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OPTIMIZATION AND CHARACTERIZATION OF ELECTRON BEAM RESIST USING ATOMIC FORCE MICROSCOPY

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ABSTRAK

Resis negatif ma-N 2403 dan 495 K PMMA memiliki resolusi yang baik untuk aplikasi litografi berkas elektron (EBL). Ketebalan resist optimal memainkan peran penting dalam paparan berkas elektron. Oleh karena itu, dalam penelitian ini, ketebalan dari kedua resist yang dioptimalkan menggunakan spincoater dalam jangkauan laju spin 1000-6000 rpm. Semakin laju spin meningkat, ketebalan resist menurun juga. Morfologi permukaan resist dikarakterisasi dengan mikroskop gaya atom. Butir butir resist nampak panjang. Dalam analisis AFM, permukaan profil resist negatif ma-N 2403 dan 495 K PMMA nampak seperti kerucut.

ABSTRACT

Negative resist ma-N 2403 and 495 K PMMA have good resolution for electron beam lithography (EBL) application. The optimum resist thickness plays significant role in e-beam exposure. Therefore, in this research, thicknesses of both resists were optimized using spincoater within spin speeds of 1000-6000 rpm. As spin speed increased, resist thickness decreased as well. Morphology of resist surfaces were characterized using atomic force microscopy (AFM). Grains of resist show long grains. In AFM analyses, surface profiles of negative resist ma-N 2403 and 495 K PMMA show cone peaks.

Keywords: e-beam resist; spincoater; e-beam lithography

INTRODUCTION

E-beam resists consist of two types, namely positive and negative e-beam resists. The parameters of resist spincoating for the use of EBL are resolution, sensitivity (Raptis *et al*., 2000), contrast (Koleva & Mladenov, 2005; Yashin *et al*., 2002), roughness (Constantoudis *et al*., 2002), contamination, resist thickness, and thickness uniformity. To fabricate quantum dot single electron transistor (QD SET), the use of high resolution resist is a must. In this fabrication, negative resist ma-N 2403 and 495 K PMMA were selected because of their high resolutions. In this research, resist thicknesses of both resists were optimized and their profiles were characterized using AFM.

The resolution of EBL in standard resists is limited by the size of the molecules of the resist and the density of the pattern strongly depends on the concentration of the developer. The e-beam resist resolutions consist of intrinsic resolution and practical resolution (Yang *et al*., 2006). High resolution e-beam resists therefore should not only show a specific sensitivity to electrons but also be thin and composed of small subunits (Arshak *et al*., 2003).

The fabrication of dense arrays introduces another problem at very small dimensions, which is the mechanical stability of the small resist features after development (Vieu *et al.,* 2000). The distance between patterns was necessary optimized, because the removal of resist depends on proximity effects, exposed area and distance between patterns.

Positive e-beam resists are resists prepared for EBL application which are made based on chemical reactions due to polymer-chain scissions, which allow the e-beam-exposed regions to be developed by washing with a solvent (Sutikno et al., 2006; Asakura et al., 2006). After development, the exposed area removes and inversely the un-exposed area remains stay.

The resolution and density of PMMA are of 10 nm (Nastaushev *et al*., 2002) and 700 Gbit/in^2 (100 Gbit/cm^2) respectively. Typical thicknesses of PMMA range from 0.4 to 1.0 m (Walsh & Franses, 2003). Well known resolution of PMMA resist of 10 nm on bulk silicon substrate is in good agreement PMMA has long been used as a positive resist for high-resolution EBL. The positive nature of this resist occurs due to polymer chain scissions which are initiated by e-beam. This involves the removal of a carbonyl group from the side of the main chain which then results in main chain scissions. These chain scissions reduce the chain length of the polymers in the exposed area making it more soluble in an appropriate solvent. A parallel process of polymerization also occurs in which the polymer units are crosslinked together to form a hard insoluble material. At lower doses the scissions process dominates, allowing PMMA to be used as a positive resist (Hoole *et al*., 1997).

PMMA is a versatile polymeric material that has been exploited for a variety of imaging and non-imaging micro-electronic applications, including the use of a radiation sensitive patterning layer, protective or structural layer, a protective coating for wafer thinning,

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bonding adhesive and a sacrificial layer (Bajuri *et al*, 2005). Nano PMMA and copolymer resists are compatible with glass, ceramic, unfilled polyethylene, high-density polyethylene, polytetrafluoroethylene, stainless steel, and equivalent materials.

