EUV Resist Materials Design for 15 nm Half Pitch and Below

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Chemically amplified resist materials with a different sensitivity were prepared to investigate impact of sensitivity on resolution at 15 nm half-pitch (hp) using a EUV micro-field exposure tool (MET) at SEMATECH Berkeley. Sensitivity at least slower than 30 mJ/cm² was required to resolve 15 nm hp patterns using current EUV resists. It is noteworthy that resolution of 15 nm hp was limited by not only pattern collapse but also pinching of patterns. The same tendency is observed in E-beam patterning at 20 nm hp. A strong relationship between pinching and sensitivity in E-beam exposure indicates contribution of photon-shot noise on the pinching. Clear correlation between diffusion length and pinching using the E-beam exposure indicates that acid diffusion is another contributor on the pinching. Bound PAG into polymer and molecular PAG with a big anchor group showed almost same character on pinching. Key conclusion here is even in a molecular PAG, we can control acid diffusion to achieve 15 nm hp resolution capability. Strategy to improve sensitivity is to utilize resist with high deprotection efficiency. Polymer with a low thermal activation energy on deprotection (low Ea polymer) was demonstrated as a key technology to achieve 15 nm hp resolution with a faster sensitivity below 26 mJ/cm². Special rinse material was effective for reducing LWR by ~ 20%. Sensitivity dependency of outgassing have been systematically discussed at first. A good linear correlation between a cleanable outgassing amount and exposure energy strongly indicates tradeoff relationship between outgassing and sensitivity. Applying a new EUV topcoat to resist demonstrated reduction of outgassing from 7.39 nm to below 0.1 nm with maintaining resolution.

Keywords: EUV lithography, chemically amplified resist, low thermal activation polymer, chemical blur, photon-shot noise, topcoat

1. Introduction
EUV lithography is one of the most promising candidates for half-pitch (hp) 20 nm node device manufacturing and below [1]. The main issue for developing EUV resist is to satisfy the ITRS target of resolution, line-width roughness (LWR), sensitivity, and outgassing simultaneously [1-4]. Although partial resolution of 14 nm hp has been recently demonstrated using chemically amplifier resist (CAR) on micro-field exposure tool (MET) at SEMATECH Berkeley, its sensitivity of 30 mJ/cm² was still far from target of 5 ~ 20 mJ/cm² [5-6]. In other words, current EUV resists and processes have serious limitations as high volume manufacturing because resolution below 15 nm hp can be achieved only for slow sensitivity that is not acceptable in view point of throughput.

CAR serves as a key technology for photolithography of 20 nm hp patterning and below because of its high sensitivity and resolution [6]. Many studies have revealed that most important process in CAR is post-exposure bake (PEB) process because deprotection reaction and diffusion kinetics of acid generated by photoacid generator (PAG) directly determines resolution and sensitivity [7-12]. Therefore, tight control of
acid diffusion and deprotection reaction opens up key routes to improve resolution and sensitivity with a reasonable process window.

Suppression of pattern collapse using an ultra-hydrophobic polymer is a key technology to resolve 15 nm hp [13-14], however, all of current EUV resists, except for only one resist, didn’t show any process window on 15 nm hp pattern [6]. Detailed understanding is still lacking to achieve actual process window on 15 nm hp patterning.

The present study aims to clarify limiting factor of 15 nm hp pattern other than pattern collapse. Accordingly, we prepared resist materials in which only quencher loading is different with each other and exposed using a MET at SEMATECH Berkeley. We also investigate herein E-beam lithography performances of these and additional resists to systematically clarify a root cause of this limiting factor. Fundamental studies have been performed regarding acid diffusion length of PAGs and deprotection reaction efficiency of polymers, and their effects on resolution and sensitivity are described. Furthermore, we report herein some topics on outgassing.

2. Experimental
2.1 Materials
A series of protected co-polymers with different thermal activation energy were synthesized according to the conventional polymerization methods [15]. An organic solution containing each of polymer, PAG and organic amine as a quencher was prepared, and the resulting solution was filtered with 0.03 mm polyethylene filter for lithographic evaluation.

