Daniel L. Rokusek held his Science Undergraduate Laboratory Internship at Argonne National Laboratory in Argonne, IL during the summer before his senior year at the University of Illinois at Urbana-Champaign. His work was a continuation of the previous summer's work in ANL's Student Research Participation program. Under the direction of Ahmed Hassanein and Jean Paul Allain, Dan investigated the erosion of candidate mirror materials for use in extreme-ultraviolet lithography devices. This work was presented at the AAAS Annual Meeting in Washington D.C., and received first place in the Technology & Engineering category of the student poster competition. Since his time at ANL, Dan received a B.S. in nuclear engineering and a minor in mathematics. He is ready to begin his Ph.D. work in nuclear engineering at the Massachusetts Institute of Technology in the fall of 2005. While at MIT, Dan plans to study plasma-material interactions with applications in fusion science and plasma technology. After he leaves MIT, Dan plans to remain in academia and aspires to one day become an educator.

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Ahmed Hassanein is Senior Nuclear Engineer at Argonne National Laboratory. Has five engineering and physics degrees including a Ph.D. (1982) and two Masters from the university of Wisconsin, Madison. Internationally recognized as one of the world's foremost lead persons in the area of modeling material response to different radiation sources. Has developed unique models and comprehensive computer package to predict materials behavior, lifetime issues, and fluid hydrodynamics under various irradiation conditions. Created the PRIME "Particle Radiation Interaction with Material Experiments" facilities at Argonne. These facilities now conduct research for DOE fusion program, Intel, SEMATECH, Philips, ASML, and others. Dr. Hassanein is author of more than 270 journal publications and technical reports in heat transfer, thermal hydraulics, radiation damage, hydrodynamics, particle diffusion and transport, atomic physics, and photon and radiation transport. He presented numerous invited talks and chaired international conferences and workshops as well as keynote speaker to universities and world-class institutions.

EROSION STUDIES OF EUVL CANDIDATE COLLECTOR MIRROR MATERIALS IN THE IMPACT EXPERIMENT

Daniel L. Rokusek, Jean P. Allain, Ahmed Hassanein, and Martin Nieto

ABSTRACT

The IMPACT (Interaction of Materials with charged Particles And Components Testing) experiment at Argonne National Laboratory was used to expose Pd, Ru, and Re-capped Ru candidate EUV light collector mirror materials to conditions similar to extreme-ultraviolet (EUV) lithography source devices, in particular high-energy singly-charged Xe ions. Experiments measured both the time-dependent atomic surface concentration evolution of candidate single-layer mirror (SLM) samples and the Xe^+-induced sputtering yield. Elemental surface information was acquired using low-energy ion scattering spectroscopy (LEISS) and sputtering yields were acquired using an in-situ quartz crystal microbalance. Sputtering results show large erosion rates between 0.5 and up to 7.0 for Pd and Ru SLM samples for energies between 500 and 1000 eV of Xe^+ irradiation at grazing incidence. Re-capped Ru SLM samples also demonstrated very high sputter yields. Time-dependent erosion rate measurements used with LEISS resulted in a high depth-resolution profile and led to the discovery of ion-induced recoil implantation of oxygen atoms to the Ru mirror surface. High concentration of oxygen throughout the Ru SLM may be detrimental to the reflectivity response of the collector mirror.

INTRODUCTION

Deep-ultraviolet lithography (DUVL), the industry's standard for manufacturing microprocessors, is projected to reach its limits in 2009. DUVL is limited by the minimum wavelength of light, 193-nm, that can be used to etch circuits on silicon wafers. Thus, a new technique is needed to create smaller microprocessors with more transistors. Extreme-ultraviolet lithography (EUVL), using a wavelength of 13.5-nm, is a process undergoing intense research and one of the leading candidates for emerging lithography techniques of next generation microprocessors [1,2]. To generate EUV light, hot, dense plasmas are required [3]. Two main configurations are used:

gas-discharge-produced plasmas (DPPs) and laser produced plasmas (LPPs). Both configurations require the collection of EUV light at the first condenser optics to an intermediate focus downstream from the high-intensity plasma. Figure 1 shows schematically both the DPP and LPP configurations showing the use of grazing incidence collector mirrors and near-normal incidence mirrors, respectively. Scaling to higher EUV powers hinders the application of EUV light for high-volume manufacturing lithography in the near future due to lifetime limits on critical components in EUV sources, in particular the plasma-facing collector mirror. Lifetime is currently defined as loss of EUV photon reflectivity of about 10% after operation with about 10^{11} shots or about 2.5 years.