Negative e-beam resists are resists prepared for EBL application which are made based on chemical reactions due to polymerization or cross-linking, followed by removal of the unexposed, uncrosslinked regions of resist (Sutikno *et al*., 2006; Asakura *et al*., 2006). After development, the exposed area remains stay. PMMA which is commonly used as positive resist can be used in a negative manner with exposure at higher dose levels (Hoole *et al*., 1997) and by controlling the localized amount of the electron dose in EBL (Asakura *et al*., 2006).

The thickness of resist is important parameter to get the best resolution in lithography. Since the thicker resist causes resolution problems due to scattering of beam energy, the thinner layer is preferable. Thin resist layers alleviate some high-energy imaging problems and produce better e-beam resolution. Resist thickness is influenced by many factors, such as solid content and viscosity of the resist, but the primary determination of thickness is the rotational spinner (Bajuri et al., 2005). In addition to controlling thickness, it is important to control thickness uniformity, surface smoothness, and overall film quality (Walsh & Franses, 2003).

METHOD AND EXPERIMENT

To optimize resist thickness, Si substrate was spincoated using either negative resist or positive resist. In the step of e-beam resist optimization, the use of Si substrate is cheaper. The samples were prepared by cutting the Si wafer each in the dimension of 10 mm x 10 mm samples as shown in Fig. 4.2. The sample cutting on the upper right side of sample was aimed to give reference point in adjusting positions whether adjusting sample position in the stage, exposure positions or looking for the exposed position for imaging.

In this research, two cleaning techniques of substrates, using piranha and cleaning using RCA-1 and RCA-2, are involved. The results are compared. These cleanings processes are aimed to remove either inorganic or organic contaminants. In addition, piranha is composed of H_2SO_4 and DI-water in ratio of 3:1. To remove organic contaminants, ten pieces of samples are dipped in piranha at 90 ºC for 30 s and then dried by spinning. The substrates could be also cleaned by making use of standard cleaning as the following sequence of steps: substrates are firstly dipped in RCA-1 solution at 75 ºC for 10 min, then in DI-water, and finally in BOE for 10-15 s. The substrates are then rinsed in DI-water, dipped again but now in RCA 2 at 85 ºC for 10-15 min, and rinsed in DI-water. Afterall the substrates are spun. The heating of RCA-1 and RCA-2 are supplied by corning hot plate. To increase the rate of contaminants removal, the substrates is agitated by hand (Hashim *et al*., 2007). The substrate cleaning using piranha is simpler and it produces surface which is cleaner than that of standard cleaning 1 (using RCA-1 & RCA-2).

The dried substrates are heated up at 200 ºC for 30 min and then cooled down to room temperature. Subsequently, the substrates are spincoated using negative resist ma-N 2403 as the following steps: ramped up 500 rpm for 6 s, spun at speed 1000 to 6000 rpm for 30 s and ramped down to 0 rpm for 5 s. Similarly, positive resist 495 PMMA A4 resist 2% in anisole is spincoated at spin speed of 1000-6000 rpm for 30 s. Negative resist ma-N 2403 layer was pre-baked at 90 ºC for 2 min and PMMA resist layer was pre-baked at 200 ºC for 2 min. A spin speed variety was created to determine the correlation between spin speeds with resist thicknesses and to obtain the optimum resist thickness which is applicable for e-beam patterning. The resist thickness was measured using spectrophotometer (Filmetrics, F20 thin film analyzer) as shown in Fig. 4.7 (Hashim *et al.*, 2007).

RESULTS AND DISCUSSION

The resulted resist thickness from 1000 rpm spin speed is non uniform and found coagulation on the substrates edges. The resist thickness start to be uniform is on the 2000 rpm spin speed. The resulted uniformity depends on cleaning process, where the low contaminant wafer produced uniform resist thickness.

Some criteria for determining the optimum resist thickness which will be applied in e-beam exposure are as the followings: (1) high reproducibility (repeat-ability) of resulted resist thicknesses, (2) clear and light resist surface, and (3) no voids found which is effected by pre-bake and high surface uniformity. The resist thicknesses resulted at 3000 rpm spin speed have light yellow resist surface colors and if the substrates are pre-baked at 90 ºC for 90 seconds resist surface colors is not changed and no voids found on the surface. On the other hand, resist thicknesses resulted at less than 3000 rpm spin speeds have red color and the colors of resist thicknesses spincoated at more than 3000 rpm spin speed are as the following: yellow (4000 rpm), green (5000 rpm) and dark green (6000 rpm). In common, resist thicknesses in the edges of substrates were thicker than those of the center of substrates. The resist thicknesses resulted at less than 3000 rpm spin speeds are very non uniform, because resist coagulated. As the spin speeds increase, from 6000 rpm to higher, the resist Ithicknesses don't significantly decrease. The correspondence between the average resist thicknesses of ma-N 2403 to spin speeds are shown in Fig.1 thicknesses don't significantly decrease. The correspondence between the average resist thicknesses of ma-N 2403 to spin speeds are shown in Fig.1

To optimize resist thickness of 495K MW PMMA with 2% anisole, a spin speed is variated from 1000 to 6000 rpm and the resist thickness is measured. The resist thickness on each sample is measured at three points and then its average value is calculated. The correlation between resist average thicknesses with spin speed was graphically is shown in Fig. 2. As spin speed increased, the resist thickness decreases.

