra X-ray photoelectron spectrometer equipped with a monochromatic Al Kα source. The structure and morphology of samples were investigated with Zeiss Sigma 300 scanning electron microscopy at an acceleration voltage of 15 kVa. Surface roughness and surface morphology of the polished silicon wafers were characterized by Atomic Force Microscopy with Multimode 8, Bruker, Santa Barbara, CA .

3. Results and Discussion

3.1. Structural and Textural Features

The XRD patterns of the SiO_2 sample were measured and were showed in Figure 1. As can be seen, the strong and broad diffraction peak at 2θ of 23° in good agreement with the positions of pure amorphous silica[28]. This result means that no discernible long-range order in the pore arrangement exists in the SiO_2. These observations illustrated that the silica microsphere were successfully synthesized using the sol-gel method.

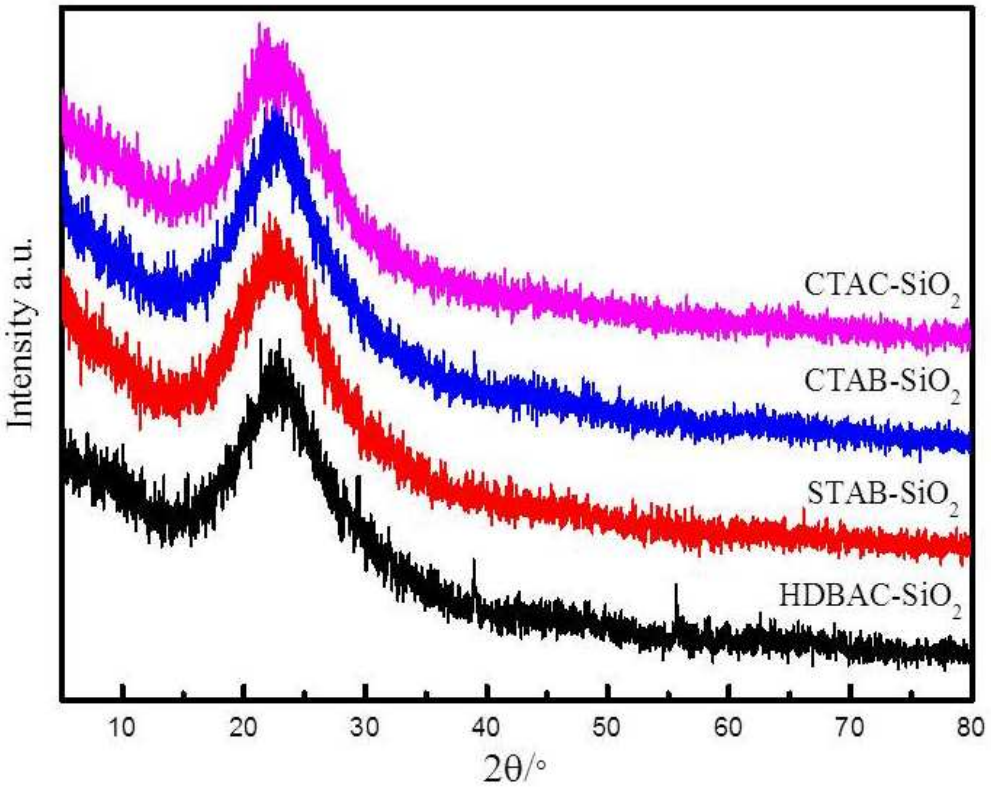

Figure 1. XRD patterns of SiO_2 microsphere.

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The composition of silica spheres was established by FT-IR spectrum as shown in Figure 2. the strong band at 3442.2 cm^{-1} can be ascribed to the absorption of -OH group of silica spheres. The absorption band appears at 2983.39 cm^{-1} indicated that the stretching vibration of $-CH_3$ in the CTAB. The peak at 1630.5 cm^{-1} can be attributed to the bending vibration of H_2O. The peak with a wavenumber of 1404.9 and 1383.8 cm^{-1} belongs to the $-CH_3$ and $-CH_2$ symmetric bending vibrations, respectively. The peak at 970.9 cm^{-1} is associated with the bending vibration of Si-OH.The obvious bands located at 1054.7 and 457 cm^{-1} can be assigned to the stretch vibration bands of Si-O-Si bond,respectively[29]. It indicates that SiO_2 is hydrophilic and there are many hydroxyl groups on the surface of it.