2.2 Lithographic evaluation using an EUV light
The resist solution was filtered and spin-coated on silicon wafer that was treated with a HMDS, and pre-baked at 100 °C for 90 sec to give a film thickness of 35 nm. The wafer was exposed with EUV light using an Exitech MET with 0.30 NA and Pseudo-PSM (PPSM) illumination at Berkeley and small-field exposure tool (SFET) at EIDEC with 0.30 NA and annular illumination (σ0.7/0.3). After the exposure, the wafer was baked (PEB) at each temperature for 90 sec, and developed with a 2.38% aqueous trimethylammonium hydroxide (TMAH) solution at 23 °C for 30 sec. The resulting wafer was analyzed by Hitachi S-9380 II SEM tool.

2.3 Lithographic evaluation using an E-beam
To determine acid diffusion length quantitatively, in-house bi-layer experimental method was applied [8]. In this method, two layers are applied on a silicon wafer: the first bottom layer (500 nm) consists of acid-labile polymer for acid detection, and the second top layer (60 nm) consists of alkaline-soluble and acid-stable polymer and PAG in order to feed acid into the first layer and catalyze acid-polymer reaction to give an alkaline solubility. The resulting wafer was exposed with E-beam to generate acid in the second layer, and baked at 90 °C for 0 sec, 10 sec, 20 sec, 40 sec, and 90 sec, respectively. After the baking, reacted polymer was developed with a 2.38 % TMAH solution for 30 sec at 23 °C, and remained film thickness was measured with optical interferometer to obtain developed film thickness (DL). Acid diffusion length (Ld) and diffusion coefficient (D) were calculated by according to the modified Fick’s equation (Equation 1).

\[ Ld = \Delta L = (2 \times D \times t_{PEB})^{1/2} \]  
(\text{Equation 1})

\[ Ld: \text{Diffusion length}, \Delta L: \text{Developed film thickness} \]
\[ D: \text{Diffusion coefficient}, t_{PEB}: \text{PEB time} \]

2.4 Acid diffusion length measurement
To determine Ea of each polymer, PEB temperature dependency of Esize was measured using an E-beam. The resist coated wafers were prepared for the analyses using the same manner described in section 2.2. Log slop of Esize and PEB temperature plots were used as Ea of each polymer in this paper.

2.5 Thermal activation energy of deprotection (Ea) determination
To determine Ea of each polymer, PEB temperature dependency of Esize was measured using an E-beam. The resist coated wafers were prepared for the analyses using the same manner described in section 2.2. Log slop of Esize and PEB temperature plots were used as Ea of each polymer in this paper.

3. Results and Discussion
3.1. Resolution limit in CAR for 15 nm hp resolution and below
The resist solution was filtered and spin-coated on silicon wafer that was treated with a hexamethylene disilazane (HMDS), and pre-baked at 100 °C for 90 sec to give a film thickness of 40 nm. The wafer was exposed with electron beam using a Ushio SOTO1 (Vacc = 57 keV). After the exposure, the wafer was baked (PEB) at each temperature for 90 sec, and developed with a 2.38% aqueous trimethylammonium hydroxide (TMAH) solution at 23 °C for 30 sec. The resulting wafer was analyzed by Hitachi S-9380 II SEM tool.
were exposed by EUV light using a MET at LBNL to get 14 nm hp to 20 nm hp patterns. In these resists, structure of polymer and PAG, and loading of PAG were kept same. Figure 1 shows the CD-SEM images of 14 nm hp to 18 nm hp patterns using a PPSM illumination with 0.30 NA. It is revealed that resolution of 15 nm hp was limited by not only pattern collapse but also pinching of patterns. Number of pinching that is observed in one SEM image with 200k magnitudes was counted by manually and summarized in Table 1 with the other lithographic performances. Figure 2 illustrates the relationship between the number of pinching and hp of line and space (L/S) patterns. It clearly shows that a narrower hp increases the number of pinching, which causes not only worse LWR but also resolution degradation. It is noteworthy that FEVS-P1507D4, which has 1.5 times slower sensitivity than FEVS-P1507D, shows extremely low number of pinching. Such difference might be originated by a photon-shot noise or high quenching ability of this resist because difference between these two resists is only amine loading, FEVS-P1507D4 has a higher loading than that of FEVS-P1507D. The sudden increase of pinching for FEVS-P1507D4 in 14 nm hp may be originated by a sudden degradation of aerial image of illumination [16] or a long acid diffusion character of this resist against this pitch. Illuminator upgrade of LBNL MET planned in this year helps to clarify which is dominant contributor on 14 nm hp resolution.