In this research, it is found that different resist shows different surface profile. As shown in Figs. 3 and 7, surface profile of negative resist ma-N 2403 is different compared with surface profile of PMMA. In addition, at the same spin speed, PMMA has smoother and more contrast surface. An interesting phenomenon occurs at the spin speed of 3000 rpm where negative resist ma-N

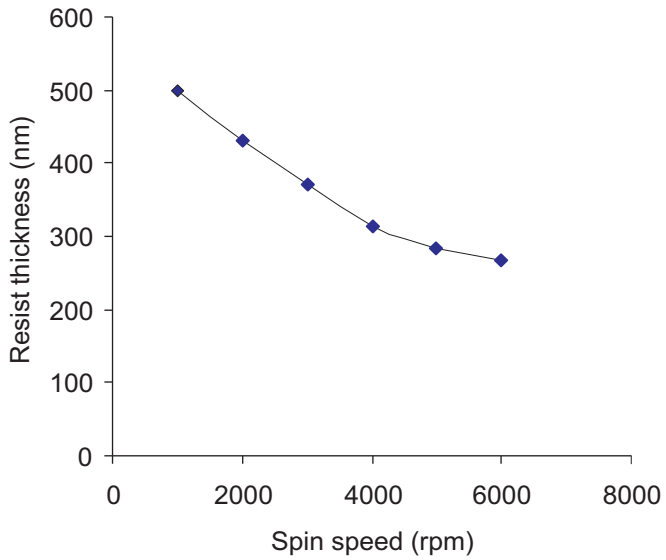

Figure 1. Resist thickness of ma-N 2403 within spin speeds range of 1000-6000 rpm (Sutikno *et al.,* 2006).

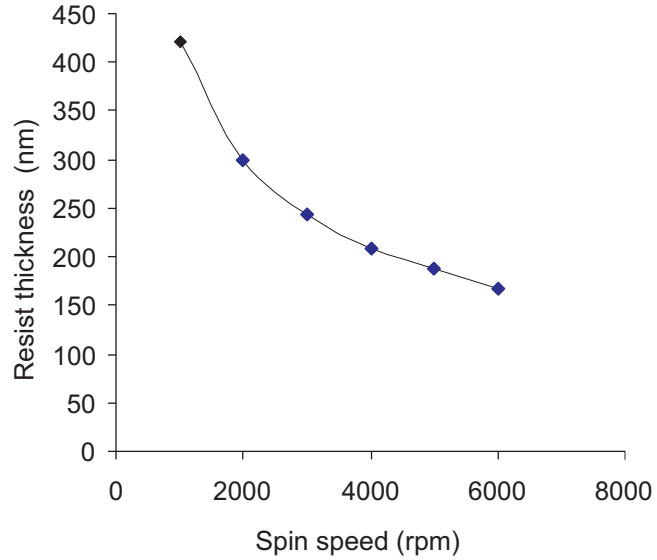

Figure 2. Resist thickness of 2% 495K MW PMMA with anisole within the spin speeds range of 1000 to 6000 rpm (Sutikno *et al*., 2006).

2403 and PMMA have reflection points namely the bend points of the decrease and the increase of RMS (root mean square) roughness. At spin speeds as high as 1000-2000 rpm, RMS roughness of negative resist ma-N 2403 are higher than those of PMMA, this can be explained as the following: viscosity of negative resist ma-N 2403 is higher so that it coagulates faster and resist thickness becomes uniform.

Profiles of negative resist ma-N 2403 show cone peaks. As spin speed in-creases, cone peaks sizes become smaller and the distances between cone peaks become smaller. As shown in Fig. 3(d), some big cone peaks are produced due to the unexpected contaminant particles. Height of cone peaks ranges 0.67-6.99 nm and there is found an outstanding peak as high as 19.71 nm in Fig. 3(d).

