III. X - RAY MICRO - LITHOGRAPHY AND THICK RESIST STUDIES AT CAMD

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CAMD (Center for Advanced Microstructures and Devices) utilizes the radiation from a 1.2-1.5 GeV electron storage ring with a critical-wavelength range 9.5-4.8Å. The storage ring at CAMD provides suitable radiation for X-ray micro-lithography and microfabrication work. High resolution, thin and thick resist processing techniques, including resist application, exposure and development were explored. New techniques such as transfer mask and equivalent filtration are introduced and demonstrated. Issues of latent image formation in exposed resist are addressed. Examples of structures fabricated using described techniques are presented.

Key words: *x-ray lithography, high-aspect ratio lithography, microfabrication, thick resist, x-ray mask, transfer mask, x-ray exposure optimization, radiation effects, latent image formation, x-ray scanner, micromachining*

1. INTRODUCTION

The J. Bennett Johnston, Sr., Center for Advanced Microstructures and Devices, CAMD, is based on a 1.2-1.5 GeV electron storage ring operating as a

dedicated synchrotron-radiation source [1-3] (photon critical-energy range, 1.3-2.6keV or, critical wavelength, 9.5-4.8Å). The scientific and engineering activities at the Center include spectro-analytical and

Fig. 2. X-Ray Micro-Lithography Exposure Ssystem XRLM3

X-ray micro-lithography studies supporting spectro-chemical and microfabrication applications [4]. The layout of the CAMD facility is presented in Fig.1. The CAMD synchrotron facility was designed to meet strict requirements of vibrational and environmental control [5].

The X-ray micro-lithography and microfabrication facility includes beamlines and exposure stations, 230m² of class 100 clean rooms, metrology equipment, thin film and resist (thin and thick) processing equipment [4,6,7] with a special emphasis on development and implementation of dry processes. The processing capabilities include resist application and development, metal film formation by sputtering, electron-beam evaporation, and micro-electroplating techniques. The operational regime of the storage ring and radiation filtration in the beamlines provide a wide choice of exposure-radiation characteristics at CAMD with photon energies ranging from 1 to 7 keV. The beamlines XRLC1, XRLM1, XRLM2 and XRLM3 are used for X-ray micro-lithography and microfabrication and have an exposure footprint up to 50 mm wide and 5 to 10 mm high. The linear power density in horizontal direction is constant (ranging from 0.2 to ~1 W/cm for different beamlines), and scanning the target in the vertical direction with a constant speed results in a homogeneous exposure field of 50 by 50mm². A schematic of an X-ray micro-lithography beamline system[8] (XRLM3) is presented in Fig.2. The beamline is located on Port 7B, has a ~5 mrad collection angle, a wavelength range of $1.5\text{\AA} \leq \lambda \leq 6\text{\AA}$, and terminates with a 125 μm thick Be window. The beamline is configured to provide 1.0 W/cm at 1.5 GeV and 100 ma storage ring operation. The end station is a multi-chamber scanning CAMD X-ray Exposure Tool[8,9].

2. CAMD X-RAY EXPOSURE TOOL.

Dry processes, and especially induced chemistry, such as plasma or radiation assisted etching and deposition, *in-situ* or self-development are becoming increasingly important. A new X-ray micro-lithography exposure tool [8,9] (Fig.3.) has been designed and installed on the XRLM3 beamline. The exposure tool is able to utilize synchrotron radiation for X-ray imaging (exposure) and radiation assisted processes. The system can provide reactive ion etching (RIE) and X-ray activation and exposure

Fig. 3. CAMD X-ray Exposure Tool for Micromachining

simultaneously. It consists of a multi-chamber X-ray Scanner. The X-ray exposure tool is designed to control the exposure conditions and can handle a variety of mask/sample assemblies. The first chamber is designated as a radiation filter and controls X-ray spectra and dose delivered to the sample using foils and inert gases. The second chamber, the exposure and reaction chamber, is equipped with a multi-axes scanning mechanism to provide desired orientation and uniform exposure of the mask/sample assembly. The exposure and filter chambers can be separated, when necessary, by a Kapton window to facilitate the

use of reactive atmospheres for induced chemical processes during exposure or dry development procedure. Two agents, X-ray irradiation and RF (13.53 MHz) induced plasma (RIE), can be used separately or in combination.

