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The Effect of Soft Bake on Adhesion Property Between SU-8 Photoresist and Ni Substrate by Molecular Dynamics Simulation

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ABSTRACT: SU-8 photoresist is a negative, epoxy-type, near UV photoresist and is commonly used in micro electro mechanical systems (MEMS) and other thick resist application fields. However, poor adhesion strength between SU-8 photoresist and metal substrate makes it difficult to bind, even contributes to the lithography failure. This significantly restricts the improvement of image resolution and depth-to-width ratio. As soft bake temperature and time are important for the interfacial adhesion property, in this article, molecular dynamics (MD) simulation was performed to investigate the effects of these two parameters on adhesion property between SU-8 photoresist and Ni substrate. According to the adsorption theory, the simulation results were analyzed and validated by experiments. It is shown that with increasing soft bake temperature the adhesional work firstly increases and then decreases, and with increasing soft bake time the adhesional work decreases. This study provides theory basis for the confirmation of soft bake parameters. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

KEYWORDS: molecular dynamics simulation; SU-8 photoresist; Ni substrate; adhesion property; soft bake parameters

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INTRODUCTION

SU-8 photoresist is a negative, epoxy-type, near UV photoresist first developed by IBM. With extremely low optical absorption in near-UV range, superior physical, mechanical, and optical qualities as well as chemical corrosion resistance and heat stability, SU-8 photoresist is commonly used for the fabrication of MEMS and other thick resist microstructures.[1] However, during SU-8 lithographic process, poor adhesion performance between SU-8 photoresist and metal substrate usually contributes to the binding failure. More seriously, the lithography can even be completely compromised.[2] These problems seriously limit further improvement of lithographic resolution and depth-to-width ratio of SU-8 photoresist. Therefore, it is necessary to investigate the adhesion property between SU-8 photoresist and metal substrate.

The adhesion property between SU-8 photoresist and metal substrate is sensitive to the process parameters such as soft bake temperature and time, exposure dose, development time, and post bake temperature and time.[3] Among which soft bake parameters are very important.[4] Inappropriate soft bake parameters usually result in poor adhesion. This may bring potential hazard and even failure to the subsequent processes.

MD simulation is recognized as one excellent method to investigate the interface behavior at the molecular level. Some researchers have successfully performed MD simulation to explore the adsorption behavior such as the adhesion energy and the intermolecular interaction of polymer–crystal, polymer–polymer, and polymer–oxides interface. Du Liqun et al.[5] performed MD to construct the interface structure of cross-linked SU-8 photoresist and Ni substrate and investigated the effect of post temperature on the interfacial adhesion property. Yin Jinghua et al.[6] simulated the adhesion energy of PI/α-Al_2O_3 and PI/SiO_2 composite systems and analyzed the effects of the nanoparticle superlattice size and doping on interface adhesion energy. Zhang Shuguang et al.[7] investigated the interactions between three kinds of water-soluble polymers and calcite crystal by MD simulation. B. Prathab et al.[8] performed MD simulations to investigate the interfacial interaction between PMMA and many other important polymers such as PAN, PC, and PVP, and to explore the adsorption behavior of MMA with several metal oxides (Al_2O_3, Fe_2O_3, SiO_2, and TiO_2).These researches provide basis for the investigation of the adhesion property between SU-8 photoresist and Ni substrate.