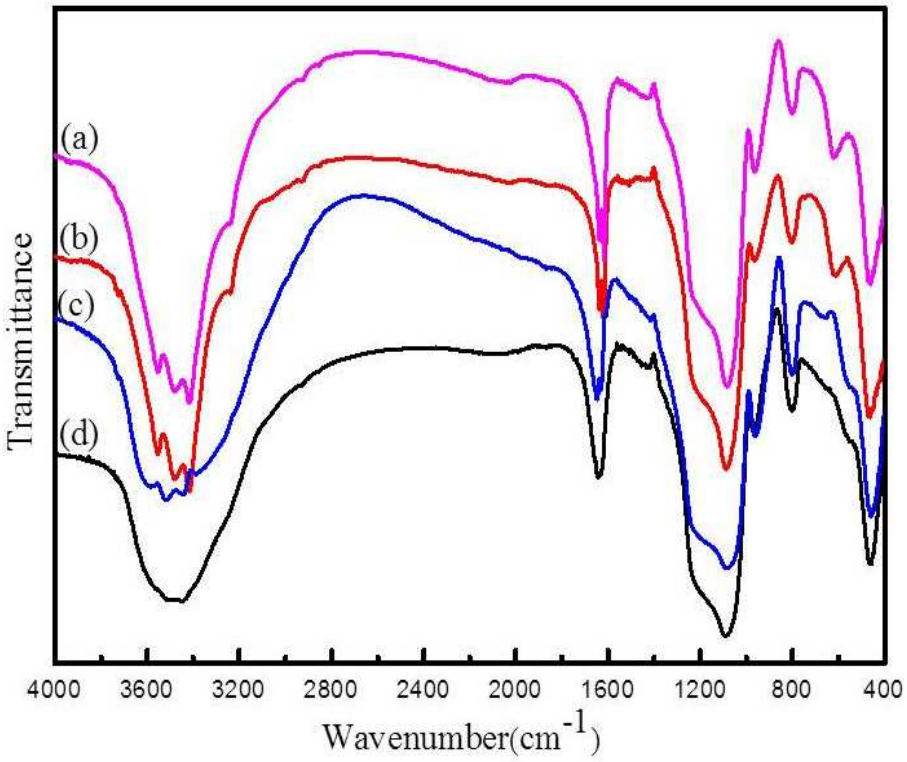

Figure 2. FTIR spectra of SiO_2 microsphere. (a) CTAC-SiO_2, (b) CTAB-SiO_2,(c) STAB-SiO_2,(d) HDBAC-SiO_2.

The TGA curves of SiO_2 from different preparation conditions are given in Figure 3. The first slight weight loss of 8% below 120℃ is attributed to the dissociation of the absorbed water. The second weight loss of 15% between 120℃ to 550℃ can be attributed to the degradation of organic parts [30].