3.2. E-beam exposure results

To systematically clarify root cause of pinching on EUV exposure, five resists with various loadings of amine were prepared and exposed using an E-beam. Figure 3 shows 20 nm hp and 30 nm hp images of these resists. As decreasing Esize from 101.5 mJ/cm² to 18.1 mJ/cm², pinching was frequently observed in patterns. These images show clearly that resolution limit is changed from collapse for slow Esize to pinching for fast Esize on 20 nm hp. Figure 4(a) describes relationship between sensitivity and LWR. In this graph, we also plotted additional samples which have different PAG loadings and amine loadings with keeping the same materials. As shown in Figure 4(a), LWR was strongly dependent on sensitivity. In addition, fairly linear correlation between Esize¹/² and LWR described in the inset indicates that LWR is strongly affected by photon-shot noise [17]. Correlation between LWR and number of pinching was illustrated in Figure 4(b). A relatively good correlation suggests that the pinching is generated by photon-shot noise, in other words, fluctuation on initial distribution of acid generation. The same conclusion should be also applied to the case of EUV exposure even though contribution of photon-shot noise is a slightly different from E-beam one.

<table>
<thead>
<tr>
<th>Resist</th>
<th>FEVS-P1507D</th>
<th>FEVS-P1507D4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quencher loading (a.u.)</td>
<td>1.0</td>
<td>1.7</td>
</tr>
<tr>
<td>Pinching number (cts.)</td>
<td>45</td>
<td>3</td>
</tr>
<tr>
<td>Resolution (nm)</td>
<td>18</td>
<td>14</td>
</tr>
<tr>
<td>Esize (mJ/cm²)</td>
<td>19.5</td>
<td>30.8</td>
</tr>
<tr>
<td>LWR (nm, 3σ)</td>
<td>--</td>
<td>3.0</td>
</tr>
<tr>
<td>EL (%)</td>
<td>no EL</td>
<td>23.2</td>
</tr>
<tr>
<td>Max. DOF (nm, CD±10%)</td>
<td>no DOF</td>
<td>200</td>
</tr>
<tr>
<td>Z-factor (mJ/nm³)</td>
<td>--</td>
<td>3.7 x 10⁻⁶</td>
</tr>
</tbody>
</table>

\[ Z\text{-factor} = (\text{Resolution})^{\frac{3}{2}} \times (\text{LER})^{\frac{1}{2}} \times (\text{Sensitivity}) \] (Equation 2)
Figure 3. CD-SEM images for various kinds of resists with different amine loadings. 20 nm hp images are shown in top, and 30 nm hp images are shown in bottom. These images were taken using an E-beam (50 keV). The inset shows sensitivity of each resists.

Figure 4. (a) Relation between LWR and Esize at 30 nm hp pattern. Resist A - E are plotted as closed circle and the other resists with a different PAG loadings are plotted as open circle. The inset illustrates relation between Esize and LWR. (b) Plots of pinching number at 20 nm hp against LWR at 30 nm hp pattern.

3.3. Contribution of acid diffusion

We have previously reported that PAG with a long acid diffusion length causes severe pinching on EUV 2x nm patterns (x is 0~8), on the other hand, PAG with a short acid diffusion length drastically improves the pinching [8]. This is reasonable because long acid diffusion should cause acid image blur and hence increase deprotection on pattern. However, in the previous work, we could not distinguish two possibilities of acid diffusion and photon-shot noise because shorter acid diffusion caused 1.5 times to 2.0 times slower Esize, i.e., smaller photon-shot noise.