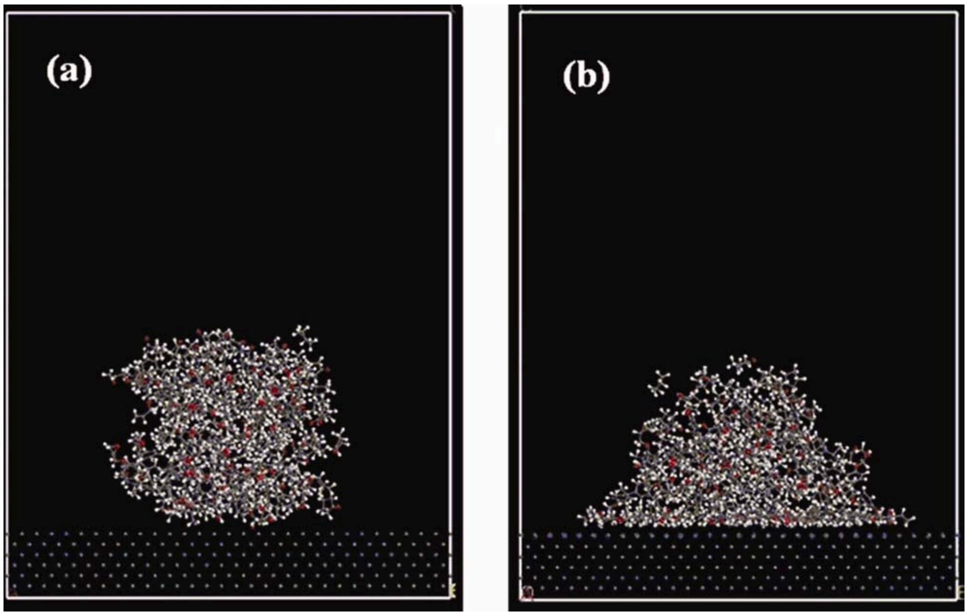

Figure 1. The model of the SU-8 photoresist and Ni substrate with the vacuum space (a) A SU-8 photoresist droplet on the Ni surface (b) The SU-8 photoresist droplet on the Ni surface reaches equilibrium after 200 ps dynamics process. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

In our earlier work, the effect of soft bake temperature on the adhesion property between SU-8 photoresist and Ni substrate has been investigated by MD simulation without considering the solvent cyclopentanone (C_5H_8O). The preliminary results indicate that at 343K the adhesion energy of SU-8 photoresist and Ni substrate reaches the maximum value, which means the interfacial adhesion property attains the best under this condition.[9] However, in that article, it was thought the structure of SU-8 photoresist only consisted of four SU-8 monomers but no solvent whose weight approximately accounts for one third of the SU-8 photoresist. Furthermore, because of the restriction of experiments condition, the research in that paper was carried out only by MD simulation without the experimental evidence.

As the purpose of soft bake is to remove the solvent,[10] the influence of the solvent on interfacial adhesion property should be taken into account. In this article, C_5H_8O molecules were incorporated into the SU-8 photoresist models. MD simulation was performed to investigate the interfacial adhesion property between SU-8 photoresist and Ni substrate and the work of adhesion was adopted to characterize the interfacial adhesion property. The effects of soft bake temperature and soft bake time on interfacial adhesion property were obtained. On the basis of the adsorption theory, the simulation results were analyzed and corresponding experiments were carried out to validate these results. This study provides theory basis for the confirmation of soft bake parameters.

MD SIMULATION

This paper focuses on the influences of two important parameters, soft bake temperature and soft bake time, on the adhesion property between SU-8 photoresist and Ni substrate.

The adhesion property between liquid and solid is generally characterized by the adhesional work, i.e., the amount of work per unit area that is needed to separate two adhering surfaces.[11] The value of adhesional work reflects the intermolecular interaction between two phases. The greater the value is, the firmer the adhesion of two phases and the stronger the interfacial intermolecular interaction. Therefore, in this paper the work of adhesion was adopted to characterize the interfacial adhesion property between SU-8 photoresist and Ni substrate.

Models Construction

Materials studio software package commercially available from Accelrys[12] was applied for simulation. DISCOVER module was used for force field calculation, with condensed-phase optimized molecular potentials for atomistic simulation studies (COMPASS). The MD simulations were studied in the isothermal-isochoric (NTV) canonical ensemble, and the temperature was controlled by the Andersen algorithm. The system was equilibrated for 200 ps with a time step of 1fs.

The interface system was constructed in an amorphous cell (Figure 1). First, metallic Ni was simulated as a face centered cubic crystal having unit cell lattice parameters of $a = b = c = 3.524$ Å, the Ni cell was cleaved paralleling to the (1 0 0) plane and extended to a super cell of $74.755 \times 74.755 \times 10.572$ Å^3. The top surface of metallic Ni was exposed to vacuum.

Second, SU-8 photoresist model, including SU-8 epoxy resin and C_5H_8O solvent which are the main components of SU-8 photoresist, was constructed. The molecular formulas of the two substances are shown in Figure 2. The relative molecular masses of SU-8 monomer and C_5H_8O molecule are 1170 and 84, respectively. The specific gravity of the SU-8 2015 photoresist is 1.2, with the mass concentration of C_5H_8O of 0.470 g·cc^{-1}. Therefore, the molecular ratio of SU-8 monomers to C_5H_8O molecules is approximately 1 : 8. An amorphous cell of SU-8 photoresist consisted of one