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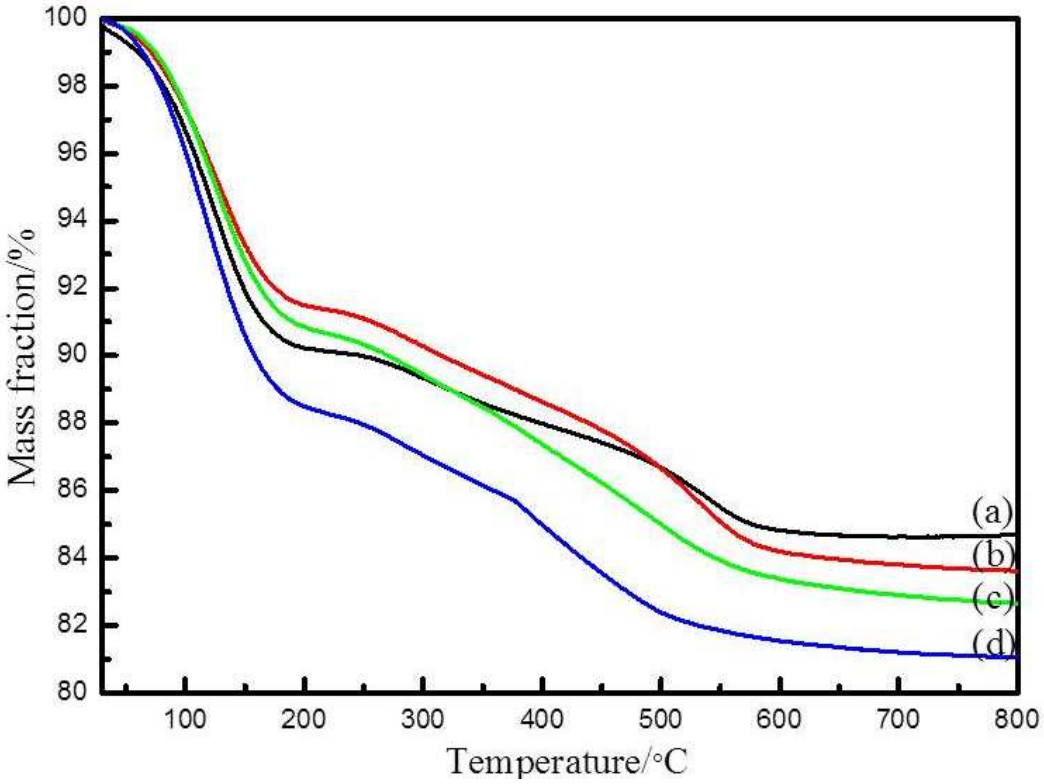

Figure 3. TGA spectra of SiO_2 microsphere. (a) CTAC-SiO_2,(b) CTAB-SiO_2,(c) STAB-SiO_2,(d) HDBAC-SiO_2.

The Brunauer-Emmett-Teller (BET) specific surface area during the synthesis of silica spheres were monitored by the nitrogen adsorption desorption isotherms Figure 4. The adsorption isotherms of silica spheres show a typical type IV adsorption isotherms, which are normally attributed to the characteristics of ordered mesoporous channels. The sorption showing a pore-condensation step around the relative pressure range p/p_0 = 0.3-0.4. Specifically, the BET surface areas of The CTAC-SiO_2,CTAB-SiO_2,STAB-SiO_2,HDBAC-SiO_2 were comparably decreased from 1155.9, 1059.0 and 1119.2 to 796.9 $m^2 g^{-1}$.The pore size of these samples were reduced from 2.4,2.4, and 2.38 to2.35 nm. The pore size did not change significantly.

Table 1. The structure parameters of all related samples.

Samples.	S_{BET}($m^2 g^{-1}$)	Pore size（nm）
CTAC-SiO_2	1155.9	2.40
CTAB-SiO_2	1119.2	2.40
STAB-SiO_2	1059.0	2.38
HDBAC-SiO_2	796.9	2.35

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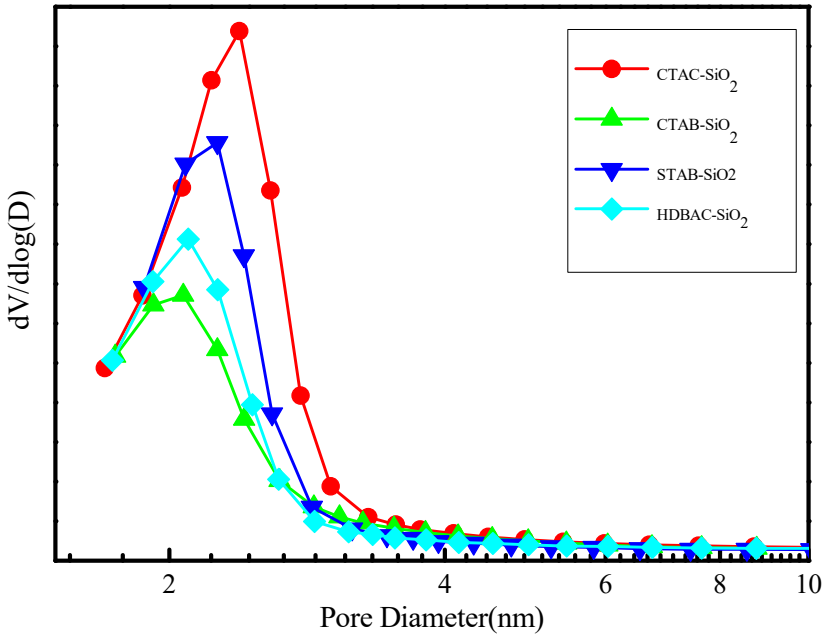