In general, as spin speed increases, the RMS roughness of negative ma-N 2403 decreases. For the nearest spin speed, for example 5000 rpm and 6000 rpm, the obtained RMS roughness sometime exchanges each other. This result is influenced not only by resist properties but also by the stability of spinner. Nevertheless, this problem does not so disturb in e-beam exposure, because in principle, all flat resist surfaces can be exposed anddeveloped. At spin speeds as high as 5000-6000 rpm, RMS roughness seem no significantly decreasing.

The grain form of negative resist ma-N 2403 is long grain. The long grains inform the characteristic of polymer materials which is the main component of resist. As spin speed increases, the mean diameter of grains decreases, the amount of grains increases and the distribution of grains becomes more uniform.

Fig. 7(a) is captured of the flat resist surface. It is

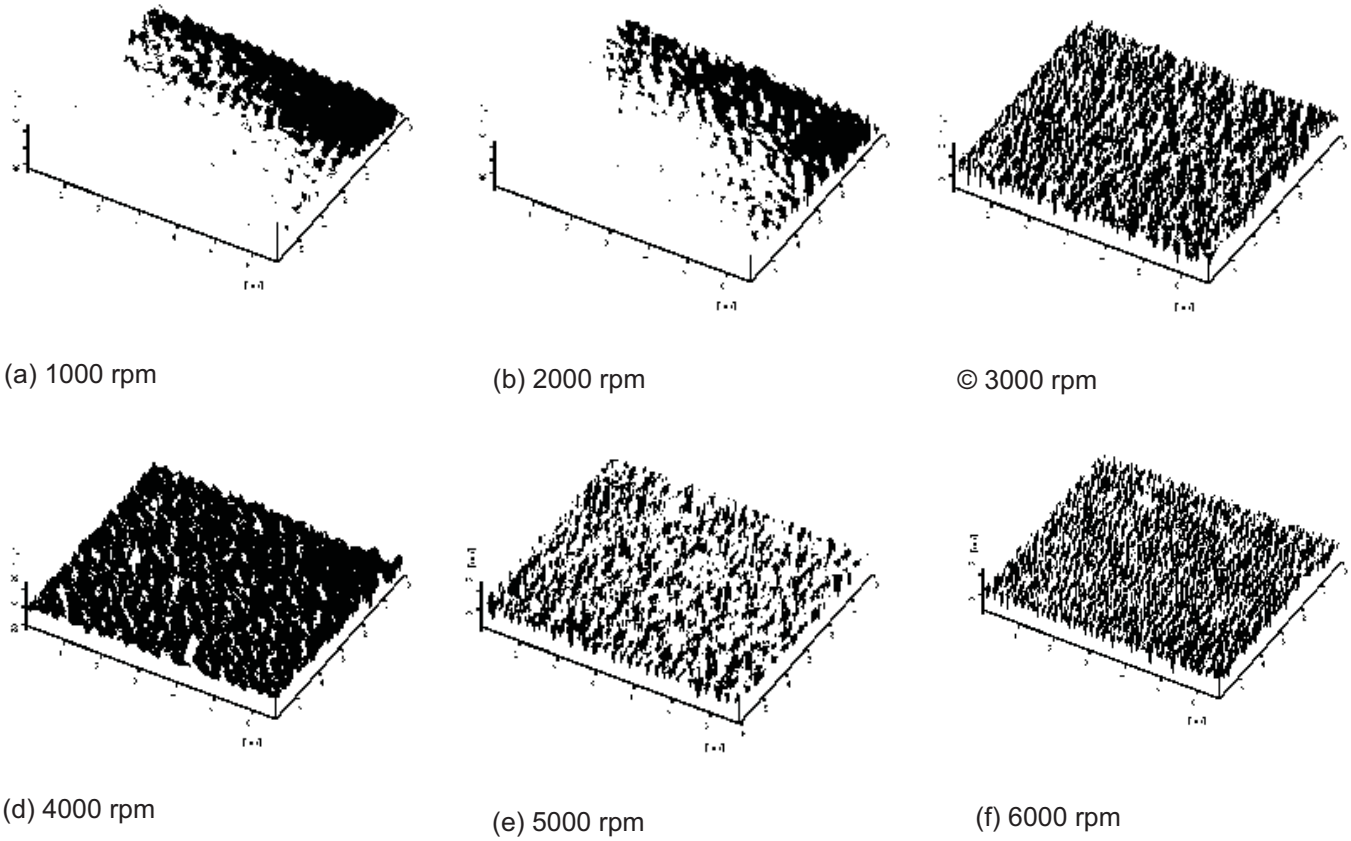

Figure 3. AFM images of 3D surface profiles of negative resist ma-N 2403 within spin speeds of 1000-6000 rpm.