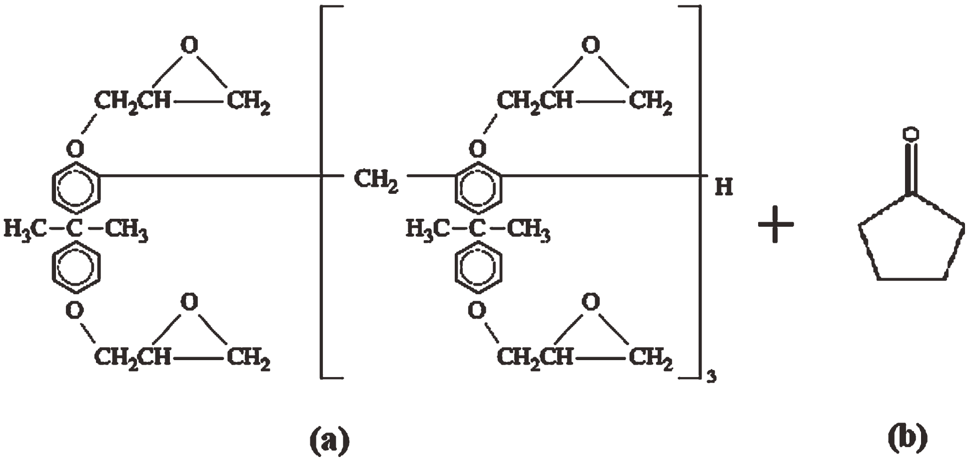

Figure 2. Molecular formulas of components of SU-8 photoresist (a) SU-8 monomer (b) cyclopentanone.

SU-8 monomer and eight C_5H_8O molecules was constructed and then extended to a (2 × 2 × 2) super cell. After removing the period boundary conditions and employing energy minimization method, the MD simulation was performed for 200ps in the isothermal-isochoric (NTV) canonical ensemble with the nonbond cut off distance of 9.5 Å. And a SU-8 solution droplet was obtained.

Finally, the droplet was placed on the Ni layer. With the displacements of Ni surface atoms in the *x*, *y*, and *z* directions constrained, energy minimization was performed on the system followed by a MD simulation for 200 ps.

Calculation of the Work of Adhesion

In the simulation, the interfacial work of adhesion between SU-8 photoresist and Ni substrate was acquired with the interfacial intermolecular interaction energy and interfacial area.[13] The work of adhesion is given by

$$W_a = E_{\text{interaction}}/A \tag{1}$$

where A is the van der Waals contact area. $E_{\text{interaction}}$ is the interaction energy between SU-8 photoresist and Ni substrate, given by

$$E_{\text{interaction}} = (E_{\text{SU-8}} + E_{\text{Ni}}) - E_{\text{total}} \tag{2}$$

where E_{total} is the total energy of SU-8 photoresist and Ni in contact at equilibrium. $E_{\text{SU-8}}$ and E_{Ni} are the single point energies of SU-8 photoresist and Ni separated in vacuum at equilibrium, respectively.

To calculate the van der Waals contact surface area, the surface of SU-8 photoresist and Ni substrate in the interface system was constructed using van der Waals's radii of all the constituent atoms and a plane superposed with the interface was set. After removing all of the Ni atoms, the bottom view of the SU-8 photoresist equilibrium conformation on the plane is plotted in Figure 3. We exported this image and transformed the color mode into black and white. The percentage of black pixels was obtained by image processing software Adobe Photoshop CS2, and the van der Waals contact surface area A was calculated with the dimensions of the interface system.

Simulation of the Effects of Soft Bake Parameters on Adhesion Property

For soft bake temperature simulation, MD simulations were performed on the interfacial systems for 200 ps at the temperatures of 328, 338, 348, 358, and 368 K, and the works of adhesion at these temperatures were obtained. For soft bake time simulation, as we known, the main function of soft bake is to remove the solvent. Obviously, at the same temperature, the longer soft bake time costs, the more the solvent volatilizes, and the lower the concentration of the solvent in solution becomes. Although the volatilization velocity of solvent at the upper surface of solution is greater than that near the interface between liquid and solid, which forms the concentration difference in SU-8 photoresist solution, the interfacial adhesion property is mainly affected by the molecules in the close vicinity of the interface but not that in the bulk solution. Therefore, it was feasible that only the concentration of the solvent at the interface was taken into account. The interfaces with different volatilization of solvent were constructed by setting the ratios of SU-8 monomers to C_5H_8O molecules as 1 : 8, 1 : 7, 1 : 6, 1 : 5, and 1 : 4. The corresponding densities of solutions were approximately evaluated as 1.200 $g{\cdot}cc^{-1}$, 1.219 $g{\cdot}cc^{-1}$, 1.227 $g{\cdot}cc^{-1}$, 1.236 $g{\cdot}cc^{-1}$, and 1.238 $g{\cdot}cc^{-1}$ by interpolation method based on the values in the data sheets of the different types of SU-8 photoresist.[14]

