# **EUVL dosimetry at NIST**

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## ABSTRACT

As part of its role in providing radiometric standards in support of industry, the National Institute of Standards and Technology has been active in advancing extreme ultraviolet dosimetry on various fronts. Recently, we undertook a major effort in accurately measuring the sensitivity of three extreme ultraviolet photoresists. It has been common practice to use photoresists as a transfer "standards" to determine the intensity and uniformity of the radiation transmitted by extreme ultraviolet steppers. In response to preliminary results from Lawrence Berkeley National Laboratory that showed that two "standard" photoresists were almost twice as sensitive as had been previously believed, NIST carried out similar measurements and confirmed the Berkeley results. However, we have found that the assumed sensitivities are more a question of system calibration than of absolute resist dose sensitivity. We will describe the facility we used to make these measurements.

Photoresists make less than perfect radiometers. They are very non-linear, sensitive to atmosphere, and difficult to calibrate. All of these characteristics led to the disparate results in assumed sensitivity values. We have developed an alternate wafer-plane dosimeter based on image plates. The dosimeter is linear over several orders of magnitude, comparatively insensitive to atmosphere, and can be re-calibrated as necessary. Moreover it can pass through a stepper as any other wafer. We will describe this dosimeter in detail.

Keywords: radiometry, dosimetry, photoresists, image plates

## **1. INTRODUCTION**

Process control is essential to any production tool. In a lithography stepper many processes need to be controlled simultaneously. These include focus, alignment, and controlling the dose delivered to the wafer plane. Currently the tools operating at 193 nm wavelength use beam splitters near the wafer to control dose in real time.<sup>1</sup> However in the extreme ultraviolet (EUV) this is not practical both because of the expense of EUV beamsplitters and because the light sources are so expensive that even a small loss in system transmittance is not tolerable.

Both the intensity of the radiation and the distribution must be measured. There are several possible methods or tools available to do this. Calibrated EUV-sensitive photodiodes are available,<sup>2</sup> however they are single-channel. An aperture can be placed in front of the photodiode, which can then be scanned, but this is time-consuming and would also involve installing additional mechanical stages and electrical connections in an already crowded vacuum system. EUV-sensitive charge-coupled devices have been available commercially for some time, however the packages are large and require several electrical connections. EUV-sensitive film is no longer available and is non-linear and can only be used once, and variations in emulsion thickness and other characteristics affecting the sensitivity cannot be calibrated out.

Thus the community has chosen to use photoresist as the wafer plane dosimeter. A reference wafer passes through the stepper just as any other wafer, and the facilities are equipped to process these wafers and measurement of the resist thickness is fully automated. On the other hand there are some drawbacks to using resist as a detector. Chemically amplified resists are very sensitive to environmental factors such as humidity and trace chemicals such as amines. Moreover, resist is very non-linear, that is, the residual thickness is not linear with dose, so open-frame exposures may not reveal non-uniformities in the field. Finally, and most importantly, resist sensitivity is a very difficult measurement to make correctly. The measurements by Lawrence Berkeley National Laboratory<sup>3</sup> (LBNL) revealed that a resist used as a reference to determine doses was in fact twice as sensitive ast had been believed for many years. We have recently

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confirmed these measurements as well as confirming that two other resists used as references had the previously believed sensitivities.

We will describe the apparatus used for the resist sensitivity measurements and present the results of these measurements. We will also present a novel idea for a wafer-plane dosimeter based on storage phosphor imaging plates.

## 2. RESIST MEASUREMENTS

## 2.1 Experimental setup

To make the resist sensitivity ( $E_0$ ) measurements we designed a new endstation for Beamline 1 (ref. 4) of the Synchrotron Ultraviolet Radiation Facility<sup>5</sup> (SURF III) storage ring. The front-end of the beamline consists of a single Mo/Si multilayer mirror of 75 mm diameter and 1.5 m radius of curvature. The mirror accepts about 16.6 mrad of the SURF III emission horizontally and the full extent vertically. The radiation comes to a focus about 90 cm from the mirror and begins expanding again. The light passes through a freestanding 250 nm Zr filter 20 cm downstream from the focus. The calculated spectrum of the incident radiation based on the calculated output spectrum of SURF III and the measured reflectivity of the mirror and transmittance of the filter has about 90% of the radiation in a 2 nm band about 13.3 nm.

The resist-coated wafer is mounted on a rotary feedthrough 42 cm downstream from the filter. Just upstream from the wafer is a pair of apertures. One aperture is about 5 mm from the wafer and defines the extent of the exposure spot; the second is 15 mm upstream from this one and displaced 12.7 mm vertically. A calibrated photodiode sits behind the second aperture. These apertures are nominally identical, 2.5 mm in diameter and have 45° bevels to reduce scatter. They are mounted on a bellows-sealed linear feedthrough, so the incident power can be measured with the second aperture in the beam, then the first aperture is placed in the beam for the wafer exposure. A diagram of the  $E_0$  testing measurement apparatus is shown in fig. 1. A shutter mounted on a rotary feedthrough (upstream of what is shown in the figure) controls the exposure time and blocks the beam during motion. A measurement run produces up to 30 exposure spots on a single wafer, each with a unique known dose.

