

## Polishing of poly(methyl methacrylate), polycarbonate, and SU-8 polymers\*

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Polymers such as poly(methyl methacrylate), polycarbonate, and SU-8 epoxy resin replace silicon as the major substrate in microfluidic system (or BioMEMS) fabrication. Chemical-mechanical polishing is an important technology for many advanced microelectromechanical systems (MEMS) and micro-optoelectromechanical system applications. In this study, the chemical-mechanical polishing of polycarbonate, poly(methyl methacrylate), and SU-8 polymers was investigated. Four types of slurry were tested for chemical-mechanical polishing of polycarbonate and poly(methyl methacrylate). Experiments were then designed and performed to investigate the effects of two key process parameters. Experimental results show that an increase in head load or table speed causes an increase in material removal rates. Within the chosen experimental parameter ranges, the variation of table speed introduced a more significant change in material removal rates than that of head load. An analysis of variance was also carried out, and it was found that the interaction of head load and table speed had a significant effect (95% confidence) on the surface finish of polished poly(methyl methacrylate) samples, while table speed had a significant effect on the surface finish of polished polycarbonate samples. Chemical-mechanical polishing is also a process well suited for polishing SU-8 structures with high aspect ratios. Polished polycarbonate, poly(methyl methacrylate), and SU-8 surfaces had nanometer-order surface roughness, acceptable for most MEMS applications.

Key words: *polymer; MEMS; chemical-mechanical polishing; surface finish; material removal rate*

### 1. Introduction

Although there are issues affecting the use of the technology [1], chemical-mechanical polishing has become a leading planarisation technique in the manufacturing of advanced integrated circuit chips [2]. As semiconductor chips are highly inte-

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grated and multi-layer electro-wired, more precise planarisation of each layer on chips is needed [3]. With decreasing device dimensions, inter-level dielectric planarisation by chemical-mechanical polishing is necessary for technologies beyond the 0.35  $\mu\text{m}$  CMOS generation [4]. Chemical-mechanical polishing has emerged as the preferred manufacturing process for planarising sub-micron multi-level metal layers in integrated circuits [5, 6]. It is also adopted in microelectromechanical system (MEMS) applications [7].

Compared to silicon dioxide, polymers excel due to their relatively low dielectric constants, minimized interconnection delays, and improved conductor packaging densities [5]. A low dielectric constant, gap filling at high aspect ratios, and global planarisation are challenging demands for dielectrics in conventional multilevel metallisation schemes in modern integrated circuit technologies [8]. Novel integrated circuit devices based on low- $k$  material/Cu structures have been proposed recently using the chemical-mechanical polishing technology [9]. Deep scratches at surfaces of low- $k$  organic layers have been observed when the surfaces are polished mechanically, and thus reduction of scratches is required in advanced chemical-mechanical polishing processes [10].

Silicon [11] and glass have traditionally been the two major materials used in MEMS. Many biomedical devices, however, require polymers such as polycarbonate and polyimide, which provide a more suitable interface with biological tissue [12]. Imprinting techniques can be used to fabricate microfluidic devices on poly(methyl methacrylate) substrates [13]. In microfluidic applications, important material properties include machinability, surface charge, molecular adsorption, electroosmotic flow mobility, and optical properties [14].

One polishing condition of chemical-mechanical polishing can make a large difference in the material removal rates of different processes [15]. Chemical-mechanical polishing may also introduce surface defects and thus increase the demand for automatic defect-detection techniques [16]. It is extremely difficult to analyse its polishing mechanisms [17, 18]. Therefore, a large number of experiments are required for each set of process conditions [19].

Conventionally, poly(methyl methacrylate) resists are patterned by electron beam lithography, followed by appropriate pattern transfer techniques. The sensitivity of poly(methyl methacrylate) resists is low, however, limiting exposure speed and throughput. To extend high resolution writing ability, other types of resists with higher electron beam sensitivity have been explored [20]. SU-8 is used mostly for high aspect ratio MEMS applications on thick resist layers using ultraviolet or X-ray illumination [21].