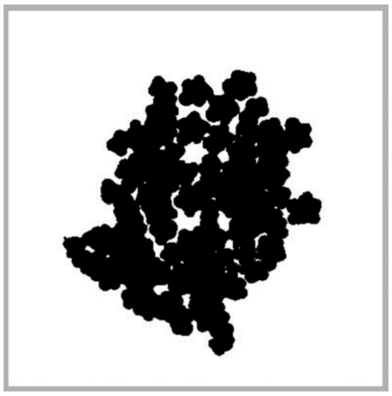

Figure 3. Bottom view of the SU-8 photoresist equilibrium conformation on the plane used for calculation of the van der Waals contact area.

EXPERIMENT RESEARCH

In order to investigate the effects of soft bake temperature and time on the interfacial adhesion property, the work of adhesion between SU-8 photoresist and Ni substrate was experimentally tested and calculated. Two sets of experiments (i.e., constant molar ratio of SU-8 monomers to C_5H_8O molecules of 1 : 8, varied soft bake temperatures of 328, 338, 348, 358, and 368 K and constant soft bake temperature of 358 K, varied molar ratios of 1 : 8, 1 : 7, 1 : 6, 1 : 5, 1 : 4) were carried out. These experiments were employed to validate the simulation results.

The works of adhesion between SU-8 2015 ((MicroChem Corporation USA, the molecular ratio is approximately 1 : 8) and Ni substrate were tested and calculated at different temperatures. The experimental processes were as follows:

1. Polish the surface of Ni substrate.
2. Put the Ni substrate into acetone and ethanol for ultrasonic washing for 20 min, dry it in a baking oven, and then put it into peltier temperature control box of contact angle measuring system (DSA100, Kruss Company, German).
3. Test the contact angles between SU-8 2015 photoresist and Ni substrate by sessile drop method at 328, 338, 348, 358, and 368 K.
4. Test the surface tensions of SU-8 photoresist at various soft bake temperatures by hanging drop method.
5. According to the theory of wetting-adsorption, the work of adhesion is given by

$$W_a = \gamma(1 + \cos\theta) \tag{3}$$

where γ is the surface tension of liquid, θ is the contact angle between liquid and solid. Calculate the works of adhesion by eq. (3) with the surface tensions and the contact angles obtained in the experiment.

Then, the works of adhesion in different interfacial molecular ratios of SU-8 monomers to C_5H_8O molecules were tested and calculated at 358 K. The experimental processes were as follows:

1. Polish Ni substrate and wash it with ultrasonic.
2. Calculate the volatile masses of the solvent in the molecular ratios of 1 : 8, 1 : 7, 1 : 6, 1 : 5 and 1 : 4, based on certain weight of SU-8 2015 photoresist.
3. Put the bottled SU-8 2015 photoresist into fume hood for the volatilization of the solvent and obtain the volatile massed with scale.
4. Take SU-8 photoresist solutions out of the bottle with syringe when the volatile masses of the solvent achieve the calculated values.
5. Test the surface tensions of SU-8 photoresist solutions in different molecular ratios and the contact angles between SU-8 photoresist and Ni substrate at 358K. Calculate the works of adhesion according to eq. (3).

RESULTS AND DISCUSSION

The Effect of Soft Bake Temperature on the Interfacial Adhesion Property

In MD simulation, the SU-8 photoresist droplet approached and dropped onto the Ni substrate forming the interface, as shown in Figure 1(b). From the simulation, the single point energies of SU-8 photoresist and Ni substrate $E_{SU\text{-}8}$, E_{Ni}, the total energy E_{total} and the interfacial van der Waals contact area A were acquired. According to eqs. (1) and (2), the interaction energy $E_{interaction}$ and the work of adhesion W_a were calculated. The MD simulations were performed on the system at 328, 338, 348, 358, and 368 K. And the relationship between the work of adhesion and the temperature is obtained shown in Figure 4, where the signs "-■-" and "-▲-" represented the simulation data and experimental data, respectively. The experimental data were obtained by testing the surface tensions of SU-8 photoresist and the contact angles between SU-8 photoresist and Ni substrate at different temperatures. It is shown that in simulation the work of adhesion firstly increases and then decreases with the increasing temperature.