In LPP EUVL sources, EUV light is collected at near normal incidence with respect to the mirror surface [3] compared to DPP sources, at grazing incidence. Multilayer mirrors (MLMs) with dissimilar EUV optical constants made of Si/Mo with a period of about $\lambda/2$ are used as LPP collector optics [4]. The conventional EUV light fuel used in both configurations is currently Xe, although Sn is also under consideration [5]. The high-intensity plasma pinch produces fast ions and neutrals that bombard and erode nearby components, including the collector optics. The collector optics surface will also be exposed to off-band radiation (outside 13.5-nm) inducing heat, debris (i.e., electrode material in DPP devices), highly charged ions, and background impurities (i.e., H, C, N, O). This paper studies in particular the interaction of intense, energetic Xe singly-charged ions with single-layer (grazing incidence) mirror materials including: palladium, ruthenium, and rhenium.

The IMPACT (Interaction of Materials with charged Particles And Components Testing) experiment at the Argonne National Laboratory studies the interaction of highly energetic and intense particles with various candidate mirror materials for EUVL, such as Pd and Ru. IMPACT features a well-collimated ion source to conduct erosion studies of the candidate mirror materials [6]. The source is capable of producing ions with energies between 50 and 5000 eV, fluxes ranging from 10^{11} to 10^{17} ions/cm^2/s, and incident angles ranging from 0 to 60-degrees with respect to normal. Temperature dependent studies up to 1000 ºC can also be performed, as the system includes an in-situ heating element to vary sample temperature. Another feature of IMPACT is the capability of performing in-situ low-energy ion scattering spectroscopy (LEISS) to facilitate the characterization of multi-component surfaces as they erode.