3. X-RAY EXPOSURE OPTIMIZATION.

Fig.4. Equivalent filtration by films and gases

To provide adequate conditions for the PMMA development process the ratio of the top to bottom absorbed dose value of $Y \leq 5$ has been recommended[10]. To optimize absorbed dose distribution through the resist thickness we introduce[8,9] a concept of equivalent filtration: the radiation spectrum passed through a film of a certain thickness t_0 is modified in a such a way that the recommended value of $Y \leq 5$ holds in next layer of the same thickness t_0 . For radiation with the photon energy away from the X-ray absorption edges, any material of a thickness t_1 can be used for equivalent filtration by employing a scaling factor $K = t_1 / t_0 = \mu_0 / \mu_1$, where μ_0 and μ_1 are the linear absorption coefficients. Based on the scaling factor, one finds the thickness of an “equivalent” filter $t_1 = K \cdot t_0 = t_0 \cdot (\mu_0 / \mu_1)$ (Fig.4.). Using PMMA as a resist, factors K for equivalent thicknesses of solids used as filters are: Beryllium $K_{Be} = 4$; Aluminum $K_{Al} = 0.071$; Kapton $K_{Kapton} = 0.86$; Silicon $K_{Si} = 0.083$. For example, when exposing a 500 μm thick PMMA sample, these coefficients will yield filter thicknesses of 2000 μm Be, 36 μm Al, 430 μm Kapton, and 42 μm Si. “Equivalent” gas pressures can be introduced in a similar way (Fig.4.) taking into account radiation path length through the gas [8,9].

4. RESIST APPLICATION TECHNIQUES.

As thicker imaging layers are required, to meet the needs of high aspect ratio lithography in micromechanics, the integrity of the substrate/resist system is more and more determined by the resist thickness. The most known method to apply a thick PMMA layer on a substrate is the traditional LIGA approach - forming a resist sheet on the substrate by

Table 1.

Thick PMMA Resist Application Techniques					
Thickness range	Resist material	Resist availability	Number of coatings	Coating thickness	Application Method
μm				μm	
5 - 10	9% solution	commercial	3 - 5	2 - 3	spin-on & bake @ 180°C
5 - 15	20% solution	in-house	1	5 - 15	spin-on & bake @ 150°C
10 - 30	20% solution	in-house	3 - 4	10 - 15	spin-on & bake @ 110°C
30 - 80	30% solution	in-house	1-2	25 - 80	spin-on & bake @ 110°C
75	solid sheets	custom	2	first coating is a 2 μm thick PMMA spin-on film, the second layer is a solvent bonded solid PMMA sheet of appropriate thickness	
125	of	custom	2		
250	appropriate	custom	2		
500 and up	thickness	commercial	2		

casting.[10] This method is always accompanied by relatively high stress at the resist/substrate interface. The stresses can be high enough to cause the resist to crack and even separate from the substrate. Another well known method is based on milling thick PMMA sheets solvent-bonded to a substrate.

We use alternative resist application techniques [11]. One of these is a modified, multiple coating spin-on method suitable for PMMA resist thicknesses up to 60-80 μ m. The other method is based on stacking PMMA layers to achieve the desired resist thickness. Using a combination of spinning and bonding, a wide variety of resist thickness can produced with a limited set of commercially available PMMA sheets. This technique produces PMMA resists with thickness from 75 μ m and up (see Table 1.). Stacked PMMA resist samples were fabricated using a number of different solvents (MMA monomer, chlorobenzene, chloroform, etc.). The adhesion strength and stability of the stacked PMMA resists was evaluated using thermal shock and mechanical cleaving tests.