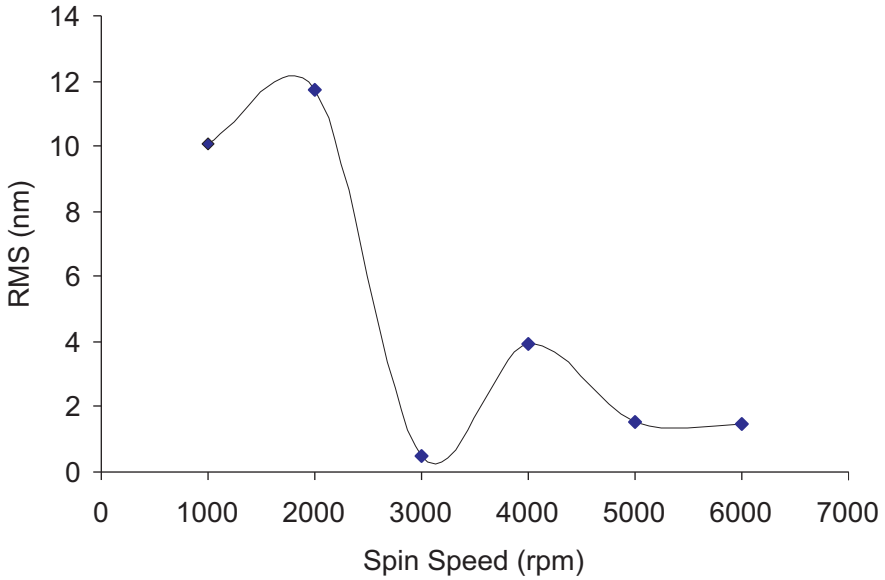

Figure 4. Average roughness of negative resist ma-N 2403 surface within spin speeds of 1000-6000 rpm.