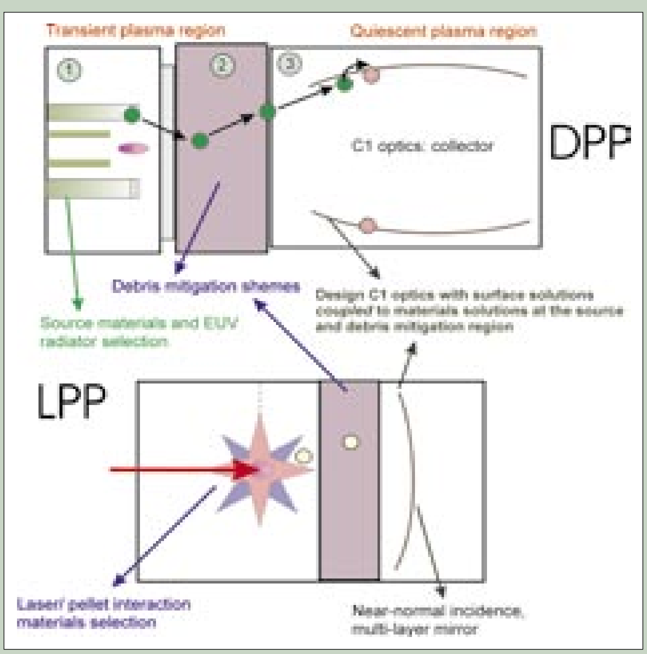


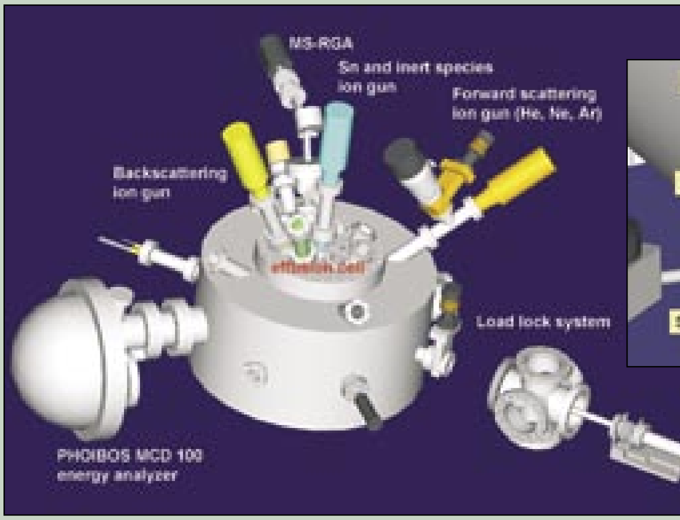
Figure 1. Schematic describing competing candidate configurations to generate EUV light in EUV lithography sources and their respective components. In discharge produced plasma (DPP) sources, grazing incidence collectors are used; whilst in laser produced plasma (LPP) devices near-normal incidence collector mirrors are applied.

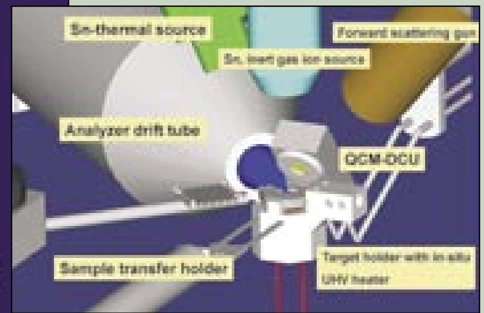
MATERIALS AND METHODS

The samples used in IMPACT are single-layer mirrors (SLMs) of palladium, ruthenium, and rhenium were fabricated at the Materials Research Laboratory at the University of Illinois at Urbana-Champaign. SLMs were grown on SiO_2 substrates, with the thin films deposited via magnetron sputtering. The composition of each sample consists of a thin film of the candidate mirror material (10 – 50-nm) on top of a 5-nm layer of Ti on top of the SiO_2 substrate. This Ti layer is deposited to minimize silicide formation of the top-most layer. Identification of samples follows the form of Xy-Z, where Xy is the element of the topmost thin film and Z is the sample number (assigned according to samples original position on a 6-in SiO_2 wafer).

Samples, generally 1-cm^2, are transferred in and out of the IMPACT chamber via a custom designed transfer-lock system. While isolated and vented, the transfer-lock chamber is opened and a sample is placed on two forks that are fixed to the end of a linear feedthrough. Once the sample is in place, the chamber is sealed and pumped down to the pressure of the main chamber (IMPACT experiments are conducted in an ultra-high vacuum (UHV) environment). A typical base pressure of 10^{-8} Torr can be achieved through the use of a magnetically-levitated turbo-molecular pump backed by a scroll pump. When an acceptable pressure is reached, a valve is opened to allow the transportation of the sample from the transfer-lock into the main chamber. Using the linear feedthrough, the sample is transferred from its position on the forks onto a boron-nitride sample holder in the main chamber. This sample holder is fixed to a rotatable, linear motion feedthrough. The axis of rotation of the feedthrough lies in the plane of the sample's surface, allowing angle-of-incidence measurements to be conducted. The linear manipulation of the sample allows for moving the sample in and out of the ion beam path.

IMPACT uses a differentially-pumped Model 1401 Ion Gun, commercially manufactured by Nonsequitur Technologies, Inc. (NTI) Beams of inert gas ions are generated by means of electron impact ionization; gases used include Xe, He, Ne, and Ar. Beams are characterized by four factors: gas species, beam energy, beam current, and spot size. The first two are relatively easy to obtain, however beam current and spot size require the use of optics contained inside of the ion gun. Varying these optical settings will focus and/or condense the beam, with an overall effect of changing beam current and beam diameter, allowing full control of the particle