Fig.5. "Plexiglas® Boxes" pattern

An example of 500 μ m thick PMMA resist attached to a Si wafer, exposed through a wire mesh mask with 2-4 Å X-ray radiation, and developed in a mixture[12] of 20 vol. % tetrahydro-1,4-oxazine, 5vol.% 2-aminoethanol-1, 60vol.% 2-(2-butoxy-ethoxy) ethanol and water is shown in Fig.5. The developed pattern is 500 μ m tall and has 15 μ m thick walls.

5. CONVENTIONAL X-RAY MASKS.

A conventional X-ray mask used in IC X-ray lithography and micromachining consists of a handling and protective frame, a pattern carrier, reasonably transparent to X-rays, and a pattern

absorber, opaque to the X-rays. The handling frame, typically a ring, determines the dimensional envelope of a mask and is an object of standardization. The importance of standardization is recognized by the lithography community , and today two X-ray mask standards are recommended: IBM and NIST standards [13,14]. Both standards are based on a mask structure developed by IBM: a ring with an extended "lip" for mounting the X-ray mask in the exposure tool (see Fig.6.). The dimensions for the IBM standard are: wafer and upper ring diameter D_w =100mm, lip-ring diameter D_L =120mm, overall ring thickness T =10 mm, lip thickness L =3mm. The NIST standard offers the following dimensions: D_w =100 mm, D_L =125mm, T =7mm, L =3mm.

Fig.6. Typical x-ray Lithography Mask

The pattern carrier provides dimensional pattern stability[15,16] and is typically a thin, low-Z material membrane, which has to be stable against radiation

Fig.7. A spiral produced in 250 μ m thick PMMA resist

degradation[16-18]. Materials such as Si membranes (2 to 10 μm thick)[13] and Be foils (300 μm)[10] offer sufficient pattern stability and are radiation resistant. Several other materials such as silicon carbide and silicon nitride also seem to be finding application as carriers. High-Z materials, like electroplated gold, or etched tungsten (in exploratory stage), with thicknesses ranging from 2 to 15 μm , are used as pattern absorbers. The choice of absorber thickness can be based on the ratio of the dose absorbed at the resist bottom in the open area to the dose absorbed at the top of the resist under the absorber [19]. For reliable processing of thick resists this ratio has to be about 10. For example, for 350 μm thick PMMA resist one would require a 13 μm Al filter and a 4 μm thick gold absorber; and for 1000 μm thick PMMA one will use a 45 μm thick Al filter and the 7.5 μm thick gold absorber.

A simple one-level resist process[19] is used at CAMD for conventional X-ray mask fabrication. This process produces 4 μm to 10 μm thick gold absorbers by transferring optical patterns. Commercially available substrates (B-doped Si membrane, 2-3 μm thick) in an IBM standard on a Pyrex glass ring were used. A patterned X-ray mask with 4 μm thick gold test pattern for micromachining is shown in Fig.6. A 250 μm thick PMMA resist pattern of a spiral, printed using a mask of this format, is depicted in Fig.7.

6. TRANSFER MASK TECHNIQUE.

A new technique suitable for the high aspect ratio

Fig.8. Multiple exposure with transfer mask

microstructure fabrication has recently been developed and established at CAMD. This transfer mask technique [19-21] is based on forming an absorber

Fig.9. PMMA pattern of a cylindrical resonator 280 μm tall

pattern directly on the surface of the resist or material of the sample to be patterned. Allowing stepwise exposure (see Fig. 8) this method can be used with soft X-rays or UV. Both, additive and subtractive methods are suitable for transfer mask formation.

For X-ray exposures, the soft ($\sim 10\text{\AA}$) X-ray lithography beamline (XRLC1) optimized for IC fabrication was used. The patterning of the PMMA resist with the transfer mask was performed in several steps [21]. Each step, a combination of exposure ($\sim 10\text{J}/\text{cm}^2$) and subsequent development (5 min) removed ~ 30 μm of PMMA. The results of these experiments are presented in Fig.9. The finest features present in Fig.9. are about 2 μm wide (gap between

Fig.10. A "bridge" produced in PMMA (transfer mask method, tilt during exposure $\pm 5^\circ$)

the cylinder and pick-up electrodes). This corresponds to aspect ratio of greater than 100:1. Using the transfer mask technique, with 4 μ m thick gold absorber, and exposing on a hard X-ray beamline, a set of holes 125 μ m in diameter were produced in 1500 μ m thick Plexiglas® sheet. New lithography techniques, such as in-situ development, radiation assisted chemistry, can be realized by using the transfer mask method.

