



In situ characterization of nano-scale pattern roughness during resist dissolution process



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ABSTRACT

Advancements in the resist process is viewed as one effective way in meeting the challenge of obtaining high quality, narrow patterns (low line edge roughness/line width roughness or LER/LWR) for leading edge lithographic technologies such as extreme ultraviolet (EUV) lithography, $\lambda = 13.5$ nm. Among these processes, resist dissolution, also referred to as resist development, is considered an important process step as this is the first instance where resist patterns are formed from resist film state. This work focuses on a fundamental approach in understanding this resist pattern formation through a visual and *in situ* characterization of the resist dissolution process. This was done using a high speed atomic force microscope (HS-AFM). Specifically, this paper presents the first successful demonstration of the quantification of resist pattern LER during resist dissolution in the typical alkali (2.38wt% tetramethyl ammonium hydroxide or TMAH)-based developer solution. Based on these results, a number of fundamental information were understood/confirmed regarding resist pattern formation behavior during dissolution; (1) Larger pattern LER can be observed at pattern side-walls nearer the line pattern surface, in comparison to those near the line bottom. Moreover, it was also understood that (2) a reduction of LER occurs in the early stages of pattern formation during dissolution. This trend in decreasing LER was also existent even at longer dissolution times, but was in smaller variations. Lastly, it was also found that (3) at longer dissolution times, partial dissolution of pattern top surface, resulting in line-pinching occurs. This in effect increases LER, especially for measurements taken in pattern side-walls near the line surface.

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1. Introduction

The fabrication of nano-scale patterns become more and more difficult as the semiconductor industry advance to more stringent pattern sizes in the sub-10 nm level [1–4]. Resist materials have played a large role in supporting these technological shifts into finer resolutions, while maintaining acceptable sensitivity and line width roughness/line edge roughness (LWR/LER). However, as pattern sizes approach the basic molecular component sizes of the resist polymers being utilized, obtaining smoother patterns (low LWR/LER) has become, and continues to be a significant issue [1]. This is especially true in extreme ultraviolet (EUV) lithography, $\lambda = 13.5$ nm, where the LWR/LER of target pattern sizes are getting more difficult to control due to e.g. patterning contrast issues related to the limited number of deliverable photons onto the resist material [5].

Resist processes (typical and alternative) are being considered to provide possible solutions [6–9]. Within the resist process, the

development or dissolution step is a process intensively researched [10–15] as it is where the first physical manifestation of such nano-scale patterns occurs.

The authors have previously reported an original and direct approach for the analysis of pattern formation during the resist dissolution process, utilizing a high speed atomic force microscope or HS-AFM (Nano Explorer or NEX by Research Institute of Biomolecule Metrology) [11]. The HS-AFM was originally developed by Prof. Ando of Kanazawa Univ. for the dynamic observation of soft biological samples in liquid [16], but was considered for this application given its high speed scanning capabilities (maximum speed: 12.5 frames s^{-1}).

With this *in situ* analysis method, the first visual evidence of nano-scale resist pattern formation (32 nm isolated line) during dissolution in aqueous tetramethylammonium hydroxide (aq. TMAH) developer solution was demonstrated [10]. Since its inception, significant technical advancements in both tool and methodology have been made. Through these improvements, the analysis of half-pitch (hp) lines and spaces (L/S) patterns, which is considered the de facto standard of measure in determining resist material lithographic performance, is also now possible [17].

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Recent research activities involving this original technique have focused on analysis possibilities from the enormous amounts of data obtained. An intensive review of acquired results up to present, point to the possibility of analyzing; the physical/chemical dynamics and quantities of resist “swelling” [17] of exposed resist film (during dissolution), pattern critical dimension variation (at “through-process” or dissolution, rinse, dry) [18], sidewall (LWR/LER) and pattern top surface roughness variation (at “through-process”), dissolution rate of exposed resist patterns, particle size analysis of resist dissolution units, etc.

This paper focuses on defining the formation of pattern roughness (e.g. LER) as it occurs during the resist dissolution process.

2. Experimental conditions/methodology

2.1. Material/sample preparation

For these experiments, the EIDEC standard resist (ESR1) [19] was utilized at 50 nm film thickness. The ESR1 is a positive-tone EUV resist composed of a hybrid polyhydroxystyrene(PHS)-methacryl polymer, onium salt type photoacid generator (PAG) and acid quencher. Optimized post application and post exposure bake (PAB and PEB) conditions were applied and dissolution was carried out using a typical aq. 2.38wt% TMAH developer.

The resist material was coated on to 300 mm ϕ silicon wafers and were exposed using an in-house EUV small field exposure tool or SFET (Numerical aperture = 0.3) which is linked to a coater/developer track system (Clean track ACT12 by Tokyo Electron) in a chemically controlled environment. The standard illumination condition used was an annular illumination of 0.7/0.3 ($\sigma_{\text{outer}}/\sigma_{\text{inner}}$). 32-nm half-pitch (hp) lines and spaces (L/S) exposed on the resist film were utilized for the dissolution analysis experiments. After patterning exposure and PEB, the target area for analysis in the silicon wafer is then cleaved to obtain the 2 mm \times 2 mm HS-AFM samples.

