



Real-space insight in the nanometer scale roughness development during growth and ion beam polishing of molybdenum silicon multilayer films



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ABSTRACT

Room temperature deposition of single and multiple layers of silicon and molybdenum has been explored *in vacuo* by scanning tunneling microscopy at growth conditions used for typical Mo/Si multilayer optics, enabling the study of the topography down to the nanometer scale. Periodic Mo/Si multilayer films with a molybdenum layer thickness of 2.5 nm and a silicon layer thickness of 5 nm show an evolution of the surface roughness that is similar to polycrystalline self-affine film growth. By applying an ion beam treatment of the silicon layers this increase of the roughness with layer thickness is completely mitigated, yielding a final roughness of the entire stack similar to that of the first ion treated silicon layer. The ion treatment step used here is most efficient at spatial frequencies around $1/10 \text{ nm}^{-1}$. Polycrystalline growth of molybdenum on this ion treated silicon layer is observed only when the layer exceeds 3 nm thickness, while smaller amounts of molybdenum do not significantly increase the surface roughness. The almost identical values for the roughness of ion treated silicon and the 2.5 nm molybdenum grown on top of ion treated silicon show that the roughness of Si-on-Mo and Mo-on-Si interfaces have a similar contribution to the optical performance of Mo/Si multilayer films. The contributions originate mainly from spatial frequencies above $1/4 \text{ nm}^{-1}$.

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1. Introduction

Mo/Si multilayers form the heart of the optical system applied in extreme ultraviolet photolithography (EUVL), employing radiation with a wavelength of 13.5 nm. Although a reflection of 75% is theoretically possible, imperfect interfaces between the two materials are responsible for a limited practical reflectivity. Interface imperfections can be a result of either surface roughening during deposition of each multilayer component, or can be caused by compound formation of the multilayer components at the interfaces. Previous studies have shown that optimal performance of Mo/Si multilayers is achieved for layer growth at room temperature [1–3]. A procedure of periodic noble gas ion treatment of the silicon layers after deposition has resulted in a reflectivity of 69% [4,5]. Recently krypton ion treatment was confirmed to be the best choice at low ion energy for optimal reflectivity at the operational wavelength [6]. *In situ* X-ray reflectometry, used to monitor layer deposition, has enabled an impression of the

interface roughness development during the build up of multilayer systems. *Ex situ* interlayer and crystallite formation in molybdenum silicon multilayers have been studied using grazing-incidence X-ray reflectivity (XRR), X-ray diffraction (XRD) and transmission electron microscopy (TEM) [1–3,5,7,8]. From these studies it has been concluded that the polycrystalline nature of the molybdenum layers is the main cause of interlayer roughness. Furthermore, the ion smoothing of deposited amorphous silicon layers has been studied *ex situ* with X-ray scattering (XRS) and atomic force microscopy (AFM) [3,9–13]. All these studies lack the direct measurement of the surface morphology during the deposition process.

In this work we investigate the deposition of both multilayer components and the ion treatment of the silicon layers using *in vacuo* scanning tunneling microscopy (STM), providing real-space insight in the nanometer scale processes relevant for improvement of the fabrication of multilayer optics. By quantifying the layer morphology at different stages of the deposition process for dimensions down to a nanometer, its effect on the “intermixed” zone between two materials can be estimated. Further improvement of interlayers in the multilayer can now focus on the larger contribution of either layer morphology or other effects like compound formation. All deposited layers are prepared under

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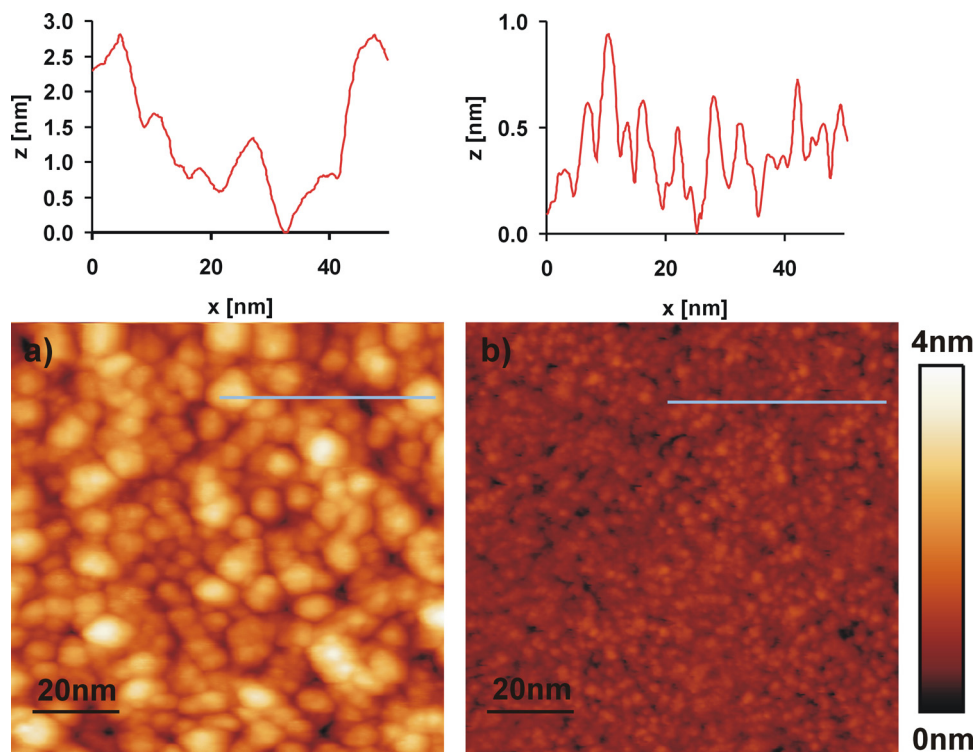


