Impact of developers on roughness of dissolution front in electron-beam resists

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We describe the impact of various developers on the roughness of dissolution front of electron-beam positive-tone resist ZEP7000. In this study, two types of developers are investigated: single-solvent developer and binary-solvent developer. Atomic force microscope observations of dissolution fronts reveal that, for a homologous series of *n*-alkyl acetate as a single-solvent developer, there are three categories of dissolution process: molecular-level dissolution, aggregate extraction development, and their intermediate. Small solvent molecules, such as methyl- and ethyl acetate, are very effective for reducing roughness. For good solvents for ZEP resist as single-solvent developer, molecular-level dissolution also occurs and the dissolution front becomes very flat and smooth. However, these developers, though effective for reducing roughness, lead to lower dissolution contrast. We clarified that binary-solvent developers consisting of these effective developers and *n*-hexane as a poor solvent, especially 1:1 ethyl acetate/*n*-hexane developer, provide both low roughness and high dissolution contrast.

Keywords: Resists, Roughness, LER, developers, dissolution

1. Introduction

Line-edge roughness (LER) of a resist pattern is a critical issue in the lithographic field. The LER could degrade the resolution as well as the linewidth accuracy as pattern size shrinks. The origin of the LER has been extensively examined from various viewpoints. It is known that LER is related to factors involving the lithographic system, such as aerial image profiles [1–7], shot noise [8, 9], and mask roughness [1, 10], and also to resist materials, such as the inhomogeneity of a resist polymer film due to oligomer components [11], molecular weight distribution [12], polymer aggregates [13–19], and phase separation structures [20–22], and also to statistical variations in photochemical events [23–25].

The contribution of factors involving the lithographic system, such as aerial image profiles and shot noise, to LER is not small, but it would not be dramatically reduced as pattern size shrinks or as higher sensitivity is required. Therefore, we believe that a realistic approach to reducing LER is to control the characteristics of resist materials and the resist processes. Moreover, it becomes more and more

important to cancel out the contribution of the lithographic system to LER by improving the resist materials and processes.

The important thing at this point is the dissolution process of resist polymers. Were resist polymers able to always dissolve uniformly from the surface in a flood-exposed region, the LER would be zero. This is self-evident, considering that roughness cannot be simulated merely from dissolution curve, the relationship between dissolution rate and exposure dose. However, rarely does resist polymer dissolve uniformly from the surface; and roughening inevitably occurs at the dissolution front. It is certain that inhomogeneous dissolution leads to large LER. Therefore, the key to resolving the LER issue is to find a developer or development condition that ensures uniform dissolution of resist polymers.

Recently, we have clarified, for the electron-beam positive-tone resist ZEP520, that the morphology of the dissolution front largely depends on the developer. This means that the generation of the roughness is deeply related to the dissolution process [26]. For amyl- and hexyl acetates, which are

high-resolution developers for ZEP520, dissolution proceeds with the extraction of polymer aggregates naturally contained in resist films. As a result, the dissolution front contains many aggregates and becomes rough. However, for methyl- and ethyl acetates, we have found that there is a condition that the aggregate extraction does not occur. This is because the flood-exposed regions dissolve uniformly keeping its surface flat without aggregate extraction for methyl- and ethyl acetates. This means that molecular-level dissolution occurs in these developers. However, with these developers, the dissolution contrast decreases because even unexposed films easily dissolve in them. The dissolution contrast is inconsistent with the roughness of the dissolution front in ZEP520.

The purpose of this study was to try to find a developer that can overcome this trade-off between the dissolution contrast and the roughness of the dissolution front. Here we report the impact of various developers on the roughness of the dissolution front of ZEP7000. In ZEP7000, higher dissolution contrast than in ZEP520 is expected for an identical developer because it is hard for unexposed ZEP7000 film to dissolve due to its higher molecular weight.

2. Experimental

The resist examined in this study was the positive-tone electron-beam resist ZEP7000 (Nippon Zeon), which is a chain-scission type containing copolymers of methyl (α -chloroacrylate) and α -methylstyrene [27]. The only difference between ZEP7000 and ZEP520 is their molecular weight properties. Molecular weight properties [the number average molecular weight (M_n), weight average molecular weight (M_w), and polydispersity (M_w/M_n)] were measured by gel permeation chromatography (GPC)-viscometry. ZEP7000 has $M_w = 476,000$ and $M_w/M_n = 1.70$, whereas ZEP520 has $M_w = 67,000$ and $M_w/M_n = 2.20$.