To determine how acid diffusion length contributes to pinching, we exposed resists with a different size of PAG including bound PAG into polymer in which Esize were adjusted by controlling amine loading. Diffusion lengths of these PAGs were determined by using the in-house bi-layer experimental method described in the reference 8. Figure 5 illustrates 20 nm hp images of these resists, and inset describes Esize and diffusion coefficient D of PAGs used in each resists. It clearly shows that shorter diffusion length causes less pinching character under the condition of same Esize. This result unambiguously supports the hypothesis that one of the major origins of pinching is acid diffusion. On the other hand, almost same character between large molecular PAG and bound PAG suggests that PAG incorporated polymer may not be necessary for at least 20 nm hp resolution.

Figure 5. E-beam patterning data for the series of resists containing PAGs with different diffusion coefficients (D).

One important question now arises, what are the major contributors on pinching in EUV? Two contributors are suggested by the E-beam data of sections 3.2. and 3.3.; one is the EUV photon-shot noise depending on sensitivity (although contribution ratio is not clear), and the other is the acid diffusion. This conclusion indicates that tight control of acid diffusion length and mitigation of photon-shot noise opens up a way to achieve practical process window on 15 nm hp. These results also suggest that bound PAG into polymer, in which acid diffusion should be shorter than molecular PAG, may not be essential technology even for 15 nm hp because FEVS-P1507D4 contains the molecular PAG though it’s a low diffusion type.
3.4. Low Ea polymer design for sensitivity

Arrhenius type plot of the E0 obtained by E-beam was illustrated in Figure 6 using our standard EUV resist. As shown in this figure, E0 was increased as lowering PEB temperature from 130 °C to 70 °C. This temperature dependency can be fitted by exponential approximation and the resulting exponential factor is used as indicator of activation energy Ea of deprotection. This Ea was converted into deprotection efficiency for the discussion and normalized based on the standard resist.

![Figure 6](image.png)

Figure 6. Arrhenius plot of E0 obtained by E-beam exposure using our standard EUV resist. The resist contains acid-labile group protected polymer and PAG that generates a sulfonic acid by irradiation of E-beam light.

Figure 7 plots normalized deprotection efficiency against E0. They are reasonably well correlated with each other. It is noteworthy that higher deprotection efficiency clearly caused faster E0. This tendency is consistent with previous reports about the deprotection reaction of typical chemically amplified resist [18]. In general, reaction-diffusion is controlled mainly by deprotection reaction in a low PEB temperature region [19]. In addition, several reports noted that difference of deprotection unit did not have a big impact on quantum yield of photo-acid generation [20]. Also, previous work indicates that EUV sensitivity is well correlated with EB sensitivity [21]. These data suggests that sensitivity is largely influenced by deprotection reactivity of polymer during PEB step.

Our hypothesis here is that low PEB temperature is necessary to suppress acid diffusion to the level that is required for 15 nm hp resolution. So, important notice from Figure 7 is that combination of low Ea polymer and low PEB temperature should be necessary to simultaneously satisfy Esize and resolution targets. In addition, low Ea polymer should have an advantage on delay stability such as post-exposure delay (PED). This may be also important character to get 15 nm hp resolution because resolution of such an ultra-narrow patterning is strongly affected by pattern profile, and this profile should be sensitive to amine contaminations in atmosphere [22].

![Figure 7](image.png)

Figure 7. Normalized deprotection efficiency versus E0 using E-beam. Deprotection efficiency was normalized based on the standard resist.

3.5. Demonstration of EUVL performances using our latest materials including process approach

Key attention is to demonstrate resolution of 15 nm hp with Esize faster than 30 mJ/cm² using low Ea new EUV resist. Table 2 compares 15 nm hp imaging data between conventional resist (FEVS-P1507D4) and low Ea EUV resist using LBNL MET with PPSM illumination (NA 0.3). It is crucial that 15 nm hp patterns are well resolved at sensitivity of 26.0 mJ/cm² with keeping LWR of 3.0 nm. In consequence with these results, Z-factor reaches $3.1 \times 10^{-9}$ mJnm³ which is best to our knowledge. However, a slight increase in pinching may come from photon-shot noise statistics.

Table 2. EUV patterning data of FEVS-P1507D4 and low Ea EUV resist: All lithographic performances were collected on hp 15 nm using a MET at LBNL PPSM illumination (NA 0.3). Z-factor was calculated using equation 2 (see reference 2).