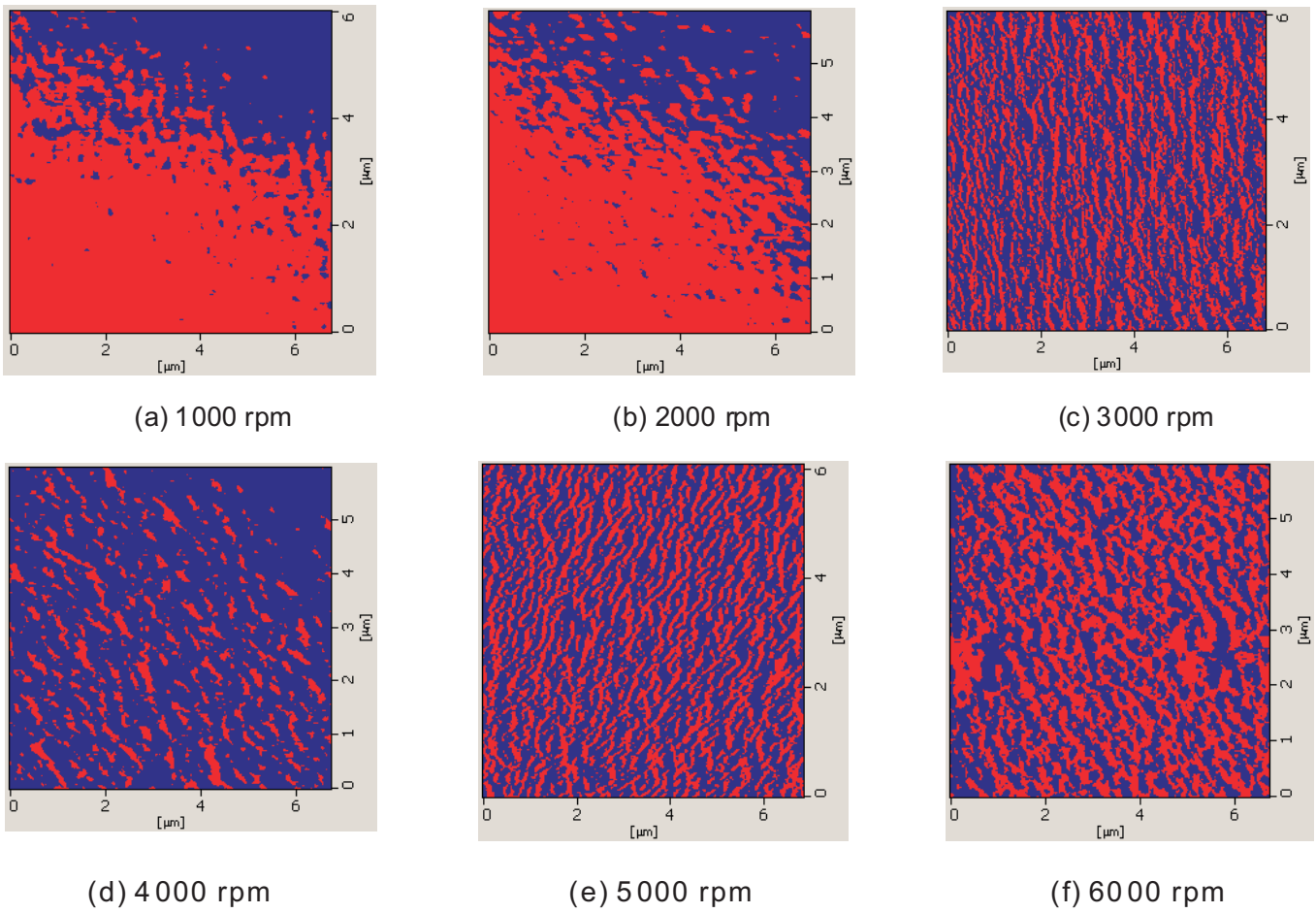

Figure 5. AFM images of grain size distribution within spin speeds of 1000-6000 rpm.

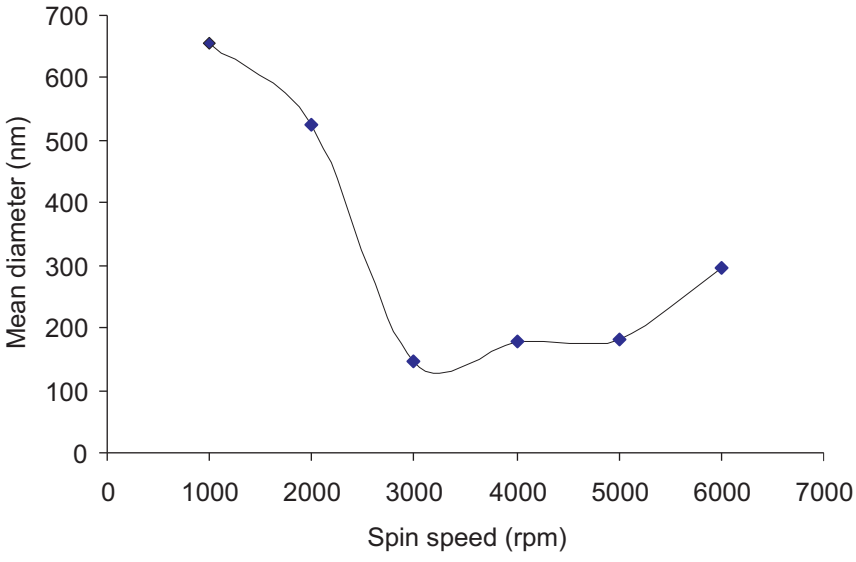

Figure 6. Average grain size of negative resist ma-N 2403 surface within spin speeds of 1000-6000 rpm.