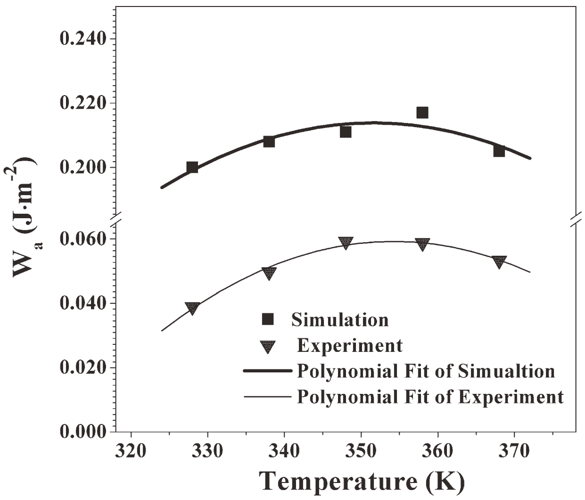

Figure 4. Dependence of the work of adhesion between SU-8 photoresist and Ni substrate on the temperature in the experiment and MD simulation.

Analysis of Simulation Results. For the effect of soft bake temperature on the interfacial adhesion property, we supposed it was related to the types and concentration of the interfacial molecules, and to the distance from SU-8 photoresist molecule to Ni surface. Therefore, in this article the influences of these factors were specifically analyzed.

First, the effect of the temperature on adsorption of polymer generally embodies in its effect on the solubility of polymer in solvent. If the solubility increases with the temperature, the adsorption quantity of solute polymer on solid substrate decreases, vice versa. In this article, the MD simulation were performed on C_5H_8O and SU-8 photoresist systems at different temperatures in DISCOVER module and the solubility parameters of the solvent C_5H_8O and solution SU-8 photoresist were calculated in Amorphous Cell module shown in Figure 5. According to the relationship of solubility parameters between the of mixed solution and its components given by

$$\delta_{SU\text{-}8/C_5H_8O} = \varphi_{C_5H_8O}\cdot\delta_{C_5H_8O} + \varphi_{SU\text{-}8}\cdot\delta_{SU\text{-}8} \tag{4}$$

where $\delta_{SU\text{-}8/C_5H_8O}$ is solubility parameter of SU-8 photoresist. $\varphi_{C_5H_8O}$, $\varphi_{SU\text{-}8}$ are the volume fractions of C_5H_8O (0.358) and

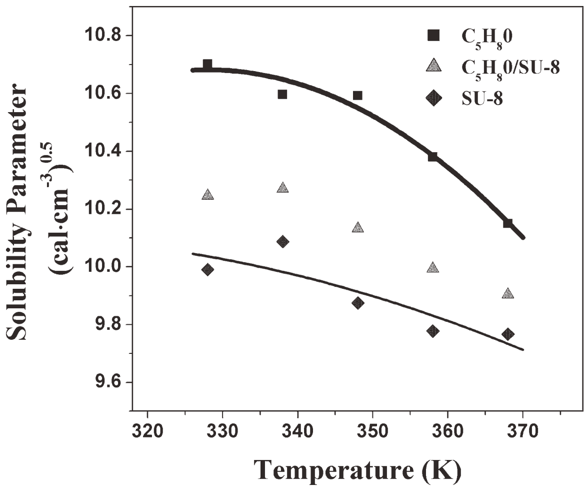

Figure 5. The dependence of solubility parameter on temperature.

SU-8 (0.642) in SU-8 photoresist, respectively. $\delta_{C_5H_8O}$, $\delta_{SU\text{-}8}$ are solubility parameters of C_5H_8O and pure SU-8, respectively. The solubility parameter of solute pure SU-8 was obtained shown in Figure 5. The more similar the solubility parameters are the better the solute dissolves in solvent. Figure 5 shows that the solubility parameters of pure SU-8 and pure C_5H_8O closed to each other with the increasing temperature. That means the solubility of solute SU-8 in the solvent C_5H_8O increases with temperature. Therefore, it is inferred that the adsorption quantity of solute SU-8 on the Ni surface decreases with increasing temperature.