Figure 4. The corresponding pore size distributions.

The particle size and morphology of SiO_2 microspheres were confirmed by SEM, as shown in Figure 5. All samples have a regular spherical shape[31]. The surface of CTAB-SiO_2 and CTAC-SiO_2 are smoother than that of other surfactants-SiO_2. The average particle sizes of SiO_2 microspheres samples are ca. 30 nm-150 nm, respectively.

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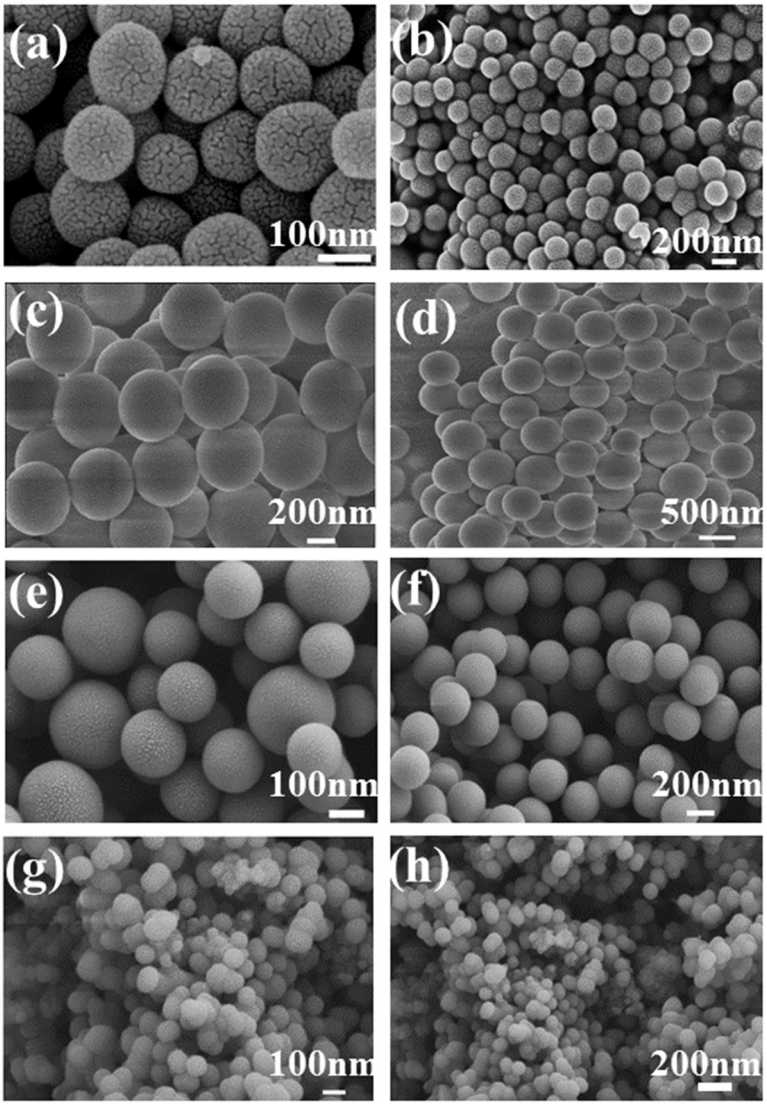

Figure 5. SEM images. (a)(b) CTAC-SiO_2,(c) (d)CTAB-SiO_2,(e) (f) STAB-SiO_2,(g) (h) HDBAC-SiO_2.