Figures 2(a) and 2(b). Diagram of the IMPACT experiment. Note that the section between the two chambers has been removed to illustrate the path of the transfer arm. Also note that additional devices are shown, namely two additional 1401 ion guns and an effusion cell. Figure 2(b) shows internally location of the target holder with respect to the ion sources, the PHOIBOS drift tube and the QCM-DCU system used for sputtering measurements.

flux level reaching the sample. A Faraday Cup is used to measure the beam current. The Faraday Cup is a device that allows for direct measurement of collected ions by having a picoammeter in series with ground. The beam's diameter is also measured with the Faraday Cup, coupled with beam profiling software. The software serves as a virtual instrument (VI), programmed with National Instrument's LabVIEW software package. The VI rasters the beam across five pinholes inside the Faraday Cup to create a spatial beam profile using the NTI ion source's octopole lenses. Profiles are cataloged for future reference and use. Once the desired beam current and diameter are obtained, the sample is positioned under the beam for irradiation. Figure 2(a) shows a schematic for both the overall IMPACT geometry and multiple ion sources along with figure 2(b) showing the internal components of the experiment. Multiple ion sources are used for LEISS and irradiation of sample SLMs.

Erosion experiments collect sputtered target atoms from the mirror sample induced by the incident, highly-intense energetic Xe (or other) ions. IMPACT uses a quartz crystal microbalance – dual crystal unit (QCM-DCU) diagnostic system for in-situ real-time erosion measurements and is shown in figure 2(b). The quartz crystal microbalance technique is a powerful, well-developed diagnostic metrology tool measuring mass loss from materials irradiated by the incident ions [7]. It can measure monolayer-level erosion of surfaces irradiated by charged-particle bombardment. This technique utilizes one crystal oscillator to measure the deposition of sputtered particles, while the other crystal oscillator is shielded from the sputtering flux and measures the background ambient only. The measured frequency difference between the two crystals (proportional to the mass of material sputtered onto the unshielded crystal) is then correlated to the amount of eroded material from the sample. Total sputtering data is extracted from the measured frequency difference using some graphical software and is used to calculate sputtering rates. Points of inflection represent changes in the total sputtering rate due to changes in surface interfaces, compounds, and ion-induced mixing. The sputtering yield is calculated from the reduced data, which is then used to calculate sputtering rates.

Beam irradiation time is determined on an individual experiment basis. The convention used to determine exposure time was based on the desire to deliver a common dose of irradiation for each trial in a set of experiments. The LEISS technique is used with multiple NTI ions sources in forward and backscattering modes with a PHOIBOS 100-MCD hemispherical energy analyzer. The LEISS technique is one of several in-situ metrology techniques in IMPACT to study the sample surface under particle bombardment. In addition, sputter rates measured with the QCM-DCU are used in conjunction with LEISS for in-situ depth profiling.