The resists were spin-coated on a wafer to a thickness of 500 nm and then prebaked on a hot plate at 200°C for 2 min. The resist films were exposed to an electron beam with an acceleration voltage of 70 kV. The exposure area was 24- μm square. The films were developed with both single-solvent and binary-solvent developers. As single-solvent developers, we used a homologous series of alkyl acetates ranging from methyl acetate to octyl acetate, 2-butanone, and good solvents for a ZEP resist polymer, such as *o*-dichlorobenzene, anisole, and diethylene glycol dimethyl ether (diglyme). As binary-solvent de-

velopers, we used mixed solvents with these good solvents and poor solvent, such as 2-propanol and *n*-hexane. Unless otherwise mentioned, the exposed resist films were developed with the above developers for 90 s at 23°C and then rinsed with 2-propanol for 30 s.

The thickness of the resist that remained on an exposed area after development was measured with an interferometer (Nanometrix, 210XP). Surfaces of exposed resist films after development were observed with an atomic force microscope (AFM) (Seiko Instruments, SPA500/SPI3800) in the dynamic force mode. Scan areas were of 1- μm square (256 $\times$ 256 pixels) for all samples. The magnitude of roughness is defined as the root-mean-square (Rms) value (1σ) of the measured height of the AFM image.

3. Results and Discussion

3.1. Single-solvent developer

3.1.1. Conventional developers

First, we obtained contrast curves of ZEP7000 after development with a homologous series of *n*-alkyl acetate. As shown in Fig. 1, as the size of the ester group in these acetates decreases, the dose to clear decreases but the dissolution contrast decreases. However, compared with ZEP520, which has lower molecular weight than ZEP7000, the dissolution contrast is still high, especially in the case of development with acetates not larger than ethyl acetate. This is because unexposed ZEP7000 films hardly dissolve even in acetates not larger than ethyl acetate because of ZEP7000's higher molecular weight. As expected, these results indicate that ZEP7000 has a great advantage for dissolution con-

Figure 1: Contrast curves of ZEP7000 resist after development with a homologous series of *n*-alkyl acetate.

Figure 2: AFM images of dissolution front of ZEP7000 with a thickness of 500 nm after development with ethyl- to amyl acetate. Exposure dose (D) and thickness loss (TL) are shown in each image.

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Next, the roughness of the dissolution front of ZEP7000 was investigated. In this paper, the surface of exposed resist after development, rinsing, and drying is considered to be the dissolution front. Figure 2 shows AFM images of the dissolution front after development with ethyl acetate to amyl acetate. For both methyl acetate (not shown) and ethyl acetate, the dissolution front is very smooth and flat. For propyl acetate, however, it becomes very rough because it contains worm-like domains more than 300 nm in width. As the exposure dose increases, the worm-like structures diminish and then dot-like structures appear. For butyl acetate, the dissolution front shows similar behavior, although the morphology changes from worm-like to dot-like the moment the thickness loss exceeds about 100 nm. For amyl acetate and hexyl acetate (not shown), which are high-contrast developers for ZEP resists, polymer

aggregates appear on the dissolution front, which means that the development proceeds with aggregate extraction as previously reported. From these results, it is clear that the morphology of the dissolution front of ZEP7000 strongly depends on the kinds of developers used.

Figure 3 shows the relationship between roughness of the dissolution front and exposure dose. The exposure dose is converted to the thickness loss of an exposed region with a corresponding dose. For methyl- and ethyl acetate, as expected from the surface morphology shown in Fig. 2, the magnitude of roughness is almost constant, between 2 and 3 nm, irrespective of thickness loss or exposure dose. For propyl acetate, the magnitude of roughness rapidly increases for the thickness loss of 100-200 nm and then levels off when the thickness loss exceeds 200 nm. For butyl acetate, the behavior is the same as that for propyl acetate, except that the magnitude of

Table 1: Effect of developer size on the dissolution process and roughness of the dissolution front

Developer	methyl	ethyl	propyl	butyl	amyl	hexyl	octyl
Molecular size	small		-		large		
Penetrability into aggregates	easy		intermediate		difficult		
Dissolution process	molecular-level		intermediate		aggregate extraction		
Roughness (nm)	2.6	2.4	27.1	26.4	18.7	15.5	15.2

Figure 3: The relationship between the roughness of the dissolution front and exposure dose.

roughness rapidly increases from the start. For amyl acetate, moreover, the maximum roughness at the level regions decreases. For hexyl- and octyl acetate, the maximum value further decreases.