The X-ray photoelectron spectroscopy (XPS) is a highly sensitive technique to explore the chemical changes in the element surroundings. The XPS spectra of the elements of Si2p,O1s and C1s are shown in Figure 6. The spectrum of SiO_2 microspheres shows strong peaks at 531.9 eV corresponding to the binding energy of O1s[32,33]. The spectrum at the BE of 533.19 eV and 532.94 eV, corresponding to Si-O. The electronic binding energy of C1s peak at 284.6 eV was corresponded to C–C bonding[34]. The peaks at 283.4 and 286.3 eV corresponded to C-Si and H-C bonding. The peaks of 103.75 and 103.9 eV were assigned to Si2p. The small shoulder peak of 102.9 eV was reported to $H_6C_2Si_2O_3$.Slurry actively reacts with the oxide surface leading dissolution of Si–O bonds to H–C–O–Si bonds[35]. This hydro-carbonated surface of oxide film was easier to be removed by mechanical parts of CMP process.

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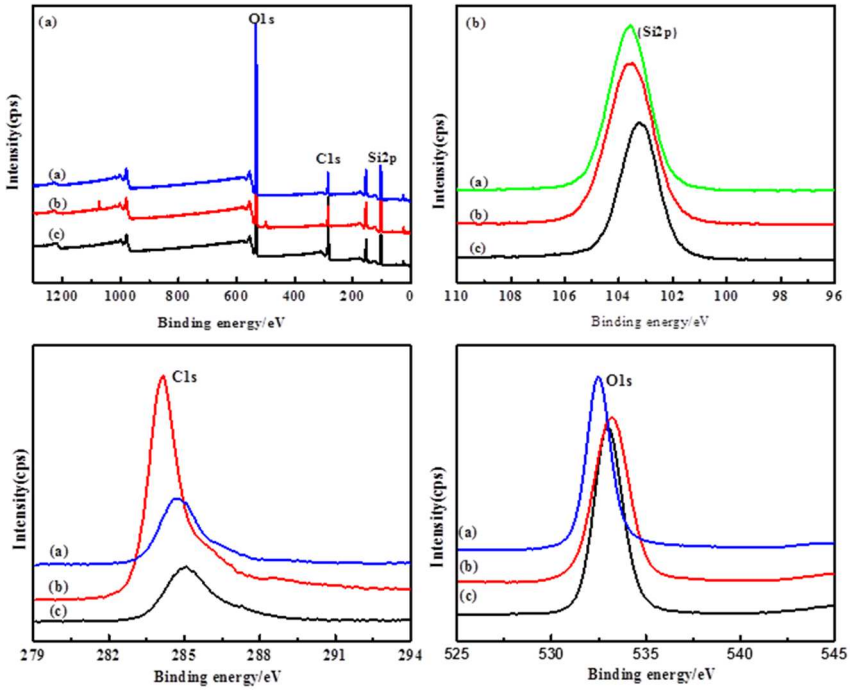

Figure 6. XPS spectra of SiO_2 microsphere. (a) CTAC-SiO_2,(b) CTAB-SiO_2,(c) STAB-SiO_2.

3.2. Polishing performance of SiO_2 microsphere

The surface quality of polished silicon wafers after polishing with CTAB-SiO_2, CTAC-SiO_2, STAB-SiO_2 and HDBAC-SiO_2 particles can be investigated through their surface topographies, which were achieved by AFM optical microscope. As shown in Figure 7, the AFM micrographs reveal that flat and smooth surfaces without distinct scratches residual particles are achieved by using the as-obtained composite particles as abrasives[36,37]. The scratches as well as other microdefects could hardly be observed[38,39]. The silica abrasive particle size plays an essential role during CMP process. It is clearly seen that CTAB-SiO_2 ,CTAC-SiO_2 abrasives have much more regular round shapes and remain independent for each particle[40,41]. There are some mechanical scratches and cracks are distributed deeply on HDBAC-SiO_2 abrasives. The HDBAC-SiO_2 particle size were about 140 nm, which was larger than the other there SiO_2 abrasives[42,43]. The surfactants group may be adsorbed on the surface of SiO_2 abrasive under the action of electrostatic attraction. The Si–OH in the slurry interacts with the silicon wafer surface in a similar fashion to the Si–OH on the SiO_2 grain surface, both in the form of bridge bonds to reduce the breakage bond energy of the Si–Si bonds inside the silicon[44,45].It is noteworthy that the optimal polishing performance can be achieved by controlling the balance between the chemical effect and the mechanical effect[46].