The first step in an  $E_0$  measurement is to measure the distribution of the radiation and to verify that the uniformity is adequate to make an accurate measurement. To do this we mounted a scintillator screen at the wafer plane on the inside of a vacuum window. The focusing mirror was tweaked to obtain the optimum uniformity and intensity. Figure 2 shows a picture obtained with a CCD camera as well as horizontal and vertical lineouts. The scale was obtained by taking a picture of a 0.5-mm grid mounted on the inner part of the window adjacent to the scintillator. The EUV spot is 2.65 mm in diameter, and the uniformity is better than +/- 2 % in the central 2 mm. The diameter of the exposed spot in the photoresist was subsequently measured using a stylus profilometer and visible-light microscope and found to be 2.65 mm +/- 0.05 mm.



Figure 1: Diagram of experimental apparatus for E<sub>0</sub> measurement at SURF III.

The next step is a cross-calibration of the two apertures. A second calibrated photodiode was placed at the wafer position. The signals of the wafer-plane and monitor photodiodes were measured alternately at several values of SURF III beam current. The correlation between the two is good, with the power at the wafer plane 6.6 %+/-1.0 % higher than the power measured by the monitor diode.

Samples were prepared, developed and analyzed at the NIST NanoFab facility. Because chemically amplified resists are so sensitive to environmental conditions, we have equipped the endstation with a glove bag, which we evacuate and fill with dry nitrogen while changing samples. The





Figure 2. Exposure beam profile. The 2D image was measured with a CCD camera, and the two lines are horizontal and vertical lineouts.

samples are transported to and from the NanoFab in a hermetically sealed container which ensures no contamination during transit. Two or three samples were spin coated and post-apply-baked on a contact hotplate in the early morning for that day's exposures. Exposures typically took 30

Thickness (nm)

min, and the delay from the last exposures typically took 50 min, and the delay from the last exposure to postexposure bake was typically 20 min. The exposed wafers were immersed and developed in 0.26 N TMAH solution for 45 s. Thickness measurements were made with a Nanometrics Nanospec<sup>\*</sup> in the middle of each exposure spot.

#### 2.2 Experimental Results

In order to expose a wafer, first a broad range of desired doses is determined, then exposure times are calculated based on the measured relationship between intensity and the current measured by the monitor diode. Before the series of exposures is made all valves are opened with the shutter closed. The shutter is opened for the determined time, then the wafer, which is mounted on a rotary stage with its axis 25 mm from the incident beam spot, is rotated to the next spot. The first set of measurements were made on TOK EUVR-P1123\* resist of 65 nm thickness. The first set of exposures spanned the dose range from



Figure 3. Thickness as a function of dose for three measurements.

<sup>\*</sup> Product names are mentioned only for experimental completeness. The mention of specific companies or products does not constitute endorsement by NIST nor by the Federal Government.

about 2 mJ/cm<sup>2</sup> to 20 mJ/cm<sup>2</sup>. Subsequent exposures were made over narrower ranges. Figure 3 shows the results of three sets of measurements. The threshold of the thickness measurement our white-light reflectometer can make is 9 nm, so the value of  $E_0$  is adjusted upward by 0.25 mJ/cm<sup>2</sup> from the dose at 9 nm thickness (this was subsequently validated by measurements on a more sensitive instrument). Our average value for the four measurements is 5.9 mJ/cm<sup>2</sup>, which compares favorably with the value of 5.8 mJ/cm<sup>2</sup> found by Naulleau *et al.* in ref. 3.

The uncertainty in our measurement is dominated by determination of zero thickness, at 10 %, and wafer processing, also 10 %. Other components contributing are photodiode calibration and cross-calibration of the two apertures, both 1 %, and determination of the area of the spot, 3 %. The root-sum-square uncertainty including all components is 15 %. All uncertainties are quoted as standard uncertainties with coverage factor k=2.

Given the NIST and LBNL results on the TOK resist, a question remained as to whether all resists or EUVL tools needed an adjustment to their dose sensitivities. To address this we have measured two additional resists that have been used as references in ASML Alpha Demo Tools (ADT). ASML has used XP4502J\* as a reference for several years, assuming an  $E_0$  value of 2.0 mJ/cm<sup>2</sup>. These measurements were made in the same manner as the previous resist. The results from our lab as well as the ASML Alpha Demo Tools and the Intel Micro Exposure Tool are presented in Figure 4. In all cases the overlap is well within the 15 % uncertainties of the measurements.

# 3. A NEW WAFER-PLANE DOSIMETER

## 3.1 Background

The varied results and uncertainties in using resist as a dose reference have led us to investigate alternative wafer-plane-dosimetry schemes. Ideally the dosimeter should be linear over the range of doses expected, reuseable many times, calibratable as needed, and convenient for those in the industry to use - it should pass through the stepper in the same manner as a wafer and should yield results in a convenient amount of time. A calibrated photodiode would seem ideal, except that in order to make two-dimensional measurements, a small aperture needs to be placed in front of the diode, and it needs to be scanned, leading to unacceptably long acquisition times. A charge-coupled device is intrinsically two-dimensional, but is too bulky for wafer-plane use. EUV-sensitive film is no longer available, plus was non-linear and susceptible to variations in response due to non-uniform emulsion.