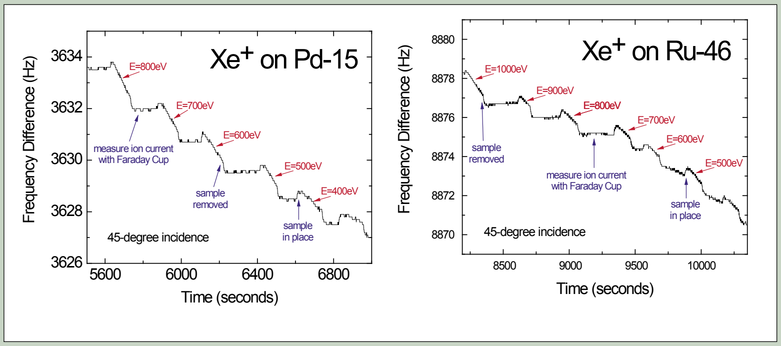
RESULTS

Figures 3(a) and 3(b) show the frequency difference on the two QCM crystals during Xe^+ bombardment of Pd-15 and Ru-46 samples, respectively, irradiated at 45-degrees incidence and $2.6\text{x}10^{14}$ $cm^{-2}s^{-1}$ flux at room temperature. Both samples were irradiated varying incident Xe^+ ion energies to study the effect of ion energy on sputtering yield. The curve in each figure was generated by inserting the sample under the beam and irradiating, then removing the sample to measure the beam current. After each irradiation, the beam energy was decreased and the sample was re-inserted under the beam and irradiated again. The flat sections seen in figures 3(a) and 3(b) correspond to no sputtering during the time the sample is removed from the beam while the beam current was measured with the Faraday Cup. Each drop in frequency measured corresponds to a particular incident Xe^+ energy, and is used to obtain the energy-dependent sputtering yield. These sputtering yields are plotted in figures 4(a) and 4(b) for Pd-15 and Ru-46 respectively. These figures show the behavior of sputtering yield as a function of energy. The sputtering yields were calculated using the following equation [7]:

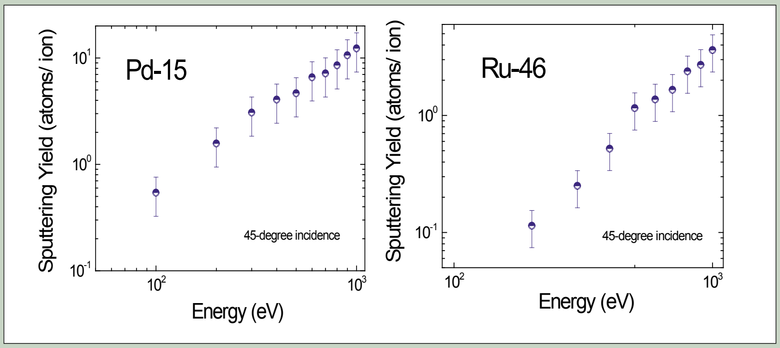
$$Y = \frac{1}{Df_i S^{QCM} \Omega m_{avg}} \frac{\Delta f}{f} M_{crystal} \quad (1)$$

Here, D represents total ion dose, S^{QCM} represents a sticking coefficient, $M_{crystal}$ represents the mass of the crystal (provided by manufacturer), Δf represents the change in frequency during sputtering, f represents background after sputtering, Ω represents the collected fraction, m_{avg} represents the mass of the deposited species, and f_i represents the dose correction factor for secondary electrons and sputtered ion current.

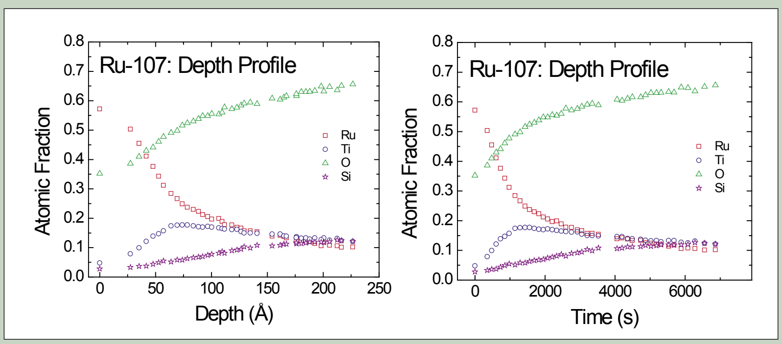
Figure 5(a) shows the surface composition evolution for the Ru-107 sample obtained by the LEISS technique, using 1000 eV



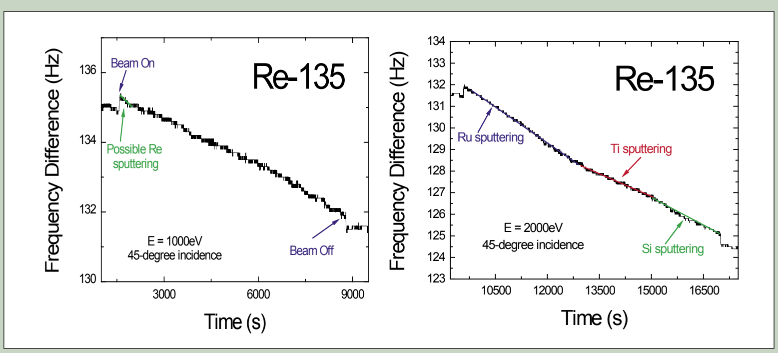
Figures 3(a) and 3(b). Frequency difference measurements from QCM-DCU diagnostic in IMPACT for Xe^+ bombardment of Pd-15 and Ru-46, respectively, at 45-degree incidence for varying energies.