SU-8 is a high contrast, epoxy-based photoresist for micromachining applications, where a thick, chemically, and thermally stable image is desired [22]. By using a faster drying and more polar solvent systems, improved coating properties and higher throughput are realized. The exposed and subsequently cross-linked portions of the film are rendered insoluble to liquid developers [23]. SU-8 is over two orders of magnitude more sensitive to X-ray radiation than poly(methyl methacrylate), and the

accuracy of the SU-8 microstructures fabricated by deep X-ray lithography is superior to ultraviolet lithography and comparable to poly(methyl methacrylate) structures [24]. The good pattern quality together with high sensitivity offers rapid prototyping and direct lithographic capability. High sensitivity, fairly good adhesion properties, and relatively simple processing of SU-8 make it a good substitution for novolac-based chemically amplified negative e-beam resists in optical mask manufacturing [25].

Chemical-mechanical polishing of polycarbonate, poly(methyl methacrylate), and SU-8 polymers was investigated in this study. Four types of slurry were tested. Chemical-mechanical polishing experiments were then designed and performed to investigate the effects of key process parameters on material removal rates and the surface finish of polycarbonate and poly(methyl methacrylate) substrates. Patterned and non-patterned SU-8 samples were prepared using the negative photolithography process, and the chemical-mechanical polishing of SU-8 was also investigated.

## 2. Chemical-mechanical polishing experiments

Poly(methyl methacrylate) and polycarbonate substrates were cut into 2 mm thick round plates with the diameter of 150 mm. Chemical-mechanical polishing experiments were carried out with an Okamoto SPP-600S polishing machine. A SUBA 800 polishing pad was used for the experiments. Four types of slurry, such as ILD1200, Simlox, Mazin SRS1, and SRS3, were evaluated for polishing poly(methyl methacrylate) and polycarbonate. ILD1200 is fumed silica polishing slurry containing ammonium hydroxide, designed for oxide chemical-mechanical polishing. Mazin SRS1 and SRS3 are colloidal silica polishing slurries for stock removal applications. Simlox is a type of slurry specially designed for removing polymers. Throughout the chemical-mechanical polishing experiments, the slurry flow rate was set to 100 cm<sup>3</sup>/min, the spindle rotation speed was kept at 40 rpm, and the oscillation speed – 2 mm/sec. Two key process parameters, head load and table speed, were varied to examine their effects on the quality and efficiency of polishing poly(methyl methacrylate) and polycarbonate.

Before and after polishing, the thicknesses of the polymer workpieces were measured using a head thickness gauge to compute the material removal rate, which was defined as thickness reduction per minute in this study. After polishing, the surface roughness of workpieces was measured using a profilometer. The average roughness height  $R_a$ , one of the most commonly used roughness parameters, was used to represent the surface roughness.

The SU-8 material was spin-coated on silicon wafers under proper conditions to produce low defect coatings. The thickness of the resist was approximately 25  $\mu$ m after the spin-coating process. Soft-baking was carried out on hot plates with two different temperatures to remove the solvents from the photoresist coating, which became photosensitive after soft-baking. The mask was aligned with the wafer so that the pattern could be transferred onto the wafer surface. The photoresist was exposed

through the pattern on the mask with high-intensity ultraviolet light. The wafer was then exposed for 30 sec. Post-expose baking was carried out on a hot plate to cross-link the exposed portions of the film. The parameters used to polish SU-8 were: table speed – 30 rpm, head load – 100 g/cm<sup>2</sup>, slurry flow rate – 100 cm<sup>3</sup>/min, spindle rotation speed – 50 rpm, and oscillation speed – 2 mm/sec. Mazin SRS3 was used as the slurry for polishing SU-8.

A scanning electron microscope and an atomic force microscope were used to analyse the polished surfaces. Hardness tests were performed on a Vickers hardness tester to measure and compare the hardness of examined materials.

### 3. Experimental results

Figure 1 shows the results of hardness tests of the polymers. The SU-8 resist is more than twice harder than the poly(methyl methacrylate) and polycarbonate materials. Hardness is the property of being rigid and resistant to pressure. With higher hardness, SU-8 is also more resistant to be scratched than poly(methyl methacrylate) or polycarbonate.