Forming the transfer mask directly on the sample surface creates new opportunities: exposure of samples with curved or dynamically deformed surfaces. Free standing features can be easily formed by tilting the sample. Fig.10. shows a “bridge” in PMMA resist produced by tilting the sample for $\pm 5^\circ$ during multiple exposures.

7. RADIATION EFFECTS AND PMMA RESIST STUDY.

The understanding of the behavior of materials and devices in an ionizing radiation field is very important, especially in view of the development and implementation of new technologies and techniques, such as X-ray lithography and X-ray micromachining (LIGA), etc. It proved to be very productive to present the radiation effects as a multi-stage radiolysis[17,18]. The results of radiation effect studies are finding

very thick (up to several cm) resists are required. The experimental study of changes in irradiated PMMA include observation of latent image formation, determination of the depth of degradation using development techniques, measuring radiation swelling[23] and thickness losses [8,9,23] after irradiation. The latent image was observed on a cross-section of exposed PMMA resist sheet [23]. An SEM image of a typical cross-section of PMMA resist is presented in Fig.11. The latent image was formed by an X-ray exposure through a metal mesh mask (500 μ m period). The formed image is >1mm deep. Exposed areas of the resist are bright, while the unexposed regions are seen as dark vertical stripes which correspond to the mask pattern. These stripes can be detected as deep as 600 μ m for a top dose of 2.7 kJ/cm³, and as deep as 1400 μ m in samples exposed with 10 kJ/cm³. Within the exposed area two regions can be clearly identified: a top region which has a “shell-like” break pattern, typical for glassy material, and a lower region which shows a “grainy” break pattern (Fig.11.). The experimental data

Fig.11. SEM micrograph of a cross-section of a latent image formed in PMMA resist.

application in selecting materials for X-ray optical elements such as masks, filters and windows, where resistance to radiolysis is required, as well as for formulating photoresists, where sensitivity to radiation is necessary.

PMMA has been used extensively as an imaging medium in X-ray and electron-beam lithography. It offers the highest resolution and contrast[22] and has become the main material in micromachining where

Fig.12. SEM micrograph of a pattern exposed in PMMA at an initial stage (15 min) of development. Developed depth is ~100 μ m.

obtained on 18 samples show an increase in the depth of the boundary with the top surface dose. The calculated absorbed dose on the boundary is within the range of 500-600 J/cm³. An example of a PMMA pattern obtained after 15 min of development (initial stage) is shown in Fig.12.

After exposure the PMMA samples exhibit swelling. Fig.13. shows a Tencor profile obtained within the first 30 minutes after exposure with a top absorbed dose of 8.4 kJ/cm³. The profile clearly indicates 0.75 μ m swelling of the exposed area. The

Fig.13. Profile of PMMA resist pattern obtained within the first 30 minutes after exposure.

left part of the profile corresponds to the unexposed region of the resist. The raised (right) part of the profile corresponds to the exposed area. The narrow peaks on the profile correspond to the resist surface under the absorber.

Surface profile measurements were performed repeatedly, covering a time span of several hundred hours after exposure. The measurements were performed for samples exposed in different conditions: vacuum, helium and oxygen. The data indicates that relaxation of the radiation induced swelling occurs over long period of time (hundreds of hours)[23].