Figures 4(a) and 4(b). Sputtering yield of Pd-15 and Ru-46, respectively, on 5-nm of Ti/SiO_2 bombarded by Xe^+ at 45-degree incidence for various energies.



Figures 5(a) and 5(b). Evolution of atomic fraction of species in Ru-107 over time and depth, respectively. Note that the profile on the right is a true depth profile.



Figures 6(a) and 6(b). Frequency difference measurements from QCM-DCU diagnostic in IMPACT for Xe+ bombardment of Re-135, at 45-degree incidence for 1000 and 2000 eV cases, respectively. Note in figure 6(a) that there may be a small region of Re sputtering. Note the high-flux sputtering in figure 6(b) shows the different layers of materials in the sample.

He^+ as the probing ions at normal incidence and room temperature. The flux of He ions was varied so that during LEISS data collection it was minimized and then increased to sputter material, thus resulting in a profile through the SLM depth. The architecture of the Ru-107 SLM was 10-nm Ru / 5-nm Ti / Si substrate. This sample was irradiated for approximately 120 minutes. The species observed in figure 5(a) correspond to Ru, Ti, Si, and O surface atom fractions measured with LEISS as a function of depth. The depth profile illustrated in figure 5(b) was obtained by multiplying the time evolution of the peaks by the sputtering rate as a function of time. The transformation of figure 5(a) to figure 5(b) required smoothing the QCM sputtering data with a Fast Fourier Transform (FFT) filter to eliminate intrinsic noise. The smoothed curve was then fit with a third-degree polynomial, which was subtracted from the original signal. The difference was fit with a cosine function, which was then removed from the original signal to obtain the true signal. The resulting curve was quite linear, with a slope corresponding to the total change in frequency of the two QCM crystals due to the sputtering. This value was incorporated into a constant, along with equation (1), giving the following for sputtering rate [8]:

$$\frac{\partial x}{\partial t} = \frac{K}{\sum y_i \rho_i} \qquad (2)$$

where K represents a constant involving current and yield values, and y_i and ρ_i represent the atomic fraction (intensity) and mass density for i=Ru,Ti,Si,O. Now, to obtain depth as the unit of measurement of the x-axis, time is simply divided by sputtering rate, that is, [s]/[Å/s]=[Å]. Knowledge of the energy corresponding to each target mass and the cross section σ for the corresponding projectile-target pair allows the calculation of surface composition of the sample. Suppose that during an energy scan with the detector, N number of peaks each with area A_i, are observed. The atomic fraction of each component y_i can be calculated by:

$$y_i = \frac{A_i \sigma_i^{-1}}{\sum_{k=1}^{N} A_k \sigma_k^{-1}} \qquad (3)$$

Therefore, the surface atomic fraction can be determined from knowledge of the elastic scattering cross section and use of the LEISS intensity spectra [9].

Re is another EUV collector mirror candidate along with Pd and Ru. Figure 6(a) shows data for 1000 eV Xe^+ bombardment of Re-135, irradiated at 45-degrees incidence and $3.3x10^{13}$ $cm^{-2}s^{-1}$ flux at room temperature. The architecture of the Re-135 SLM sample is Re/Ru/Ti/SiO_2, with layer thicknesses of 2-nm / 10-nm / 5-nm / substrate, respectively. The data shows immediate and rapid sputtering of the Re layer, represented by the first downward sloping segment in the figure during the first three minutes of irradiation. At the end of this region there exists a point of inflection representing the end of the Re layer and the beginning of the Ru layer. The bombarding flux was predetermined, based on a calculation for a fixed sputtering time of five minutes.

To illustrate the different regions of the sample, the same experiment was performed at new location on Re-135, this time with 2000 eV Xe^+ ions. The results of this experiment are shown in figure 6(b). Notice three distinct regions, corresponding to the Ru, Ti, and Si layers. Also note that no Re layer is observed, as the Xe^+ flux is too large for such a thin layer to be detected by the frequency difference measured on the QCM-DCU in the short time interval.