Fig. 1. 100 nm $\times$ 100 nm STM topography images of a 75 nm Si/Mo multilayer film (10 periods of 5 nm Si and 2.5 nm Mo) without (a) and with (b) periodic ion treatment of the Si layers. Line scans of the frames are shown on top.

relevant conditions by using a deposition set-up that is also used for the development of multilayer films for EUV optics. After the experimental details, we first describe the evolution of the surface roughness for multilayer films up to 75 nm thickness, with and without ion beam polishing. Subsequently, we focus in more detail on the growth and ion polishing of silicon and the evolution of surface roughness during the deposition of molybdenum on silicon.

2. Experimental details

Layers were grown at room temperature onto the native oxide of super-polished silicon substrates in an ultra-high vacuum (UHV) environment better than 10^{-8} mbar. Silicon and molybdenum layers were deposited by electron beam evaporation [1]. A Kaufman type hot cathode ion source, providing 100 eV krypton ions, was used to modify the surface of freshly deposited silicon layers. A fluence of 1.5×10^{16} ions/cm² under 45° angle of incidence was used, resulting in removal of 0.5 nm of Si [14]. Quartz crystal oscillator microbalances were used to control the amount of deposited material with an accuracy better than 1% of the reported value and to monitor growth rates. Amounts and rates are reported as layer thickness and thickness change assuming bulk density. Deposition rates are kept constant for both materials at 0.025 nm/s. In this paper, layer stacks are denoted in the order of deposition, i.e. layer1/layer2, with layer2 being the top layer. Samples were transported from the coating facility via a vacuum transfer system, base pressure 1×10^{-9} mbar, to be analyzed with the aid of an STM system manufactured by Leiden Probe Microscopy. The STM chamber was operated at a base pressure of 1×10^{-9} mbar as well. The STM imaging was performed with mechanically cut Pt/Ir tips, using a sample bias of 2 V and a current of 0.2 nA as typical tunnel parameters. STM performance was verified on HOPG where the observed atomic spacing was used for lateral calibration and on polycrystalline gold where the step heights on the grains were used to

calibrate heights. In order to prevent possible influences on the film growth by previously deposited layers and/or prolonged exposure to residual gas during transport and analysis, a fresh silicon wafer was used for every experiment.

3. Results and discussion

3.1. Multilayer growth and scaling

From previous studies it is known that the ion treatment step of silicon during multilayer deposition has large impact on the surface roughness [4,11]. In order to quantify this effect, first multilayers with and without periodic ion treatment are compared. In order to get an overview of the surface morphology of such a multilayer, the evolution of the surface roughness (sometimes also referred to as vacuum interface width) versus the amount of deposited material is investigated. One should keep in mind that the use of two materials in the film will generate regions where the film growth will be complicated by interlayer formation. Fig. 1 shows an example of the surface roughness of two of these multilayers. The STM images show the top surface morphology of a silicon/molybdenum multilayer film consisting of 10 periods that are always molybdenum terminated. Each molybdenum layer is 2.5 nm and each silicon layer is 5.0 nm thick, adding up to 7.5 nm per period. Fig. 1(a) displays the morphology of the top surface without ion treatment of the silicon layer and Fig. 1(b) the morphology after application of an ion treatment of each silicon layer. In order to clearly display the effect of the ion treatment, the height contrast of Fig. 1 is the same for both pictures. The height differences are illustrated with the respective line-scans. It is observed that the height variations are reduced by approximately a factor of 3 due to the ion treatment of the silicon layers. Furthermore, Fig. 1(a) shows that a wide variety of feature sizes with diameters up to 10 nm have evolved during film growth. With ion treatment, the characteristic lateral length scale of the morphology is only a few nanometers.

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