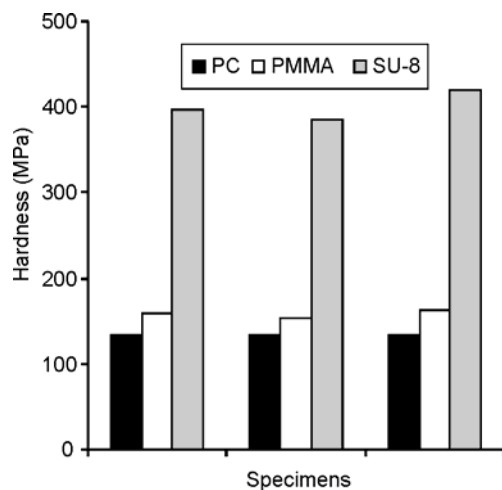


Fig. 1. Hardness of poly(methyl methacrylate) (PMMA), polycarbonate (PC), and SU-8

Since poly(methyl methacrylate) samples are harder than polycarbonate, after chemical-mechanical polishing they have shallower scratches and thus better surface finish than polished polycarbonate samples.

As shown in Fig. 2, the smoothest poly(methyl methacrylate) and polycarbonate surfaces were obtained by chemical-mechanical polishing using Simlox, which also produced relatively high material removal rates. Therefore, it was selected to be the slurry for polishing poly(methyl methacrylate) and polycarbonate, and was used to evaluate the effects of key process parameters.

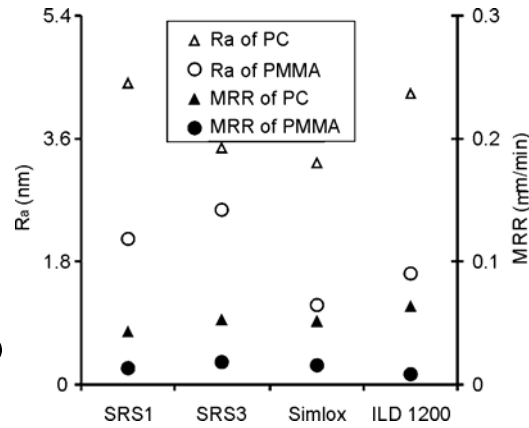


Fig. 2. Surface roughness  $R_a$  and material removal rate (MRR) of poly(methyl methacrylate) (PMMA) and polycarbonate (PC) polished using four types of slurry

Two process parameters, head load and table speed, were investigated for polishing poly(methyl methacrylate) and polycarbonate substrates, while the other conditions were fixed. Three levels (low, median, and high) for each parameter were assigned: 20, 30, and 40 rpm for table speed, and 75, 100, and 125  $\text{g}/\text{cm}^2$  for head load, in order to limit the number of experiments. 100  $\text{g}/\text{cm}^2$  head load and 30 rpm table speed are polishing conditions commonly used with the Okamoto SPP-600S chemical-mechanical polishing machine for typical applications. 20 and 40 rpm for table speed, and 75 and 125  $\text{g}/\text{cm}^2$  for head load were additionally chosen being low and high factor levels, respectively, in order to investigate the effects of the two key process parameters.

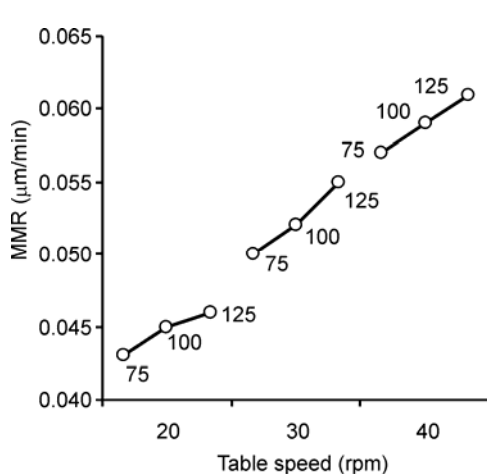


Fig. 3. Effects of head load and table speed (in  $\text{g}/\text{cm}^2$ ) on the material removal rates (MRRs) of polycarbonate

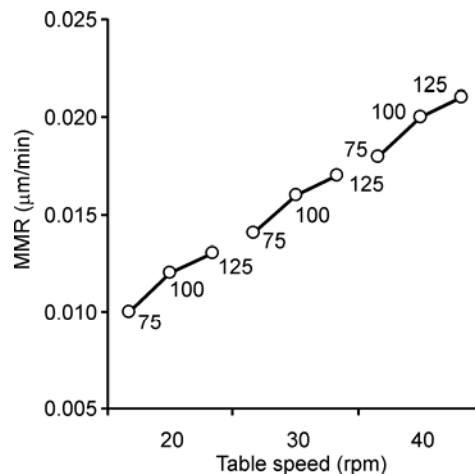


Fig. 4. Effects of head load and table speed (in  $\text{g}/\text{cm}^2$ ) on the material removal rates (MRRs) of poly(methyl methacrylate)

Figures 3 and 4 show the effects of head load and table speed on the material removal rate in polishing polycarbonate and poly(methyl methacrylate), respectively.