Figure 7 shows LEISS data for Re-133 obtained with a 1000 eV He^+ beam. The architecture of the Re-133 SLM sample is the same as that of Re-135. Here, Re-133 was bombarded with 1000 eV He^+ ions at normal incidence and room temperature. The scans do not distinguish the Ru peak from the Re peak since they are very close in energy (868.8 eV and 902.9 eV, respectively). Since the use of He^+ ions as scattering particles limits the identification between Ru and Re atoms, Ne^+ ions were chosen as scattering particles to achieve better mass resolution due to higher energy coupling between incident and target masses.

Figure 8 shows a LEISS scan of Re-135. This scan was performed using 1000 eV Ne^+ ions incident on Re-135 at normal incidence and room temperature. The peak observed on the left at E = 608 eV corresponds to Ru, while the peak on the right at E = 749 eV corresponds to Re. Ne^+ ions were used because they are heavier than He^+ ions and therefore better resolve the two peaks based on the principles of LEISS [9], as evident in the figure. The disadvantage with using Ne^+ beams, however, is that impurities such as oxygen and carbon (i.e. M = 16 or 12 amu) cannot be detected.

DISCUSSION AND CONCLUSIONS

In order for a candidate mirror material to be used in a EUV-light generation device, its behavior while being irradiated must be understood and potential degradation mechanisms mitigated. Because different energy particles can exist in practical applications, it was necessary to study the sputtering yields of candidates Pd, Ru, and Re with variation in incident particle energy. The plots shown in figures 4(a) and 4(b) illustrate such behavior for energies ranging from 100 to 1000 eV. The data shows that as incident particle energy increases, the sputtering yield also increases. Figures 3(a) and 3(b) show that the higher energy cases have a larger change in frequency vs. time than that of the lower energies. This correlates to larger sputter rates and calculated sputter yields presented in figures 4(a) and 4(b). These sputtering yield measurements have two important results. The first is that at energies greater than 500 eV, Ru will sputter more than Pd-based collector mirrors; therefore any fast ions generated from the creation of EUV light will have to be mitigated if Ru mirrors are used. The second finding is that for either Pd or Ru, 1000 eV particles cannot be tolerated since both will have over unity sputter yields induced by Xe^+ bombardment. Recall, Xe is used as a light generator for EUV light in plasma-based EUVL sources.

Figures 5(a) and 5(b) graphically illustrate the composition of the Ru-107 SLM sample as a function of time. The ability to profile a sample with LEISS while measuring the sputter rate in-situ is quite powerful, as individual monolayers of a sample can be examined. In addition, knowing how the surface composition changes in time will allow for design decisions of EUVL collector mirrors as they are irradiated by Xe^+ particles. In the case of Ru-107, the known composition was verified, however mixing between layers was observed. Such mixing was assumed to occur, however the level of oxygen at the surface was higher than anticipated. The time behavior of oxygen suggests that oxygen is present throughout the sample. As oxygen is diffuses from the SiO_2 substrate, it mixes with the other metals and oxides are formed. In addition, ion-induced recoil implantation of oxygen atoms to the surface is also found. This is an important result and the first time this phenomenon has been discovered for an SLM collector mirror. Such oxygen segregation could be detrimental to EUV reflectivity and degrade the collector mirror.

The curve in figure 6(a) shows the possibility of Re sputtering in the first four minutes. It is difficult to conclude whether there is a point of inflection suggesting the transition from Re to Ru. Because the slope is not linear, it is concluded that the layer is not purely Re. Rather, Re and Ru mixing has occurred as sputtering

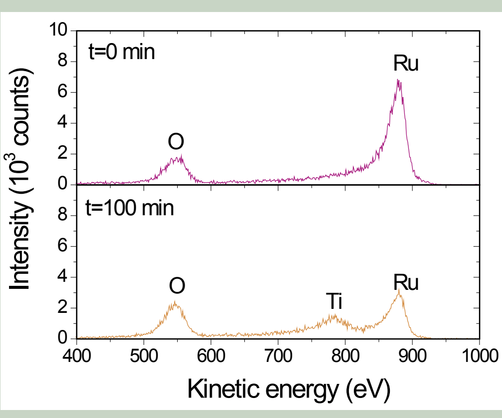


Figure 7. Intensity of species of Re-133 for increasing time. Note that this plot shows intensity, and not atomic fraction. Because Re is not observed in the profile, atomic fractions cannot be assigned to the Ru, Ti, and O species.