For more accurate analysis, the relative concentrations of C_5H_8O molecules in SU-8 photoresist system at the interface at different temperatures were analyzed through FORCITE module. As only one double bond is contained in a C_5H_8O molecule, its concentration could represent C_5H_8O molecular concentration. We edited all of double bonds in the interfacial system as a set and calculated their concentrations at 328, 338, 348, 358, and 368 K. The dependence of the relative concentration of C_5H_8O molecules near the interface on the temperature is shown in Figure 6. It indicates that the C_5H_8O molecular concentration first increases and then keeps steady with the increasing temperature.

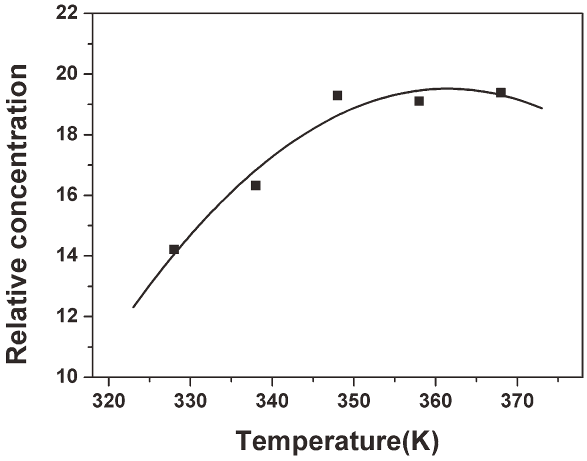

Figure 6. Dependence of the relative concentration of C_5H_8O molecules near the interface on the temperature.

Second, the interfacial intermolecular interaction energy between Ni substrate and SU-8 or C_5H_8O molecules was researched. In order to eliminate the influence of molecular quantity in system on simulation result, two types of construction methods were adopted in this paper. One method was constructing the interface between Ni substrate and only one C_5H_8O molecule or one SU-8 monomer, as shown in Figure 7(a,b), respectively. Other one was constructing the interface between Ni substrate and the amorphous cell (layer)of C_5H_8O molecules or SU-8 monomers with the density of 770 $g{\cdot}cc^{-1}$ or 430 $g{\cdot}cc^{-1}$, respectively, as shown in Figure 7(c,d). After a relaxation of the structure to a state of minimum potential energy, MD simulations were performed on the systems for 50 ps. The works of adhesion of C_5H_8O single molecule/Ni system and C_5H_8O layer/Ni system respectively were 0.226 $J{\cdot}m^{-2}$, 0.224 $J{\cdot}m^{-2}$, and the ones of SU-8 monomer/Ni system and SU-8 layer/Ni system respectively were 0.186 $J{\cdot}m^{-2}$ and 0.189 $J{\cdot}m^{-2}$. It indicates that the works of adhesion calculated through the two methods agree with each other and are independent of the quantity of molecules incorporated in model systems. Moreover, the work of adhesion of C_5H_8O/Ni interface is more than that of SU-8/Ni

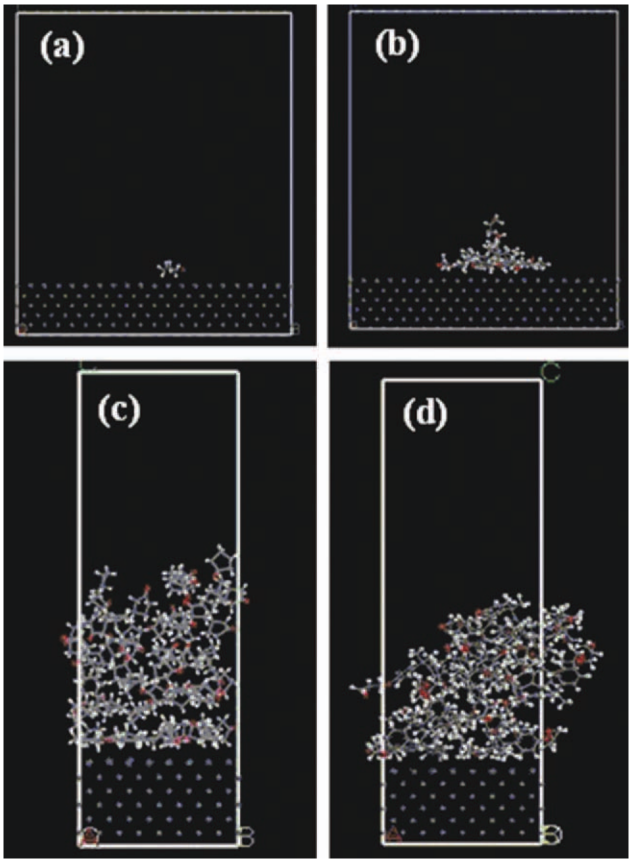

Figure 7. The interface models (a) C_5H_8O single molecule/Ni (b) SU-8 monomer/Ni (c) C_5H_8O layer/Ni (d) SU-8 layer/Ni. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