<table>
<thead>
<tr>
<th>Resist</th>
<th>FEVS-P1507D4</th>
<th>Low Ea EUV resist</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quencher loading (a.u.)</td>
<td>1.7</td>
<td>1.7</td>
</tr>
<tr>
<td>1:1 hp Resolution (nm)</td>
<td>14</td>
<td>14</td>
</tr>
<tr>
<td>Top down SEM image at Esize of 15 nm hp</td>
<td><img src="image.png" alt="image" /></td>
<td><img src="image.png" alt="image" /></td>
</tr>
<tr>
<td>Pinching number (cts., 15 nm hp)</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Esize (mJ/cm², 15 nm hp)</td>
<td>30.8</td>
<td>26.0</td>
</tr>
<tr>
<td>LWR (nm, 3σ, 15 nm hp)</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>LWR (nm, 3σ, 15 nm hp)</td>
<td>4.0</td>
<td>4.0</td>
</tr>
<tr>
<td>Max. DOF (nm, CD&lt;; 10%)</td>
<td>200</td>
<td>no data</td>
</tr>
<tr>
<td>Z-factor (mJnm³)</td>
<td>$3.7 \times 10^{-9}$</td>
<td>$3.1 \times 10^{-9}$</td>
</tr>
</tbody>
</table>
In order to overcome photon-shot noise statistics, we focused on pattern smoothing technology during development and rinse processes. It is well known that diffusion of acid during PEB process smooths out high-frequency roughness [10]. However, acid diffusion degrades latent image contrast, so smoothing during PEB process and also in exposure process shouldn’t be effective for reducing roughness. Here, we describe how to smooth uncertainty during rinse process by using special rinse materials developed by AZ Electronic Materials Ltd. and Tokyo Electron Ltd.

Table 3 summarizes imaging data of 24 nm hp L/S pattern obtained by (a) normal DIW rinse process and (b) ~ (d) special rinse processes using AZ rinse A ~ C. In these experiments, a standard rinse recipe was simply applied and was not optimized at all to obtain better LWR. As shown in Table 3, LWR was significantly improved by up to 20% depending on type of rinse material. Most effective rinse material in this work is AZ Rinse C. Although we have not taken PSD analysis, resulting smoothen line edge strongly indicates that AZ rinse materials interact with resist patterns, and reduce high-frequency roughness taking some steps in rinse process. It is emphasized that all of rinse materials reduce microbridge, and therefore improve resolution. These effects should be expected to work even in 15 nm hp node.

Table 3. CD-SEM images of FEVS-A resist under the various kinds of rinse conditions: (a) DIW, (b) AZ rinse A, (c) AZ rinse B, (d) AZ rinse C. EUV patterning was performed using MET at LBNL with rotated dipole illumination (NA 0.3) to resolve 24 nm hp.

<table>
<thead>
<tr>
<th>Rinse</th>
<th>FEVS A</th>
<th>EVS A</th>
<th>FEVS A</th>
<th>FEVS A</th>
<th>FEVS A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top-down RMS roughness (nm)</td>
<td>0.5</td>
<td>0.6</td>
<td>0.7</td>
<td>0.8</td>
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<tr>
<td>Bottom-up RMS roughness (nm)</td>
<td>0.5</td>
<td>0.6</td>
<td>0.7</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>LWR (nm)</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Minimum LWR (nm)</td>
<td>4.2</td>
<td>4.2</td>
<td>4.2</td>
<td>4.2</td>
<td></td>
</tr>
</tbody>
</table>

3.6. Process approach for improving tradeoff between resolution and outgassing

Outgassing from resist film during EUV exposure step is a serious issue in EUV lithography because outgassing volatiles deposit on EUV mirror and hence cause loss of EUV light reflection [24]. Although outgassing has received a great deal of attention due to its impact on throughput, only a few study have been reported regarding chemistry of outgassing [21], [24-26].