These results are summarized in Table 1. This roughness behavior can be explained by the relationship between the size of the developer and the size of free-volume holes in resist films. The van der Waals (VDW) volumes of conventional developers are shown in Table 2. From the results in Fig. 3, it is clear that there is a critical molecular size between ethyl- and propyl acetate, which is about $100 \text{ \AA}^3$. Therefore, the average size of free-volume holes is expected to correspond to this critical molecular size. Moreover, these free-volume holes are not homogeneously dispersed in a resist film. Regarding the factor of inhomogeneous distribution of free-volume holes, we have already focused on polymer aggregates 20-30 nm in size, which are naturally contained in resist films [14, 17, 28]. Polymer density inside the aggregates is higher than that of surrounding polymers. In other words, the size of free-volume holes inside the aggregates or the number of free-volume holes is smaller. Therefore, the solubility of the aggregates itself, or the penetrability of a developer solvent molecule into the aggregates, is

Table 2: Calculated VDW volumes of a homologous series of n-alkyl acetate and other good solvents

Component	VDW volume ^{a,b,c} $\text{\AA}^3$
Conventional developers	
Methyl acetate	74.4
Ethyl acetate	91.4
Propyl acetate	108.3
Butyl acetate	125.3
Amyl acetate	142.2
Hexyl acetate	159.2
2-Butanone	82.2
Good solvents	
Anisole	109.6
<i>o</i> -Dichlorobenzene	111.0
2-Methoxyethyl acetate	125.1
Diglyme	140.7

^a The VDW volume was calculated by Bondi's method [29], based on the atomic hard-sphere model.

^b VDW radii used for calculations [29]: C(1.70 $\text{\AA}$), H(1.20 $\text{\AA}$), O(1.52 $\text{\AA}$), Cl(1.75 $\text{\AA}$).

^c Covalent bond length used for calculations: C-C(1.54 $\text{\AA}$), C=C(1.34 $\text{\AA}$), C-H(1.10 $\text{\AA}$), C-O(1.43 $\text{\AA}$), C=O(1.23 $\text{\AA}$), C-Cl(1.77 $\text{\AA}$).

the key to understanding this roughness behavior.

For small developers such as methyl- and ethyl acetate, the dissolution proceeds at the molecular level because the developers easily penetrate the aggregates through the free-volume holes. As a result, the dissolution front becomes smooth and flat. For large developers such as hexyl- and octyl acetate, on the other hand, the dissolution proceeds with aggregate extraction because it is hard for relatively large developers to penetrate polymer aggregates themselves and then polymers surrounding the aggregates dissolve first. As a result, the dissolution front

Figure 4: Contrast curves of ZEP7000 after development with various good solvents.

becomes rough because of polymer aggregates appearing there. For propyl- and butyl acetate, whose molecular size would be about the same as the average size of free-volume holes, two regions, in which molecular-level dissolution and aggregate extraction development occur, coexist. Therefore, large undulations, such as worm-like structures appear, on the dissolution front. This morphology would depend on the initial distribution of free-volume holes in resist films before exposure, the distribution of voids formed after chain scission induced by electron-beam exposure, and the size of developer. This is a kind of critical fluctuation. The magnitude of roughness at the level regions also strongly supports that there are three categories in the dissolution process: molecular-level dissolution, aggregate extraction, and their intermediate.

This explanation is applicable to development with 2-butanone, which is a high-sensitivity developer for the ZEP polymer. Figure 5(a) shows AFM images of dissolution front of ZEP7000 after development with 2-butanone. It is clear that the dissolution front is also flat and smooth irrespective of the exposure dose. Since the VDW volume of 2-butanone ($82.2 \text{ \AA}^3$) is smaller than that of the critical molecular size (about $100 \text{ \AA}^3$) as shown in Table 2, 2-butanone also easily penetrates the aggregates and then dissolves them. As a result, resist polymers dissolve at the molecular level.