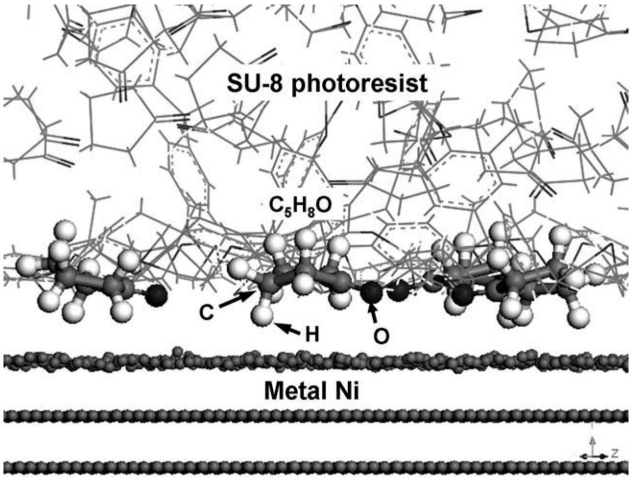

Figure 8. Snapshot of the final state of the simulated interfaces between SU-8 photoresist and cyclopentanone (colors: carbon atom-gray, hydrogen-white, oxygen-black).

interface, i.e., in the same contact areas, the interaction energy between C_5H_8O molecules and Ni atoms is more than that between SU-8 monomers and Ni atoms. For further analysis, due to the polarity of carbonyl group in cyclopentanone molecule and the high electronegativity of oxygen atom, the van der Waals' force between carbonyl group and metal Ni becomes stronger. This is validated in Figure 8 which shows that the oxygen atom in a cyclopentanone molecule closes to Ni surface than other five carbon atoms. Additionally, take the case of 328 K for example, the distance between Ni surface and all of cyclopentanone molecules on Ni surface is calculated as 3.06 Å, less than that between SU-8 photoresist and Ni surface of 3.32 Å. It also indicates that the interaction between cyclopentanone and metal Ni is stronger than that between SU-8 molecule and metal Ni.

Finally, the effect of temperature on the distance between SU-8 photoresist and Ni surface was investigated. The average distances between the Ni surface and the SU-8 photoresist atoms less than 5 Å away from Ni surface at different temperatures were listed in Table I. It is obvious that the distance increases with temperature, while the interfacial intermolecular interaction energy decreased with the increasing distance between SU-8 photoresist and Ni surface. Therefore, the interfacial intermolecular interaction energy decreases with the increasing temperature.

Therefore, the dependence of the work of adhesion on temperature shown in Figure 4 could be interpreted as following. The adsorption quantity of the solute SU-8 monomers decreases with the increasing temperature. It means the SU-8 molecules decrease and are replaced by C_5H_8O molecules at the interface. As the interfacial energy of C_5H_8O/Ni system is greater than that of SU-8/Ni system, the increase of C_5H_8O molecular concentration at the interface contributes to the increase of interfacial intermolecular interaction energy, i.e., the increase of the work of adhesion. When the intermolecular interaction in SU-8 solution equilibrates with interfacial adsorption force, the ratio of SU-8 monomers to C_5H_8O molecules remains unchanged. However, the further increase of temperature induces the increase of the distance from solution molecules to substrate. The interaction energy between these two phases begins to decrease and the work of adhesion declines.

Table I. The Interfacial Molecular Distances at Different Temperatures

Temperature (K)	Molecular distance (Å)
328	3.32
338	3.91
348	4.15
358	4.57
368	4.99

Table II. Surface Tensions and Contact Angles at Different Temperatures

Temperature (K)	Surface tension ($mN·m^{-1}$)	Contact angle (°)
328	37.75	88.23
338	35.68	66.80
348	35.05	46.28
358	33.48	40.85
368	29.59	36.64

Experimental Results and Analysis. The surface tensions of SU-8 photoresist and the contact angles between SU-8 photoresist and Ni substrate at 328, 338, 348, 358, and 368 K were tested in the experiments. The results in Table II show that the surface tension and the contact angle decrease with the increasing temperature. In Figure 4, the work of adhesion calculated by eq. (3) firstly increases and then decreases with the increasing temperature, which agrees well with the result in the simulation. However, in Figure 4, the work of adhesion in the experiment is less than that in the simulation. It is possibly because the Ni surface is ideally pure and clear in the simulation but is inevitably covered with oxide film, oil stain, or cyclopentanone steam which decrease the surface energy of nickel in the experiment.