Figure 8 illustrates calculated Esize for NXE:3100 (Esize) versus cleanable contamination thickness relative to our reference resist. The cleanable contamination thickness were obtained from witness-based outgassing measurement that was taken at Sematech Albany with a ROX tool. An excellently linear relationship was observed between them. It is well known that PAG decomposes under high energy radiation sources such as EUV and deep UV and causes low molecular weight volatile species of benzene and diphenyl sulfide in case of PAG containing triphenyl sulfonium [27-28]. This linear relationship in Figure 8 is therefore reasonable because increasing exposure energy enhances photodecomposition of PAG and therefore increases amounts of volatile species. This result also shows that increasing Esize to obtain smaller photon-shot noise, in other word better resolution, causes worse outgassing.

Breakthrough technology to improve both outgassing and resolution, simultaneously, is to apply topcoat onto resist surface. Topcoat was provided by Nissan Chemical Industries Ltd. for study of outgassing. This topcoat is designed to prevent outgassing from resist layer and protect out-of-band radiation, so this topcoat is named as an outgassing and out-of-band protection layer (OBPL).

Figure 8. Plots of relative cleanable contamination thickness versus Esize of NXE:3100 (calculated by using correlation between E0 of ROX and Esize of NXE:3100) . Contamination growth experiments were performed by using a ROX at Sematech.

Figure 9 (a) shows schematic illustration of stacks for measurement of outgassing: (i) resist (60 nm thickness) and (ii) resist (60 nm thickness) and OBPL-A (30 nm thickness, provided from Nissan Chemical Industries Ltd.). Figure 9 (b) shows
Figure 9. (a) Schematic illustrations of stacks for measuring outgassing with and without topcoat (OBPL-A, Nissan Chemical Ltd.): (i) resist (60 nm) and (b) resist (60 nm) and topcoat (OBPL-A, 30 nm). (b) Cleanable contamination thickness for each stacks using EUVOM at EIDEC. Dose-to-gel, indicated as D2G in figure, were 5.86 mC/cm² for “(i) resist” and 4.21 mC/cm² for “(ii) resist and topcoat”.

cleanable contamination thickness for the each stacks. These data were measured using EUVOM at EIDEC. Cleanable contamination of outgassing was obviously reduced by covering resist with OBPL-A from 7.39 nm thickness to below 0.1 nm that is detection limit of ellipsometer. Furthermore, both Esize and resolution were not degraded by applying OBPL-A as shown in Table 4. These clearly show that covering resist with topcoat is useful approach to satisfy outgassing requirements with keeping sensitivity, resolution, and LWR targets.

4. Conclusions

The present study has shown the potential factor that determines 15 nm hp resolution by using both EUV exposure and E-beam exposure tools with a high contrast. The reason why only resist with slower sensitivity than 30 mJ/cm² resolves 15 nm hp is explained by a photon-shot noise and its impact on the pinching that limits 15 nm hp resolution. On the other hand, we show here that the pinching is successfully improved by using PAGs with a controlled diffusion length. In-house acid diffusion measurement method clearly indicates both a molecular PAG and polymer bound PAG satisfy this target diffusion length. In addition, low Ea polymer design improved sensitivity by 13 % with keeping LWR and resolution. This opens up a new route to satisfy resolution and sensitivity requirement, simultaneously. Furthermore, additional process approach with special rinse can reduce LWR by ~20 %. In accordance with these results, Z-factor reaches $3.1 \times 10^{-9}$ mJnm³ even on 15 nm hp. This is the best value to our knowledge, but still need efforts to achieve ITRS target of $3.7 \times 10^{-10}$ mJnm³. EUV topcoat, which is newly developed by Nissan Chemical Ltd., drastically reduces a cleanable outgassing contamination from 7.39 nm to below 0.1 nm without major degradation on resolution and LWR. These results indicate that our materials design now provides a novel method to improve resolution with a reasonable sensitivity of 20 mJ/cm² and a practical process window for hp 15 nm and below device manufacturing.

Acknowledgement

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using a SFET and outgassing test using an EUVOM. We acknowledge to Mr. Rikimaru Sakamoto and Mr. Noriaki Fujitani and Mr. Ryuji Ohnishi (Nissan Chemical Industries, Ltd.) for providing topcoat material (OBPL-A) and their valuable comments. The authors would like to thank AZ Electronic Materials Ltd. for providing rinse materials. We appreciate Tokyo Electron Ltd. for supporting special rinse process.

References


