The NIST EUV facility for advanced photoresist qualification using the witness-sample test

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ABSTRACT

Before being used in an extreme-ultraviolet (EUV) scanner, photoresists must first be qualified to ensure that they will not excessively contaminate the scanner optics or other parts of the vacuum environment of the scanner. At the National Institute of Standards and Technology we have designed and constructed a high-throughput beamline on the Synchrotron Ultraviolet Radiation Facility (SURF III) in order to provide data on the contamination potential of the outgas products of a candidate resist by simultaneously irradiating a witness substrate and a nearby resist-coated wafer with EUV radiation, the so called witness sample test that is currently the resist qualification method required by ASML. We will present results from four sample resists that were subjected to the test.

Although the witness-sample test based on irradiating the resist with EUV radiation at 13.5 nm most closely reproduces conditions in a scanner, the availability of suitable EUV sources to conduct such tests has led to development of an alternative method which uses e-beam irradiation in place of EUV radiation. We will also present the results of a comparison of these two methods.

Keywords: extreme ultraviolet, EUV lithography, photoresist, multilayer mirror, contamination, witness-plate

1. INTRODUCTION

The materials that typically outgas from EUV-illuminated photoresists are carbon-rich and are prone to photon- or electron-induced cracking, and can subsequently contaminate EUV-illuminated optical surfaces [1]. In order to ensure cleanliness of their EUV tools, ASML has developed a protocol for qualifying photoresists to be used in their NXE EUV scanner systems [2]. The original form of the protocol incorporates a witness sample illuminated with a beam of intense UV and/or EUV radiation in the presence of a photoresist irradiated with narrow band EUV centered at 13.5 nm. The first test criterion is that the relative change in reflectivity of the witness plate after exposure of a 300 mm resist-coated wafer be less than 2%, i.e., $\Delta R/R < 2\%$. This reflectivity loss is correlated to the thickness of the carbon that is measured with spectroscopic ellipsometry (SE) using the conversion factor of 1.3 [$\Delta R/R\%$/nm] based on measurements performed by ASML and TNO. [3] The NIST facility incorporates a 200 mm wafer placed 5 cm from the witness sample. For the 200 mm wafer the $\Delta R/R$ criterion will be scaled appropriately. The second test criterion concerns the cleanability of the deposit from a

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resist-coated 300 mm wafer by atomic hydrogen cleaning. The test of cleanability as specified by ASML is that ΔR/R must be < 0.16% after a post-exposure H-atom cleaning with this specification, however, directly related to a set of atomic percentages of elemental and molecular residual contamination as measured by XPS. .

1.1 Beamline and chamber design

In order to meet the requirements of the witness sample test, the facility must meet several key criteria:

- The EUV intensity at the witness sample must be high enough so that the photo-induced carbonization rate is intensity saturated, i.e., the intensity is above the point at which the rate becomes independent of intensity;
- The resist must be uniformly exposed to a clearing dose (E_0) ±20% with 13.5 nm radiation over the entire wafer of at least 200 mm diameter;
- The background pressure must be near 10^{-9} mbar to avoid unwanted interference from background gases;

The SURF III storage ring located at NIST has a peak output just below the 13.5 nm wavelength, making it an ideal source for EUV metrology and related activities [4]. A schematic of the witness-plate beamline is shown in Figure 1. The beamline incorporates a single grazing incidence toroidal mirror placed 2.6 m from the tangent point of SURF III and images the EUV source 1.3 m further downstream with a magnification of ½. The mirror is Rh-coated and operates at an angle of 10˚ grazing. The collection mirror chamber and the sample chamber are separated by a gate valve-mounted Zr filter which blocks out long wavelength radiation and serves as a vapor barrier preventing upstreaming of resist-outgas molecules, which can damage the toroid. The resulting illumination is broadband with a spectrum from 6 nm to 20 nm. The witness sample is placed at the focus of the collection mirror where light forms a 1 mm x 2.5 mm (FWHM) near-Gaussian spot with a peak intensity of approximately 50 mW/mm^2.