The observed swelling effect can be associated with the accumulation of large numbers of small volatile molecular fragments produced as a result of the degradation of the polymer, (radiolysis) [24]. During the relaxation period these fragments migrate to the surface and escape. At this stage we observe thickness losses. The thickness losses in vacuum are significantly smaller than in He and O₂. Losses as high

Fig.14. Profile showing PMMA resist losses after exposure (5 torr He, ~40kJ/cm²)

as ~20 μm were measured in PMMA exposed in 5 Torr He with a surface dose of 40kJ/cm². (Fig.14.). Exposures performed in O₂ and He at higher pressure (20 Torr) with low surface doses (2-3 kJ/cm²) did not induce higher losses. The results of these experiments clearly indicate enhanced losses when using gases versus vacuum. Thickness losses in vacuum can be associated only with radiolysis of the resist, while in a presence of a gas, especially ionized, additional reaction can be expected (e.g. in oxygen plasma). The ionized noble gases also can be aggressive oxidizing agents. Ionized Helium has high electron affinity, and it is not known to form stable compounds, therefore, its chemical action is expected to be of a catalytic nature[8,9].

8. Thin Film Thermal Sensors

In microfabrication technology the issues of thermal effects and the ability to measure temperature and absorbed energy in micro-systems are becoming important. Using thin metal resistors as thermal sensors[25-27] has proven to be very successful. In these studies thermal resistors demonstrated high sensitivity and fast response allowing measurements of temperature rise and relaxation time of thermal

Fig.15. Thin gold film resistors on a 4" silicon wafer ("HP ScanJet" image).

effects in x-ray masks in conditions similar to the real x-ray lithography exposures. The exceptional radiation stability of metals [17,18] makes this technique especially suitable for calorimetry, radiometry and

dosimetry of high fluxes of ionizing radiation., such as X-ray radiation produced by a synchrotron. The sensors are based on temperature dependence of electrical resistance of a metal. Different designs of metal thermal sensors can be realized, such as a pile of thermal resistors, or interlaced resistors.

The resistors were fabricated in gold on a silicon

Fig.16. SEM Micrograph of a single serpentine shaped gold thin film resistor

wafer using optical lithography methods[28] (ORIEL UV exposure station). A pattern consisting of eight resistors was formed on a 2 µm thick insulating layer of SiO₂ in order to prevent contact of metal resistors with Si substrate (Fig.15.). Each resistor is a 38 mm long and 7-10µm wide winding wire, covering an area

Table 2.
Resistance and calibration coefficient

τ , nm	$R(25^{\circ}C)$, Ω	β , $\Omega/^{\circ}C$
30	6160	10.67
50	3394	7.24
100	1207	2.93
180	816	2.36
250	474	1.24

of 0.5 x 2 mm² (Fig.16).

Gold resistors were prepared with five different thicknesses: 30, 50, 100, 180, and 250 nm. The gold was evaporated on top of a 50 Å thick layer of chrome in order to provide good adhesion. The wafers were then subjected to a gold and chrome etch to form gold wires of appropriate width. Resistance measurements were performed using an 8-digit display Keithley

2001 Multimeter model with 8-channel data acquisition scanning card.

The resistance $R(T)$ of a metal depends on the temperature T linearly[29] over a very wide range of temperatures:

$$R(T) = R(T_0) + \beta \cdot (T - T_0),$$

where β is the temperature calibration coefficient. The measured values of $R(25^{\circ}C)$ and of $\beta(30-100^{\circ}C)$ for all fabricated thicknesses τ are presented in Table2.

To compare the electrical properties of a fabricated thin gold film with data available in the literature, the values of electrical resistivity ρ and its temperature coefficients α for pure gold were collected from several sources[29-31] and statistically analyzed[28]. The collected values for ρ and α were found to be distributed in a reasonable range (~15%): $\rho(25^{\circ}C) \pm \sigma_{\rho} = 2.29 \pm 0.10 \mu\Omega \cdot cm$; $\alpha(25^{\circ}C) = 3.2 \cdot 10^{-3} 1/^{\circ}C$ and $\alpha(100^{\circ}C) = 2.5 \cdot 10^{-3} 1/^{\circ}C$. These values were used to calculate resistance of resistors for all fabricated thicknesses. Fig.17. illustrates how the value of electrical resistance of thin gold film is converging to a value calculated for bulk gold properties as the resistor thickness increases. The 30nm thick resistors show resistance about twice the value of the expected, while the resistance of 250 nm thick gold film coincides with the calculated value. The convergence of the temperature coefficient of