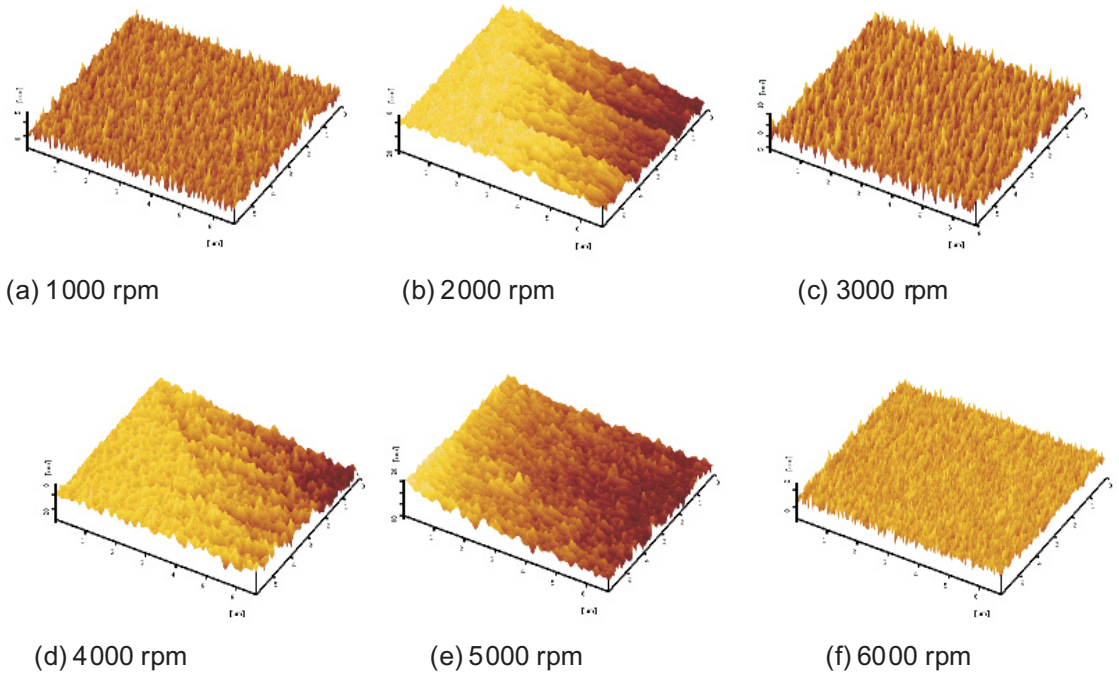

Figure 7. AFM images of 3D surface profiles of 495K PMMA within spin speeds of 1000-6000 rpm.

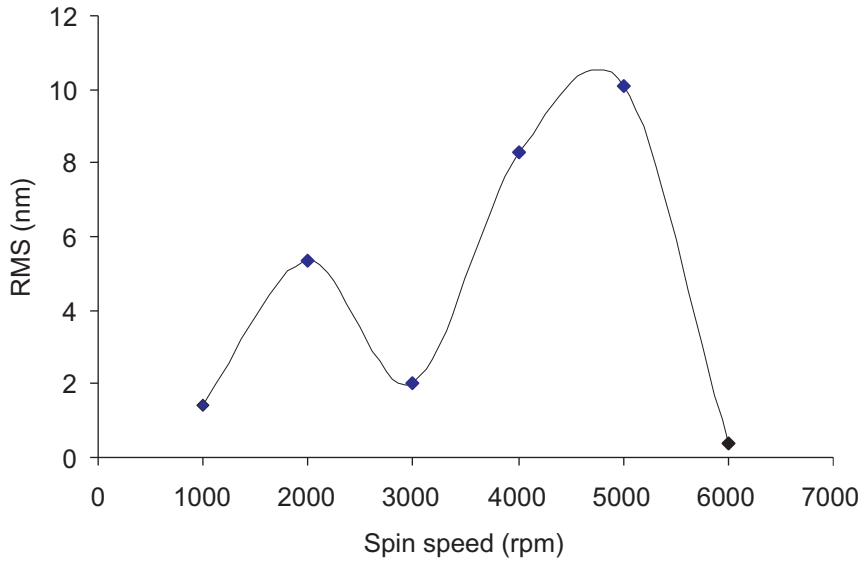

Figure 8. Average roughness of 495 K PMMA resist surfaces within spin speeds of 1000-6000 rpm.