The Effect of Soft Bake Time on Interfacial Adhesion Property

Figure 9 is the dependence of the work of adhesion between SU-8 photoresist and Ni substrate on the molecular ratio. The signs "-■-" and "-▲-" represent the experimental and simulation results, respectively. The simulation results were obtained by performing MD simulation on the SU-8 photoresist/Ni substrate system with the molecular ratios of SU-8 monomers to C_5H_8O molecules of 1 : 8, 1 : 7, 1 : 6, 1 : 5, and 1 : 4 at 358 K. The experimental results were obtained by testing the surface tensions and the contact angles with the Ni substrate of the SU-8 solutions in different molecular ratios at 358 K.

In Figure 9, the work of adhesion decreases with the increase of the molecular ratio. That is because with the increase of soft bake time, the solvent continuously volatilizes, the C_5H_8O molecules decreased at the interface, and the molecular ratios of the SU-8 monomers to C_5H_8O molecules increase. According to the preceding analysis, interfacial intermolecular energy of C_5H_8O /

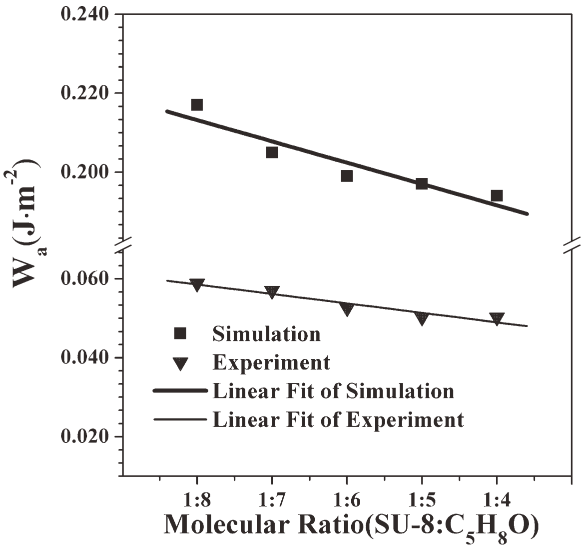

Figure 9. Dependence of the work of adhesion between SU-8 photoresist and Ni substrate on the molecular ratio in the experiments and MD simulation.

Ni is greater than that of SU-8/Ni. Therefore, the volatilization of solvent results in the decrease of the adhesional work.

Even though the data in the experiment are less than those in the simulation, which could be attributed to the oxide film, oil stain, or cyclopentanone steam covered on Ni surface, the variation tendency of experimental results agreed well with that of simulation ones.

CONCLUSIONS

1. In this article, the adhesion property between SU-8 photoresist and Ni substrate is characterized by the work of adhesion. The MD simulation is performed on the interface between SU-8 photoresist and Ni substrate and the effects of soft bake temperature and soft bake time on the interfacial adhesion property are obtained. The results show that the work of adhesion firstly increase and then decrease with the increase soft bake temperature, and decrease with the increasing soft bake time.
2. According to the adsorption theory, the effects of soft bake temperature and soft bake time on the interfacial adhesion property are investigated and analyzed. With the increase of temperature, the adsorption of SU-8 molecules to Ni surface decreases while that of C_5H_8O solvent increases, moreover the interfacial intermolecular interaction energy in C_5H_8O /Ni system is greater than that in SU-8/Ni system and thus the work of adhesion increases. When the soft bake temperature rises to certain extent, the ratio of SU-8 monomers to C_5H_8O molecules remains stable. Further increase of temperature induces the increase of distance from SU-8 solution to Ni substrate and the decrease of interfacial interaction energy and thus the work of adhesion declines. With the increase of soft bake time, the solvent C_5H_8O decreases at the interface. As the interfacial intermolecular interaction energies per unit contact area in C_5H_8O /Ni system are more than those in the SU-8/Ni system, the work of adhesion decreases.
3. The surface tension of SU-8 photoresist and the contact angle between SU-8 photoresist and Ni substrate were tested in the experiment. The interfacial work of adhesion and the effects of soft bake temperature and soft bake time on the interfacial adhesion property were acquired. The effect results obtained in the experiment agree with those in the simulation.

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