Fig.17. Comparison of calculated and experimental resistance values for thin gold film resistors (T=25°C)

resistance with increasing film thickness was observed. The 250 nm thick resistors were found to exhibit behavior very close to the bulk material. The thermal sensitivity and precision of fabricated resistors was determined from several sets of measurements, each set containing 10 values of average (of 100, 30,

Fig.18. Resistance measurements for 250 nm thick gold film resistors. Each point is an average for 30 individual measurements ($t_0 = 23.2^\circ\text{C}$).

or 10) and standard deviation of a resistance. Fig.18. shows one of these sets of measurements, performed using 250 nm thick resistors with the resistance-temperature change of $\beta = 1.24 \Omega/^\circ\text{C}$. During the time of these measurements the temperature change was less than 0.1°C , as recorded by a thermocouple and the resistance monotonic increase was 0.025Ω . This observation suggests that the substantial part of the standard deviation comes from the temperature drift. The standard is $7.8 \cdot 10^{-4} \Omega$ for a single data point, which is less than the difference in resistance between any two consecutive points. In terms of temperature a typical standard deviation is 0.001°C . The data presented in this section lead us to believe that the

Fig.19. Temperature profiles measured in vertical direction on a silicon wafer during x-ray exposure in vacuum and in helium ($t_0 = 22^\circ\text{C}$).

thermal sensitivity of the fabricated resistors is better than 0.001°C .

The measurements of thermal behavior of silicon wafers were performed in situations equivalent to X-ray exposure conditions typical for micro-machining. Temperature rise measurements were conducted in the above described X-ray exposure station. Temperature profiles observed in a silicon wafer during the exposure are shown in Fig.19. This figure represents the temperature distribution in a vertical direction over the range of 40 mm. The temperature rise recorded in the center of the X-ray beam in different conditions was: in vacuum - $\Delta T = 43.7^\circ\text{C}$; in He atmosphere - $\Delta T = 13.9^\circ\text{C}$ at 1 torr, $\Delta T = 11.3^\circ\text{C}$ at 5 torr, and $\Delta T = 10.6^\circ\text{C}$ at 10 torr. Relatively uniform temperature distribution within the 40 mm field can be attributed to a high thermal conductivity of the silicon[26-28] wafer. An estimation of the temperature rise in vacuum based on thermal radiation[30] losses yields an average temperature rise of $\Delta T \approx 40^\circ\text{C}$, which is in reasonable agreement with the observed value (see Fig.19.)

9. SUMMARY

- A series of studies in synchrotron radiation based X-ray micro-lithography is underway.
- An X-ray micro-lithography exposure system able to facilitate radiation assisted processes has been built. The system consists of a beamline and X-ray scanner.
- A concept of exposure radiation equivalent filtration was introduced. The equivalent filtration and the choice of X-ray mask pattern carrier and absorber are essential for X-ray exposure optimization.
- Thick PMMA resist application techniques have been developed. Using a combination of spinning and bonding a wide variety of resist thickness can be produced
- The X-ray mask configuration for conventional imaging and issues of mask standards were presented.
- The transfer mask technique has been introduced and demonstrated. High aspect ratio and free standing 3-D resist patterns were formed using the transfer mask method.
- The X-ray radiation effects in materials were addressed. Latent image formation in PMMA resist was observed, PMMA swelling and thickness losses were measured.

- Preliminary experiments of radiation-induced PMMA self-development process demonstrated higher efficiency of He and O₂ vs. vacuum.
- Gold thin film thermal resistors were fabricated and tested. The electrical properties of thin gold films were studied and compared with bulk properties
- The fabricated resistor demonstrated high sensitivity and precision ($< 0.001^{\circ}\text{C}$) and were used in a preliminary study of synchrotron X-ray radiation thermal effects in Si wafers.

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