It is clear that the roughness behavior of ZEP7000 is basically the same as that of ZEP520 [26]. This indicates that the roughness behavior strongly depends on the developers irrespective of molecular weight. The most important point is that the development with small solvent developers such as methyl- and ethyl acetate, and 2-butanone proceeds

Figure 5: AFM images of dissolution fronts of ZEP7000 after development with *o*-dichlorobenzene (a) and 2-butanone (b). Exposure dose (*D*) and thickness loss (*TL*) are shown in each image.

at the molecular level, resulting in a dissolution front that is smooth and flat.

3.1.2. good solvents

From the results in the previous section, it is clear that the dissolution front becomes smooth and flat only for developers whose molecular size is small. These solvents have low thermodynamic compatibility with the ZEP polymer. Next, we investigated the dissolution front by using solvents with high thermodynamic compatibility, i.e., good solvents. As good solvents, four solvents were selected: anisole, *o*-dichlorobenzene, and diethylene glycol dimethyl ether (diglyme), which are used as a thinner of ZEP polymer, and 2-methoxyethyl acetate. The van der Waals (VDW) volumes of these good solvents are shown in Table 2. As clearly seen in the table, the molecular size of all good solvents is larger than the critical molecular size.

Figure 4 shows contrast curves of ZEP7000 after development with various good solvents. For comparison, the curve for ethyl acetate is also shown. The most important finding here is that the dose to clear for ethyl acetate is smaller than that for *o*-dichlorobenzene. In other words, the dissolution rate is higher for ethyl acetate than for *o*-dichlorobenzene at the same exposure doses, although *o*-dichlorobenzene is a good solvent for the ZEP polymer, whereas ethyl acetate is only a developer that cannot dissolve the ZEP polymer (Fig. 1). This is direct evidence that the penetrability of a solvent into a polymer matrix, which is strongly related to the molecular size of a developer, becomes dominant factor in dissolution rate over the thermodynamic compatibility, as the case may be. This indicates that developers, whose thermodynamic compatibility is high but whose molecular size is large, such as *o*-dichlorobenzene, penetrate gradually into a polymer matrix and then dissolve it.

Figure 5(b) shows AFM images of dissolution fronts of ZEP7000 after development with *o*-dichlorobenzene. It is clear that the dissolution fronts are also basically flat and smooth irrespective of the exposure dose, similarly to the case of ethyl acetate [Fig. 2(a)] and 2-butanone [Fig. 5(a)].

In this case, we believe that the high thermodynamic compatibility of *o*-dichlorobenzene with the ZEP polymer plays an important role in reducing the roughness of the dissolution front. Because of its high thermodynamic compatibility, *o*-dichlorobenzene cannot help but slowly penetrate the aggregates, crazing polymers, although the VDW volume of *o*-dichlorobenzene is larger than the average size of the free-volume holes in the aggregates. This is strongly supported by the lower dissolution rate for *o*-dichlorobenzene as previously explained. That is, large solvents having thermodynamic compatibility with resist polymers dissolve the polymers slowly and sequentially from the surface. As a result, the dissolution front becomes flat and smooth.

Similar dissolution behavior is expected for other good solvents. But unfortunately, we were unable to observe surface morphologies of exposed resists after development with other good solvents. This is because of the adhesion of polymers, which are precipitated during rinsing or drying. The good solvents we used dissolve unexposed polymers well. When the number of unexposed polymers dissolved in a good solvent is large, they are liable to precip-

Figure 6: Contrast curves of ZEP7000 after development with three binary-solvent developers with different volume ratios of a good to poor solvent: ethyl acetate/*n*-hexane (EA/HEX), 2-butanone/*n*-hexane (MEK/HEX), and *o*-dichlorobenzene/*n*-hexane (ODB/HEX) without rinse.

itate during rinsing or drying because the molecular weight of unexposed polymers is large. Actually, the number of adhering polymers is largest for anisole for which the number of dissolving polymers is largest as shown in Fig. 4.

From these results, it is clear that a good solvent, such as *o*-dichlorobenzene, is also effective in reducing the roughness of the dissolution front.

3.2. Binary-solvent developer

High-sensitivity developer, such as a small solvent or a good solvent with high thermodynamic compatibility, has a great advantage in reducing the roughness of the dissolution front. However, these solvents generally have a disadvantage in dissolution contrast. This is because they also dissolve unexposed regions and then the dissolution contrast decreases. The dissolution contrast actually improves by using higher molecular weight resist ZEP7000, as compared with lower molecular weight resist ZEP520, but not yet enough for fine-pattern delineation.