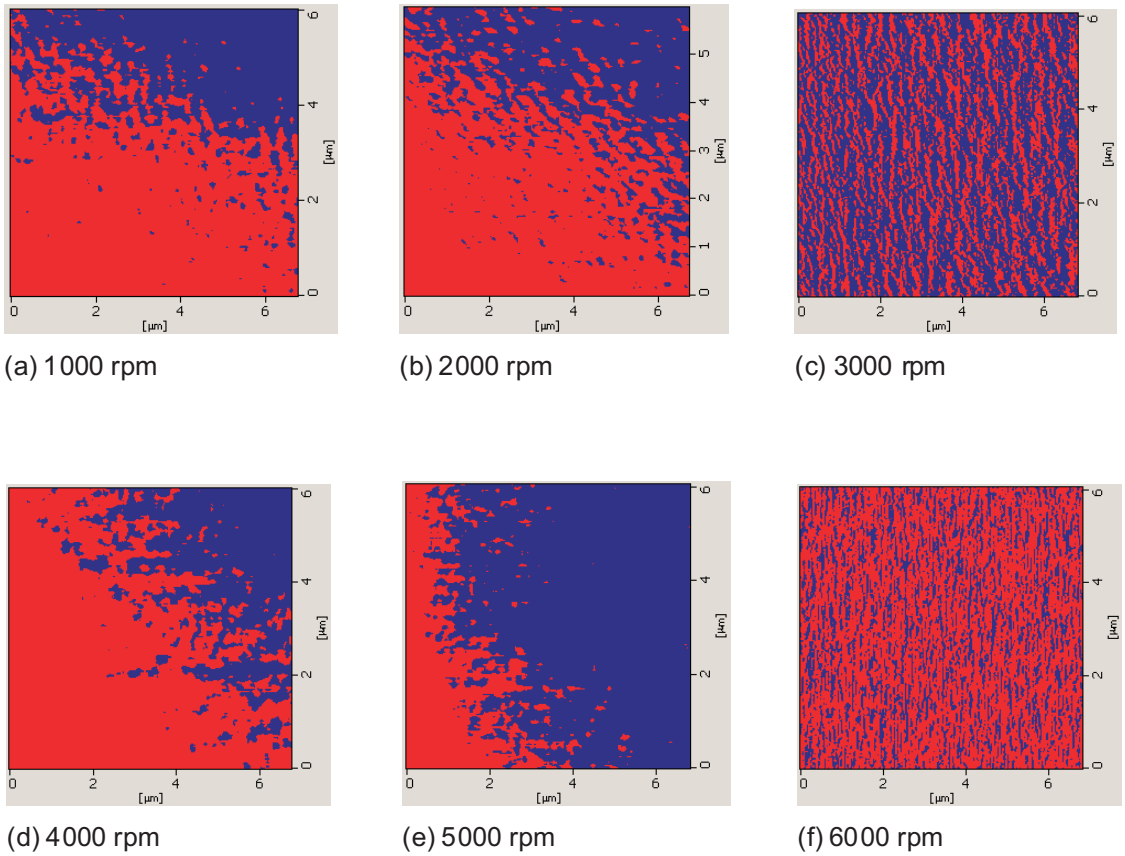

Figure 9. AFM images of grain size distribution within spin speeds of 1000-6000 rpm.

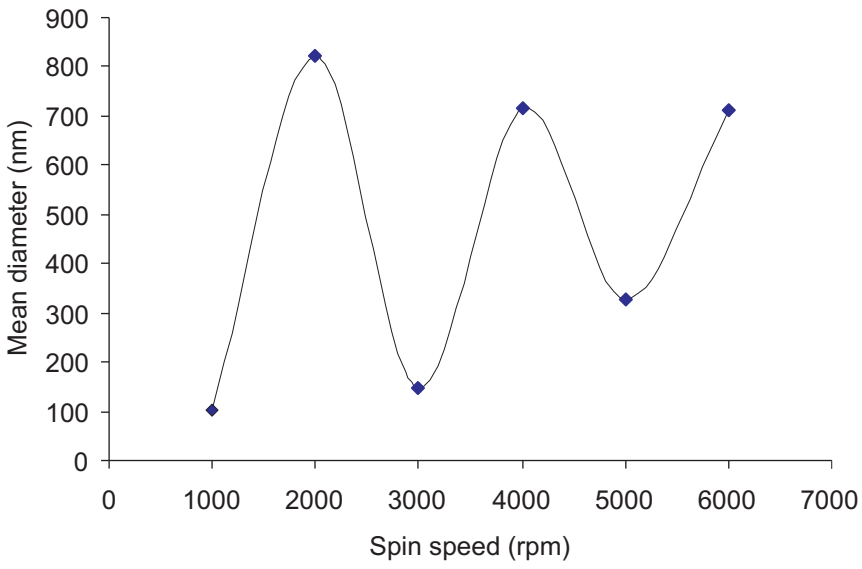

Figure 10. Average grain size of 495 K PMMA resist surface within spin speeds of 1000-6000 rpm.

noteworthy that resist which spincoated at 1000 rpm shows coagulation in the sides and edges of substrates. For Fig. 7(b), substrate is probably unexpec-tedly scratched so that there are two trenches in the deposited resist.PMMA resist show cone peaks. Compared with negative resist ma-N 2403, the density of cone peaks of 495 K PMMA resist seem higher. Heights o cone peaks range 2.60-7.67 nm and there had been found an outstanding peak as high as 37.08 nm in Fig. 7(d).

The graphs of average roughness have two peaks as depicted in Figs. 4 and 8. In Fig. 4, the left peak is higher than the right one. Conversely, in Fig. 8, the left peak is lower than the right peak. For 495 K PMMA resist, the form of grains is also long grains and as spin speed increases, the main diameter of grains increases, the amount of grains decreases and the distribution of grain also become more uniform.

CONCLUSION

The optimum resist thicknesses of Ma-N 2403 and 495 K PMMA were achieved at spin speeds of 3000 rpm. The resist surface of ma-N 2403 which resulted of 3000 rpm is high contrast, yellow colored, low roughness and no coagulation. The surface of 495 K PMMA is nearby the same as that of negative resist ma-N 2403, the difference is on its color only where PMMA is light brown colored.

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