Material removal rates increase for increased head load and table speed. These results approximately agree with the Preston equation [1], which theoretically expresses that the material removal rate is proportional to the pressure applied to the workpiece. Another trend revealed from Figs. 3 and 4 was that within the chosen experimental parameter ranges, the variation of table speed introduced a more significant change in material removal rate than that of head load. Since polycarbonate is softer than poly(methyl methacrylate), its material removal rate is higher than that of poly(methyl methacrylate).

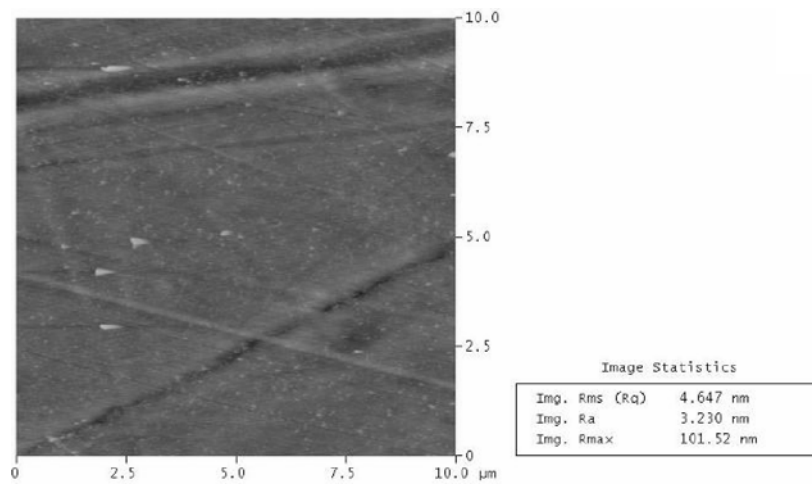


Fig. 5. Atomic force microscope image of polycarbonate polished with a table speed of 30 rpm and head load of 100 g/cm<sup>2</sup>

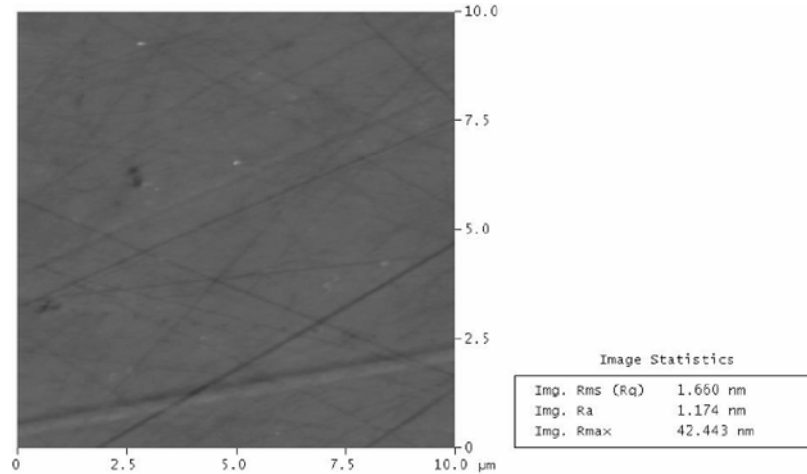


Fig. 6. Atomic force microscope image of poly(methyl methacrylate) polished with a table speed of 30 rpm and head load of 100 g/cm<sup>2</sup>

Figures 5 and 6 show examples of atomic force microscope images of polished polycarbonate and poly(methyl methacrylate), respectively. In order to draw any conclusions whether there were any significant effects of head load and table speed on the surface roughness of polished poly(methyl methacrylate) and polycarbonate, an analysis of the variance (ANOVA) of two-factor factorial experiments was carried out using the measured surface roughness values. Tables 1 and 2 are the ANOVA tables for polished polycarbonate and poly(methyl methacrylate), respectively.