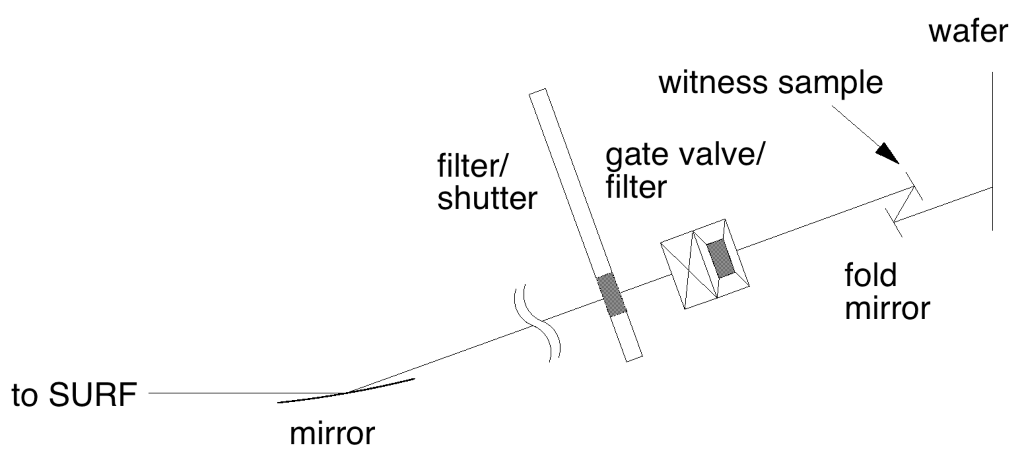

Figure 1: Layout of the optical path in the resist testing beamline,

The testing chamber is equipped with two calibrated photodiodes, one to measure the power incident on the witness sample and the other to measure the power at the resist. Both photodiodes are remotely retractable so as to allow monitoring of the power at any time during an exposure. To compensate for the synchrotron beam decay during the test, the algorithm that controls the speed of the wafer translation is adjusted based on beam current to maintain the proper exposure level.

The witness sample is a Ru-capped MoSi multilayer aligned at 10˚ angle of incidence, which relays a narrow-band reflection onto a fold mirror that also operates at 10˚ angle of incidence. The fold mirror sends the EUV light on to a 200 mm diameter photoresist-coated silicon wafer which is rotated and translated in order to uniformly expose the photoresist to a clearing dose (E_0). Under normal operating conditions a photoresist with a 5 mJ/cm^2 dose-to-clear can be exposed in about 20 minutes. The sample chamber is equipped with a null-field ellipsometric imaging system (NEIS) [5] that provides real-time monitoring of carbon growth during witness-plate testing. Real time images can be used to provide a real time growth rate estimate that can be calibrated by post exposure *ex-situ* SE or XPS measurements, and these estimates can then be used to check on the appropriate scaling factor to extrapolate the results to a 300 mm wafer. The beamline incorporates an integral glove box to allow for witness-sample and wafer exchange in a N_2 purge environment to minimize pumpdown time and contamination of the photoresist and/or the sample chamber. The sample chamber has a base vacuum pressure of $<10^{-9}$ mbar and can typically be ready for operation within 12 hours after a sample exchange.

Processing of the photoresist including spin coating and developing are done at the Center for Nanoscience and Technology (CNST) cleanroom facility located on the NIST main campus. Post-exposure analysis facilities including 2D spectrally-resolved ellipsometry, XPS and EUV reflectometry [6] are also available on the NIST campus.

1.2 E_0 determination

The testing facility can be used for a determination of E_0 as the first step in the process. The algorithm that controls the dose on the wafer controls the translation speed of the and the rotation speed of the wafer in such a way that each point on the wafer is 5x oversampled by the EUV spot and in such a way that each point at a given radius can be given a selected dose. Thus an E_0 measurement can be made by choosing the algorithm to sweep through a range of doses as a function of radius that incorporate the expected value and then measuring the actual radius for the onset of clearing after the development of the resist. Figure 2 presents an example of just such a determination.

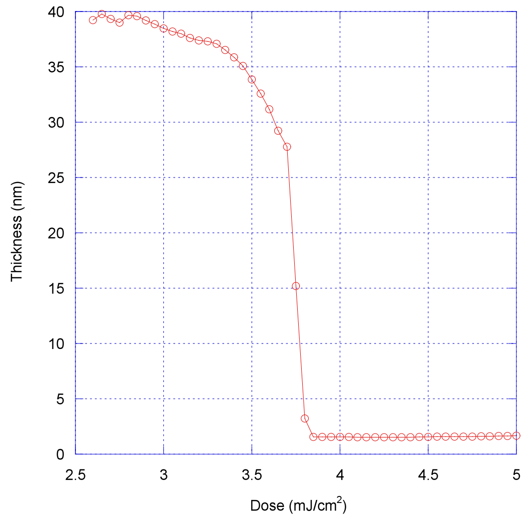

Figure 2. Developed resist thickness vs. dose.

1.3 EUV photoresist witness sample results

Figure 3 shows the results of tests on four resists labeled A- D. The thickness determinations were all done with SE *ex situ*. The data indicates a factor of three range from the least contaminating resist (D) to the most contaminating (B). Reproducibility of the results is acceptable for the testing to qualify resists.