To obtain both higher dissolution contrast and a smooth dissolution front, we investigated binary-solvent developers consisting of a good solvent and a poor solvent. As a poor solvent, 2-propanol, or isopropyl alcohol (IPA), is typically used for diluting developer and rinsing. However, we have already found that 2-propanol causes swelling of the ZEP polymer and is therefore not adequate as a poor solvent [30]. This is because 2-propanol is a small solvent and is also compatible with the methacrylate part of the ZEP polymer. On the other hand, *n*-hexane does not swell the ZEP polymer because *n*-hexane is a relatively large molecule, whose VDW volume ($113.2 \text{ \AA}^3$) is larger than the critical molecular size. In addition, *n*-hexane is a non-polar molecule, which means that it is not compatible with ZEP polymer.

Figure 6 shows contrast curves of ZEP7000 after development with three binary-solvent developers with different volume ratios of a good to poor solvent: ethyl acetate/*n*-hexane (EA/HEX), 2-butanone/*n*-hexane (MEK/HEX), and *o*-dichlorobenzene/*n*-hexane (ODB/HEX) without rinse. As the volume ratio of poor solvent increases, the dissolution contrast increases, although the dose to clear increases.

One problem is the existence of resist residue, which was observed around the dose to clear for 3:1 EA/HEX and 3:1 MEK/HEX developers. On the other hand, there is no residue irrespective of the volume ratio for ODB/HEX developer, although the reason is not clear at present. One possibility is that some of the polymers were cross-linked by exposure and then they became less soluble in these developers. For poly (methyl methacrylate) film [31], whose imaging mechanism is the same as the ZEP polymer, it has been reported that there are three components after exposure; fragmented polymers, unfragmented

polymers, and cross-linked polymers.

These partially cross-linked polymers would become soluble in a binary-solvent developer as the exposure dose increases because the degradation process becomes dominant. This is the reason no residue was observed in the development condition for lower sensitivity, such as for both EA/HEX and MEK/HEX developers with higher volumes of poor solvent. The resist residue is no longer a problem for binary-solvent developer with a higher ratio of poor solvent. Moreover, these partially cross-linked polymers would also become soluble in good solvents, such as *o*-dichlorobenzene. Therefore, no residue was observed irrespective of the volume ratio for ODB/HEX developer.

Consequently, a binary-solvent developer is applicable to the development process of ZEP7000. Moreover, a 1:1 binary-solvent developer is optimum from the viewpoints of dissolution contrast, resist residue, and sensitivity.

Figure 7 shows AFM images of dissolution fronts after development with these three binary-solvent developers in the ratio of 1:1. It is clear that molecular-level dissolution also occurs for EA/HEX and MEK/HEX developers, although the dissolution front is slightly rough. For ODB/HEX developer, the dissolution front is apparently wavy. Figure 8 shows the relationship between the roughness and the thickness loss. The magnitude of roughness is higher than that for good solvent only. However, roughness is still small compared with the case of hexyl acetate development (Fig. 3). This indicates that binary-solvent developer is also effective for reducing the roughness of the dissolution front. Especially for EA/HEX developer, roughness is clearly reduced compared with the case of hexyl acetate developer, although the contrast curves are about the same. We can overcome the inconsistency between the roughness of the dissolution front and dissolution contrast by using binary-solvent developer consisting of good solvent and poor solvent.

4. Conclusion

We investigated the effect of developers on the roughness of the dissolution front. Surface morphology observation and roughness measurement revealed that, for a homologous series of *n*-alkyl acetate, there are three categories of dissolution process: molecular-level dissolution, aggregate extraction development, and their intermediate. Small solvent molecules, such as methyl- and ethyl acetate,

Figure 7: AFM images of dissolution fronts of ZEP7000 after development with these three binary-solvent developers in the ratio of 1:1 without rinse. Exposure dose (D) and thickness loss (TL) are shown in each image.

Figure 8: The relationship the between roughness and thickness loss for 1:1 binary-solvent developers.

are very effective for reducing roughness. For good solvents for the ZEP polymer, molecular-level dissolution also occurs and the dissolution front becomes very flat and smooth. Moreover, binary-solvent developer consisting of good solvent and *n*-hexane provides both roughness and dissolution contrast. We will evaluate line-edge roughness of resist patterns formed with these binary-solvent developers in the future.

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