Table 1. ANOVA table for polished polycarbonate

Source of variation	Sum of squares (nm <sup>2</sup> )	Degrees of freedom	Mean square (nm <sup>2</sup> )	F value
Factor A: head load	2.35	2	1.18	0.87
Factor B: table speed	22.79	2	11.40	8.44
Interaction of A and B	10.23	4	2.56	1.90
Error	12.18	9	1.35	
Total	47.55	17		

Table 2. ANOVA table for polished poly(methyl methacrylate)

Source of variation	Sum of squares (nm <sup>2</sup> )	Degrees of freedom	Mean square (nm <sup>2</sup> )	F value
Factor A: head load	0.68	2	0.34	0.47
Factor B: table speed	0.50	2	0.25	0.35
Interaction of A and B	12.53	4	3.13	4.35
Error	6.47	9	0.72	
Total	20.18	17		

Table 3. Effects of the factors (critical  $F$  values:  $F_{0.05,2,9} = 4.26$  and  $F_{0.05,4,9} = 3.63$ , for 95% confidence or risk  $\alpha = 0.05$ )

Factor	Polished material	
	Polycarbonate	Poly(methyl methacrylate)
Head load	No significant effect ( $F$ value $0.87 < F_{0.05,2,9}$ )	No significant effect ( $F$ value $0.47 < F_{0.05,2,9}$ )
Table speed	Significant effect ( $F$ value $8.44 > F_{0.05,2,9}$ )	No significant effect ( $F$ value $0.35 < F_{0.05,2,9}$ )
Interaction	No significant effect ( $F$ value $1.90 < F_{0.05,4,9}$ )	Significant effect ( $F$ value $4.35 > F_{0.05,4,9}$ )

The summary of the ANOVA results is shown in Table 3, in which  $F_{0.05,2,9} = 4.26$  and  $F_{0.05,4,9} = 3.63$  are the critical  $F$  values for 95% confidence (risk  $\alpha = 0.05$ ). Only the interaction of head load and table speed had a significant effect, but individually

head load and table speed had no significant effects on the surface finish of polished poly(methyl methacrylate) samples. Further analysis of the roughness data obtained from atomic force microscope measurements revealed that to obtain smooth poly(methyl methacrylate) surfaces, the table speed of 40 rpm and head load of  $75 \text{ g/cm}^2$  should be used. On the other hand, only table speed had a significant effect, but individually head load and the interaction of head load and table speed had no significant effects on surface finish of polished polycarbonate samples. Further analysis of the roughness data obtained from atomic force microscope measurements revealed that to obtain smooth polycarbonate surfaces, the table speed of 20 rpm should be used.

The patterns of the SU-8 resist before and after chemical-mechanical polishing were studied using a scanning electron microscope. To get a better and more accurate view of the profile of the patterned SU-8, the wafer was cross-sectioned and examined under a microscope.

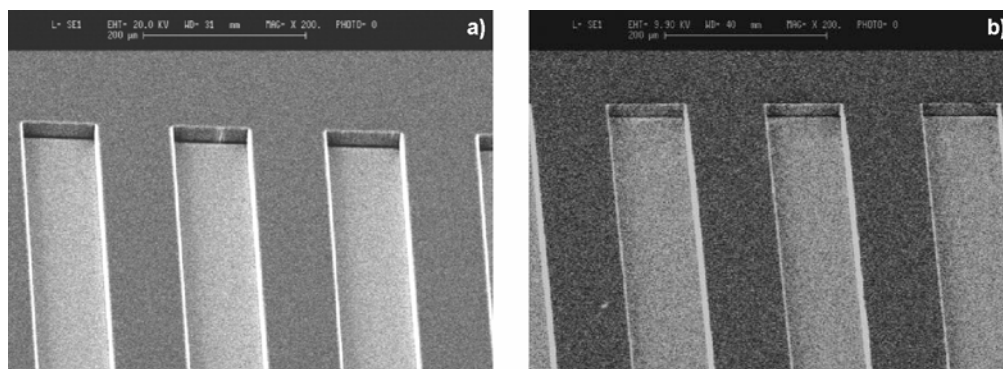


Fig. 7. 100–100  $\mu\text{m}$  channel pattern before (a) and after (b) chemical-mechanical polishing