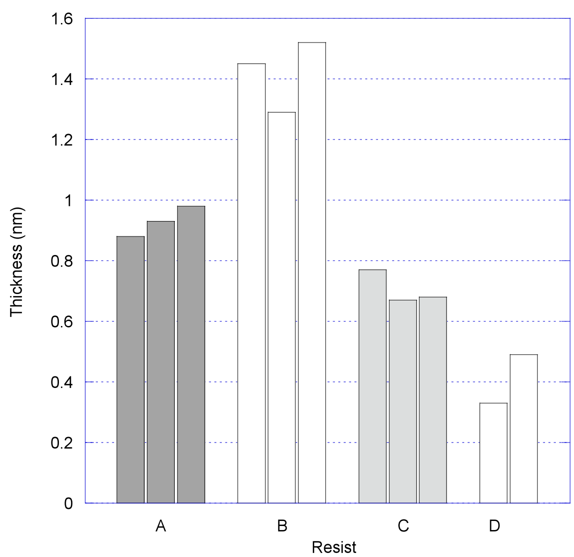

Figure 3. Thickness of carbon spots for EUV exposures of four resists (A, B, C and D) determined with spectroscopic ellipsometry using a Cauchy model fit for deposited carbon for dielectric constant of the film. The numbers indicate sequential number of a run for a given resist.

Figure 4 shows SE thickness profiles on resists A, B, and C. Since the EUV spot on the witness sample is Gaussian in shape, the flat top seen in the central region of the thickness profiles is indicative of a region in the beam that is photon-saturated. This photon saturation was observed for all four of the resists tested in the commissioning of the facility and is a necessary condition of the qualification protocol.

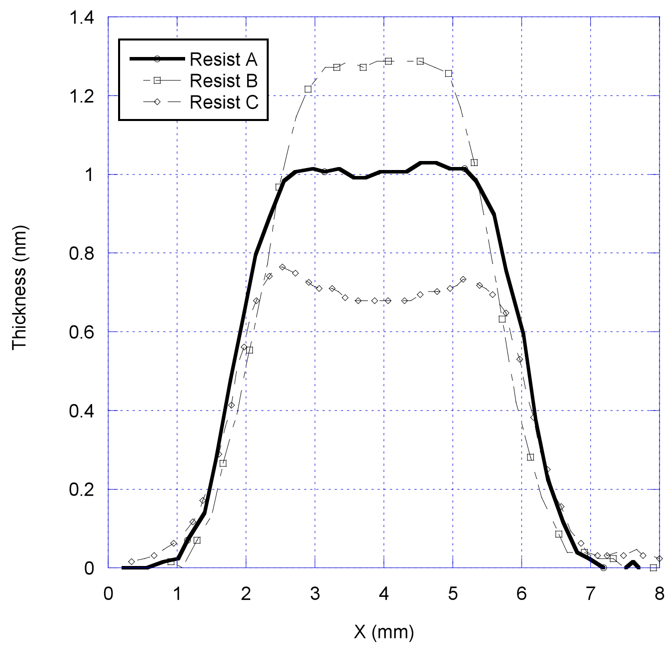

Figure 4. Distribution of carbon thickness across EUV exposure profile as determined with spectroscopic ellipsometry for resists A, B, and C

1.4 Post-exposure H-atom cleaning

As mentioned, the second criterion in the witness sample test requires that the deposition be cleanable with atomic hydrogen to a level such that $\Delta R/R < 0.16\%$ as determined by ASML through atomic percentage measurements with XPS. NIST is presently exploring the use of a H-atom filament source as well as several different plasma sources.

2. ELECTRON-INITIATED WITNESS PLATE TESTS

Testing based strictly on photons requires a high intensity source as used in a scanner, which is cost prohibitive, or a synchrotron, which is an option only available to a few institutions like NIST. Since outgassing in resist can be caused by secondary electrons from either photons or electrons, ASML investigated doing resist outgas qualification for EUV using electrons instead of photons. By building a research test set up, ASML determined the required test details for electron based testing, and demonstrated correlation to early work on photon-induced contamination.

2.1 E-beam witness-plate test facility

The ASML resist outgas testing set up, shown in Figure 5 is based on electron exposure of a 300 mm wafer and a 25 mm diameter Si wafer covered with a nominal 50 nm of Ru in an ultra-clean vacuum environment. Two e-guns are used, one for wafer exposure with a nominal 20 mm diameter beam and one for witness sample exposure with a nominal 2.5 mm beam. Operating current and voltage of each e-gun is set such that the witness sample intensity ''bakes in'' all contamination and at the wafer, current density can be adjusted to match the resist supplier recommended E_0.