For these experiments, image scanning was performed over an area of 1000 \times 1000 nm (at 400 \times 400 pixels). This was done at an optimized HS-AFM scan rate of 0.5 frames s^{-1} . “Biolever fast” cantilevers (BL-AC10FS-A2 by Olympus) equipped with carbon nanofiber tips (radius of curvature: \sim 7 nm) were utilized. For the experimental procedure utilizing the HS-AFM [20], the first step is to look for target pattern, in liquid (in this case; de-ionized water or DIW). After defining the exact position of the resist pattern, the developer solution is injected continuously into the analysis area while the DIW is being dispensed in the same rate (using an auto-inject-dispense module). This allows continuous and stable scanning of the pattern surface even as the DIW is replaced with the developer solution. However, during this process step, a temporary dilution of the standard developer solution occurs. This temporary dilution of the developer allows a slower, clearer observation of the initial stages of the pattern formation process but in effect, also extends the total dissolution process time, in comparison to typical values.

All HS-AFM measurements were performed in a controlled environment inside a class 1 clean room.

2.2. LER analysis during resist dissolution

In typical HS-AFM results obtained, multiple images representing the state of the target resist film area at different times during dissolution are obtained. Fig. 1 shows an (a) example of the typical HS-AFM images (where the target pattern is located) at advancing dissolution time t , and (b) a cross-section diagram representing the line patterns in these images from which LER is extracted, at various positions in the resist pattern height (referred to as $Z_{\text{threshold}}$,

hereafter). The $Z_{\text{threshold}}$ convention applied in the discussions considers the resist line pattern's top surface at 0 nm.

These analyses were specifically focused on the left LER (at 3σ), as the right side-wall of the pattern had evidence of noise due to what is referred to hunting effect [21] (a type of oscillation occurring in systems utilizing proportional-integral-derivative or PID controllers) which occurs as scanning probe “climbs” the pattern wall. LWR is also not discussed for the same reason. The LER values shown are the average of the center 5 lines measured per image.

2.3. Limitations of methodology/scope of discussions

As described in Section 2.1, this LER measurement methodology using the HS-AFM utilizes a cantilever with a tip radius of \sim 7 nm. This tip radius size (coupled with the relatively thick film thicknesses utilized: \sim 50 nm) becomes a limitation in terms of measurable pattern size [20]. Thus at present, the minimal pattern size that can be effectively analyzed is 32-nm hp L/S. Smaller cantilever tip radiuses (e.g. Super Sharp Silicon™ by <http://www.nanosensors.com>) which can allow the analysis of smaller pattern sizes are currently being investigated for stable application with the HS-AFM. However, at this time, the 32-nm hp L/S is considered sufficient for the comparative analyses of pattern formation characteristics during resist dissolution.

Moreover, the authors recognize the possible effect of the cantilever tip conditions (tip size and shape, tangential alignment of cantilever and sample, etc.) on the quantitative values obtained. For this reason, discussions are focused not on the absolute values but more on the relative change/trends in LER formation during the dissolution process. Such relative comparison can be done with the condition that all images are obtained in one measurement cycle; where such cantilever tip conditions are assumed the same.

Lastly, in the interpretation of the results obtained here, it is noteworthy to re-emphasize that the results and discussions made are focused on the observation/measurement of LER formation “during the dissolution process”. Such values may change when the resist pattern undergoes the succeeding processes of rinse and drying [18], thus no direct comparisons were made with results obtained using scanning electron microscopy or CD-SEM (where patterns observed have gone through rinse and drying). From the point-of-view of experimental methodology, another factor that prevents a direct comparison of the measurements made by the HS-AFM versus CD-SEM is the sample size. As mentioned in Section 2.1, the samples prepared for HS-AFM analysis are 2 mm \times 2 mm. Such small sample sizes enable high speed AFM scanning. However, these sample sizes are too small for evaluations at the CD-SEM the authors have available.

It is obvious that the analysis of LER transformation as obtained during the dissolution process in comparison to the resulting patterns after drying (as observed at the CD-SEM) is itself an important topic to pursue. Such an investigation will require numerous samplings of the same resist material at various dissolution times (requiring an accurate control of dissolution time before rinsing and eventually, drying). Thus, at this time, discussions were limited to understanding the formation process of LER during dissolution.

3. Results and discussions

3.1. HS-AFM experimental results

Fig. 2 show the *in situ* pattern formation analysis results of a 32-nm hp L/S pattern exposed on the ESR1 resist during dissolution in aq. 2.38wt% TMAH developer, at various dissolution times. The upper bracket shows the two-dimensional images obtained in this analysis while the lower bracket shows the same images after

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