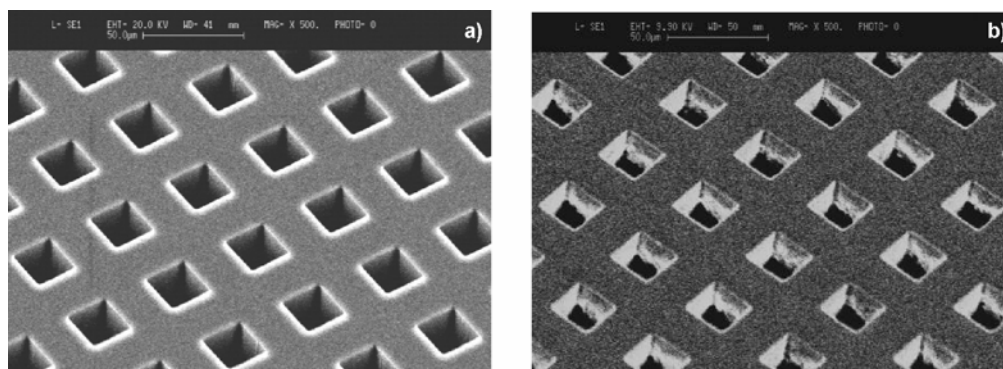


Fig. 8. 25–25  $\mu\text{m}$  pattern before (a) and after (b) chemical-mechanical polishing

Figures 7 and 8 show examples of scanning electron microscope images of SU-8 patterns before and after chemical-mechanical polishing. The shapes of the patterns



were retained after the chemical-mechanical polishing process. Figure 9 shows the cross-section of the patterned SU-8 before and after chemical-mechanical polishing.

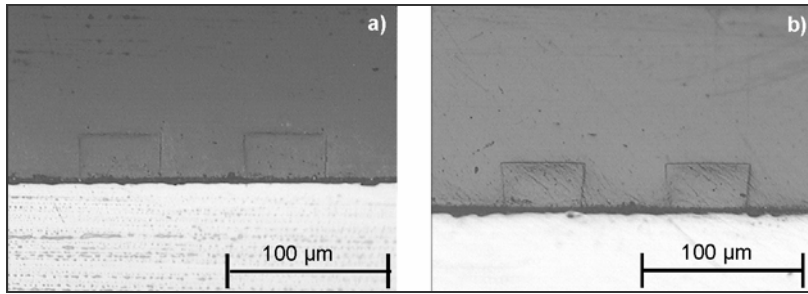


Fig. 9. Cross-section of 50-50- $\mu\text{m}$  channel pattern before (a) and after (b) chemical-mechanical polishing

The shapes and edges of the patterns were the same before and after the polishing. There was little or no sign of distortions or deformations in the patterns of the SU-8 resist. This shows that the chemical-mechanical polishing is well suited for planarising SU-8.

#### 4. Conclusions

Four types of slurry were tested for chemical-mechanical polishing of polycarbonate and poly(methyl methacrylate) substrates. Experiments were designed and performed, and ANOVA was also carried out to investigate the effects of two key process parameters. The following conclusions could be drawn:

- The smoothest poly(methyl methacrylate) and polycarbonate surfaces were obtained by polishing using Simlox slurry, designed for the removal of polymers, which also produced relatively high material removal rates.
- An increase in head load or table speed causes an increase in material removal rates. Within the chosen experimental parameter ranges, the variation of table speed introduced a more significant change in material removal rates than head load.
- The interaction of head load and table speed had a significant effect on the surface finish of polished poly(methyl methacrylate) samples, while table speed had a significant effect on the surface finish of polished polycarbonate samples. To obtain smooth poly(methyl methacrylate) surfaces, the table speed of 40 rpm and head load of 75 g/cm<sup>2</sup> are recommended. To obtain smooth polycarbonate surfaces, a table speed of 20 rpm is recommended.
- Chemical-mechanical polishing is a process well suited for polishing SU-8 structures. The parameters used to polish SU-8 were: table speed – 30 rpm, head load – 100 g/cm<sup>2</sup>, slurry flow rate – 100 cm<sup>3</sup>/min, spindle rotation speed – 50 rpm, and oscillation speed – 2 mm/sec. Mazin SRS3 colloidal silica polishing slurry for stock

removal applications was used, because the hardness of SU-8 is more than twice that of poly(methyl methacrylate) or polycarbonate.

- All polished polycarbonate, poly(methyl methacrylate), and SU-8 surfaces had nanometer-order surface roughness heights, acceptable for most MEMS applications.

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