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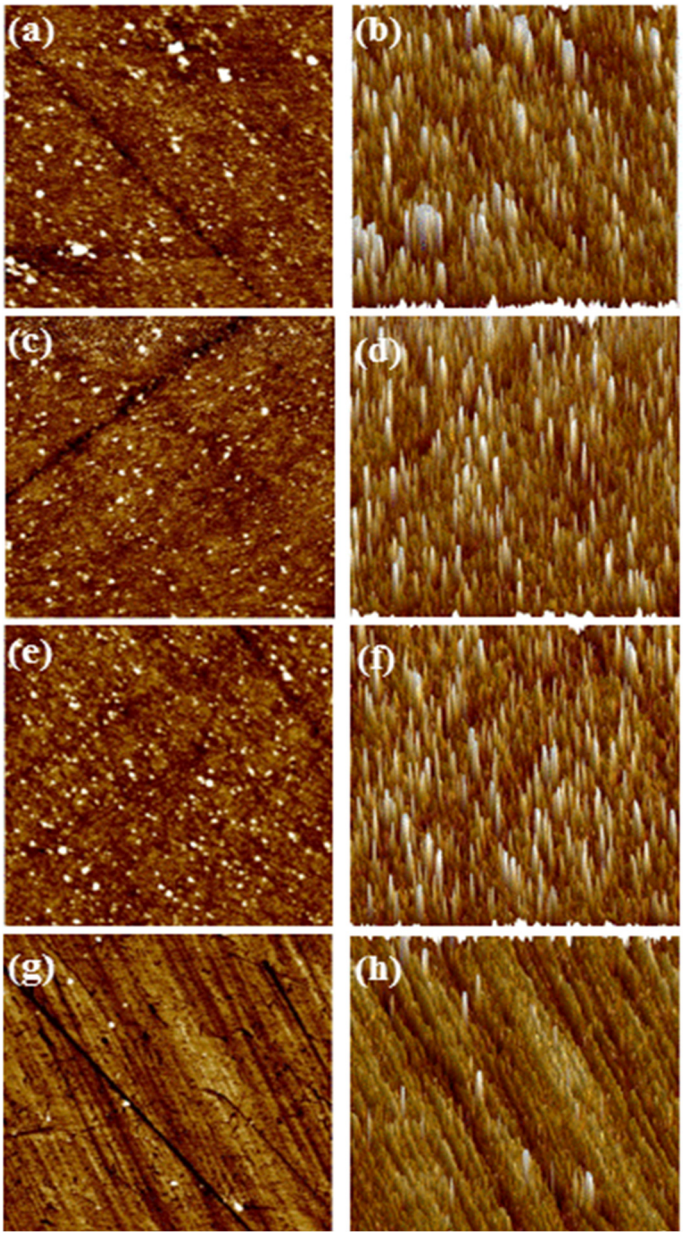

Figure 7. AFM images of the surfaces after CMP with SiO_2 microsphere. (a)(b) CTAC-SiO_2,(c) (d)CTAB-SiO_2,(e) (f) STAB-SiO_2,(g) (h) HDBAC-SiO_2.

4. Conclusions

In summary, the controllable 50-150 nm sizes SiO_2 microsphere were successfully synthesized by the Stöber method with a series of cationic surfactants. The spherical SiO_2 exhibited an improved surface quality in the chemical-mechanical polishing process. The polishing results indicate that the SiO_2 abrasives can achieve a substantial improvement of surface planarization. The novel SiO_2 abrasives were successful applications in CMP.

Author Contributions: J.G. carried the main responsibility for the writing of the manuscript. X.J. supervised the preparation work and contributed to writing the manuscript. L.Z., Y.J.,Q.W. Y.W. assisted J. G. carried out the synthesized and characterized experiment. J. G. was responsible for analyzing the obtained data. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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