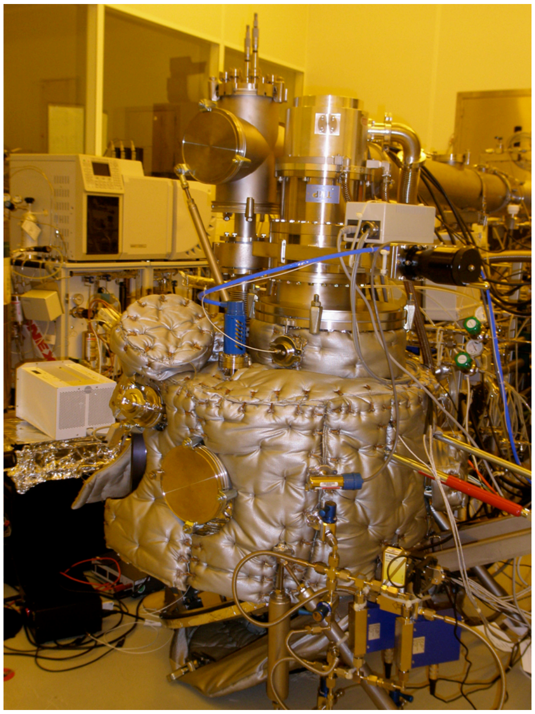

Figure 5: E-gun based research resist outgas tester at ASML

Although early correlation of electron based contamination from outgassing to photon based outgassing was demonstrated, ASML wanted more rigorous confirmation made of the photon correlation to ensure that electron based qualification of NXE resist would be acceptable. Based on testing four resists different from those in the earlier work, Figure 6 shows the correlation of electron and photon induced contamination, confirming the correlation, and that it is a linear relationship.

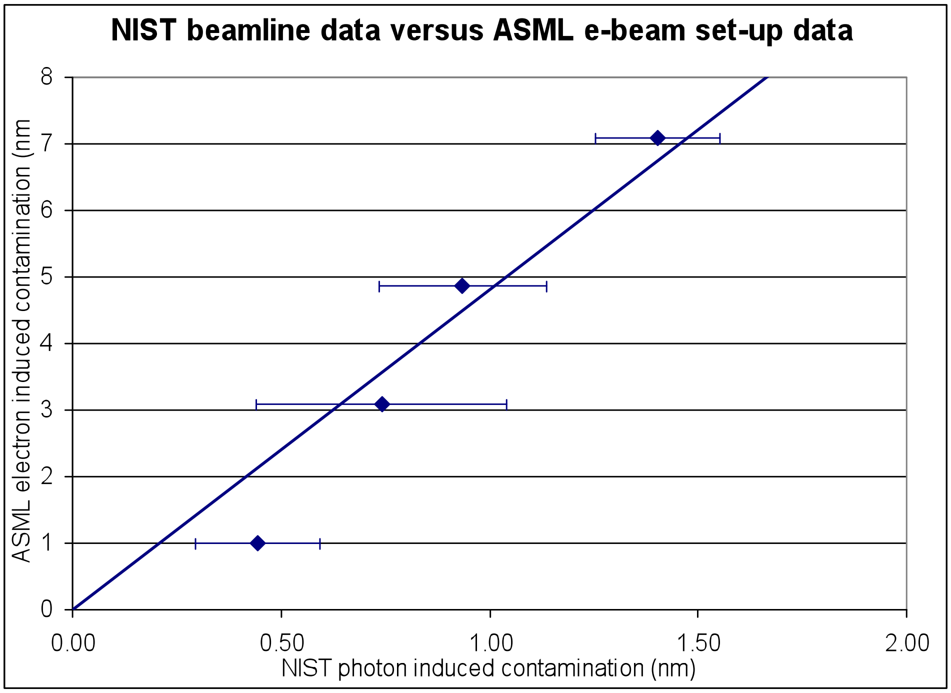

Figure 7 Correlation of ASML facility's electron-beam-induced and NIST facility's photon-induced resist outgas contamination.

Conclusions

The work summarized in this paper is critical for several reasons. It confirms the linear correlation of electron induced resist outgas contamination to photon induced resist outgas contamination, which means that resist outgas qualification can be done with electrons. Also shown is the correlation of contamination film thickness to change in reflectivity.

NIST has a unique photon based test capability, tied to critical SE metrology and reflectometry that will enable them to qualify resists for scanner use.

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