Figure 4. NIST E0 results along with results from two ASML alpha demo tools (ADT1, ADT2) and the Intel MET with new calibration based on LBNL results.

Imaging plates are two-dimensional detectors that use a storage phosphor to store a latent image. The storage phosphor is commonly a halide such as  $BaF_2$ , which is doped with a rare earth such as Eu. An incident x-ray or EUV photon creates a charge cloud after it is absorbed, and some of the electrons in the charge cloud are trapped into meta-stable halide vacancies in the crystal lattice. These states have lifetimes on the order of hours to days. The plates are read out by scanning a red laser, which excites the electrons into the conduction band. Those electrons that fall back into the valence band give off blue photons, which are detected by a photomultiplier tube behind a blue filter. These imaging plates are linear over several orders of magnitude incident dose.

Imaging plates are used almost exclusively in the life sciences. The most common type has a polymer backing, storage phosphor layer, and a front protective coating. The total package is a few hundred µm thick. The protective coating is

typically >10  $\mu$ m thick polymer coating such as polyethylene terephthalate (PET).<sup>6</sup> This thickness is sufficient to block all of the incident EUV radiation as well as particles. However, there are uncoated imaging plates available, which are used mostly for tritium detection. These are also EUV sensitive, however they saturate at doses of order 1  $\mu$ J/cm<sup>2</sup>. Presumably, an EUVL dosimeter would need to be sensitive in the range expected for EUV resists, around 10 mJ/cm<sup>2</sup>.

A custom imaging plate can be constructed that is appropriate for EUVL wafer-planedosimetry applications. This would require attenuating the incident radiation by about four orders of magnitude. And since the package should be as close as possible to the thickness of a wafer, the attenuating layer should be part of the plate itself, meaning that it must be transparent to both the incident red and emitted blue photons during the readout process. A low-density transparent polymer would be ideal. We have calculated the necessary thickness, and depending on the polymer and the attenuation required, thicknesses in the 1 µm to 3 µm range are suitable. We have contacted several vendors about the feasibility of depositing uniform coatings on the plates, and some have indicated that this is possible.

3.2 Experimental Results



Figure 5. Transmittance results from 600 nm polycarbonate (solid line) and three thicknesses of polyimide noted in the legend.

We have obtained candidate free-standing films of polyimide and polycarbonate. Figure 5 shows the transmittance of three thicknesses of polyimide and a 600 nm film of polycarbonate. It is apparent from these measurements that film thickness of about 2 µm is necessary to achieve the four orders of magnitude attenuation we seek. To simulate this we have inserted the 1555 nm polyimide film in our incident beam to expose uncoated image plates. We use the measured transmittance at 13.5 nm, photocurrent measured from our normalization photodiode, and controlled exposure time to expose the plates to different doses. In a typical run we expose between five and 22 spots. Figure 6 shows an image of three spots obtained during one run. It is apparent that our beam is non-uniform, having an approximately gaussian shape in the vertical dimension.

Since we measure the total number of photons incident on the plate, some processing is necessary to obtain information about saturation levels. First we find the number of counts in the entire spot to find the number of counts per unit energy. This must be done in a region well away from saturation, where the response is linear. Then a small spot, in our case about 0.4 mm by 0.4 mm, where the beam is fairly uniform, is selected to find the response as a function of dose.



Figure 6. A close-up showing the image plate exposed to three different EUV fluences.

Figure 7 shows the response of the image plate/filter combination as a function of dose in mJ/cm<sup>2</sup> for one image plate/reader combination. It is evident that saturation occurs at about 5 mJ/cm<sup>2</sup>; using a 2000 nm film would increase this by about an order of magnitude, bringing the saturation level into the desired region. A second image plate-reader combination from a different vendor has shown a higher saturation level, however this combination displayed some noise at lower doses.

One vendor has indicated a willingness and ability to coat tritium-sensitive image plates with a variety of polymers. A second vendor has provided us with plates with thin polymer overcoats. Preliminary results from these plates are positive, however the noise level is somewhat higher than is necessary for a wafer-plane dosimeter.



Figure 7. Image plate counts vs. dose. The image plate was exposed behind a 1555 nm polyimide film.

# 4. SUMMARY

We have measured the dose to clear of three photoresists that are used as references in the industry. We've found that one resist is almost twice as sensitive as had previously been believed and thus impacted a research tool dose calibration, and confirmed that two others are correct. These results point to the desirability for a wafer-plane dosimeter that is linear, insensitive to atmospheric conditions, and can be re-calibrated as needed. We propose that image plates with thin polymer coatings are appropriate for this application. We have measured the saturation levels of some image plates attenuated with polymer films and initial results are encouraging. One image plate in particular shows excellent linearity and signal-to-noise. A 2 um polyimide coating applied to this image plate should yield a saturation level of 50 mJ/cm<sup>2</sup>, which is likely appropriate for EUVL wafer-plane-dosimetry applications.

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