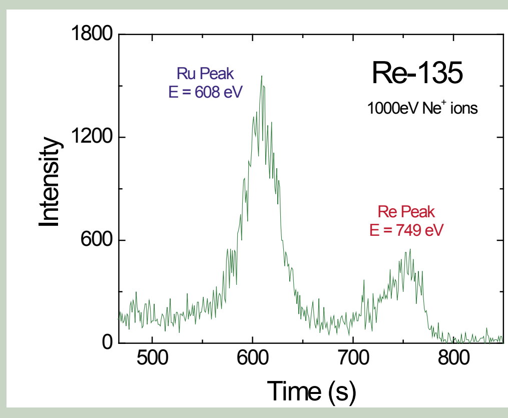


Figure 8. LEISS scan of 1000 eV Ne^+ bombardment of Re-135. Note that the Ru and Re peaks are now clearly resolved.

of the two components gives the non-linear slope. Even with the low-flux Xe^+ beam, the Re layer was eroded too quickly to obtain useful data, and therefore it is concluded that the Re layer appears to be too thin to be studied at the incident flux level used. In order to obtain meaningful data, future experiments should be performed on a sample that has a thicker Re layer and/or performed at much lower ion flux levels. This result does however point to the propensity for Re to erode under Xe^+ bombardment. Therefore, collector mirrors designed with Re cap layers should be avoided.

Another important result is the time evolution of the Re-capped SLM. The time scans shown in figure 7 of Re-133 show the evolution of various species on the sample surface. It was known that the mirror sample's architecture was Re/Ru/Ti/Si. The scans show a large Ru peak, an ever-present O peak, and eventually a Ti peak. It is not explicitly obvious that Re is present. The main result here is that oxygen again is found to preferentially segregate to the surface. This seems to be a dominant mechanism for Ru-based mirrors and thus may make Ru a poor candidate EUV collector mirror material.

The Re peak was not clear in the LEISS spectra, and this is probably due to the closeness of energy of the Re and Ru peaks. To support this claim, it is noted that 1000eV He^+ ions in an LEISS scan will resolve a Re peak at E = 902.9 eV and a Ru peak at E=868.9 eV. The difference in energy of these two peaks is only 34 eV equivalent to the full-width-at-half-maximum of the scattered peak, and therefore it is concluded that the larger Ru peak is effectively hiding the Re peak. To acquire data with better mass resolution, Ne^+ ions were used. The heavier ions will profile the surface more accurately than the He^+ ions, however because Ne^+ ions are heavier than oxygen the O peak will not be seen. Figure 8 shows the case for LEISS spectra using a Ne^+ beam. Note the good separation in energy peaks for both Re and Ru. Thus the ability for IMPACT to use a variety of source gases allows flexibility and versatility for identification of various candidate EUV mirror materials whose masses may not be that much different.

In summary, Pd, Ru and Re-capped Ru candidate EUVL collector mirror materials have been tested in IMPACT under various bombardment conditions. All experiments using relatively high-energy Xe^+ ions led to large erosion levels of candidate EUVL collector mirror materials (i.e. Pd, Ru and Re). Therefore, high energy Xe ions must be avoided and mitigated. Oxygen segregation induced by ion-bombardment and oxygen diffusion from SiO_2 interface regions present another challenge to collector mirrors, especially Re and Ru-based systems, which have a high affinity for oxygen. Therefore, selection of a collector mirror with low oxygen affinity, low erosion yields and high EUV reflectivity is preferred.

ACKNOWLEDGEMENTS

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