Pure Silicon Oxide by Electron Beam Induced Deposition for Nanooptical Applications

H. Wanzenboeck, M. Fischer, G. Hochleitner, E. Bertagnolli

Vienna University of Technology, Institute for Solid State Electronics
Floragasse 7/1, A-1040 Vienna, Austria

Micro- and nanostructured surfaces have increasingly been adopted for optical applications such as lithography masks, imprint masks, photonic crystals and optoelectronics. Silicon oxide takes a predominant position among transparent materials and the precise geometry and accurate position of structures if often vital.

Introduction

Optical devices for the nanoregime have to satisfy strict specifications on the material purity, on the accurate position of individual features, and on geometric tolerances of (occasionally even) 3-dimensional structures. Silicon oxide is among the most used materials for optical devices and finds applications in lens systems, photomasks and optical MEMS systems. This work reports on the progress of direct-write deposition of silicon oxide as optical-grade material.

Siloxanes such as tetramethylorthosilicate (TMOS) are frequently used as precursor for chemical vapor deposition of silicon oxide films. With plasma assisted CVD of TMOS highly transparent and adherent films with a low content of carbon residue were obtained [1]. FTIR spectra of SiO_x films deposited from TMOS are very similar to those of pure SiO_2 and had a high transmission of 90% in the wavelength range from 400 to 800 nm. Amorphous plasma enhanced chemical vapor deposited (PECVD) silicon oxide films suitable for integrated optics applications have been produced [2]. Film properties matching the corresponding properties of silicon thermal oxide were obtained by optimizing the composition of the reactant gas flow.

Focused gallium ion beams have also been used for local material deposition using tetramethoxysilane as a precursor. [3] Deposition rates of 0.33 $\mu m^3/nC$ were observed, but the material was strongly contaminated with gallium. FEB induced deposition combines the advantage of a direct write process (one exposure) with extremely high flexibility of deposit geometries (zero- to three-dimensional) at nanometer scale [4]. Due to the absence of gallium the electron beam is capable to overcome the contamination obstacle. Silicon oxide films with improved insulating properties were also deposited using an electron beam [3]. However, material purities of EBID deposited materials have not been in a satisfactory range for optical applications such as photomask repair. In this work we report on an electron beam induced deposition process for chemically pure silicon oxide. The high material qualities achieved facilitate applications as electrical insulator and for optical purposes.

Experimental

Locally confined deposition of silicon oxide is induced with a focused electron beam.

The deposition position of the silicon oxide structure could be accurately controlled by scanning the electron beam only over predefined surface areas. The deposition mechanism is based on a localized chemical vapor deposition (CVD) initiated by the energy of the focused electron beam. Electron beam induced deposition was performed with a custom-adapted Zeiss NTS “LEO 1530 VP” equipped with a 3-stage pumping system for the electron column, a high-current upgrade and a custom-tailored gas injection system for simultaneous inlet of up to 3 process gases. Beam control by a pattern generator allows fabrication of arbitrary features. The precursor gases used for silicon oxide deposition were siloxanes and oxygen. The electron beam was focused down below 10 nm diameter. At 1 kV beam voltage a 2.5 nA electron current was obtained. Substrates used for deposition of silicon oxide were silicon and gallium arsenide for chemical analysis, and quartz glass or calcium fluoride for optical characterization. For optical measurements a gold film with a 100x100 micron aperture for beam transmission was deposited by physical sputtering and structured by optical lithography. Optical transmission was performed with a microlens set-up with a UV/Vis spectroscopic detector. Chemical composition was analyzed by energy dispersive X-ray analysis (EDX). With GaAs carriers a clear distinction between deposited silicon oxide and gallium arsenide was feasible. A sample of pure quartz glass was used as reference for interpretation of EDX-results. The surface topography was determined with a “Digital Instruments 3100” atomic force microscope (AFM) utilizing a diamond-coated cantilever in tapping mode.

Results

Square area structures were deposited (Fig. 1(a)) and display well confined side faces. The surface of the deposited layer was remarkably smooth as illustrated by the AFM. The height of the 600 nm high deposition was very homogeneous; only on two opposite sides slight shoulders were observed as a result of the turnaround position in meander scan.

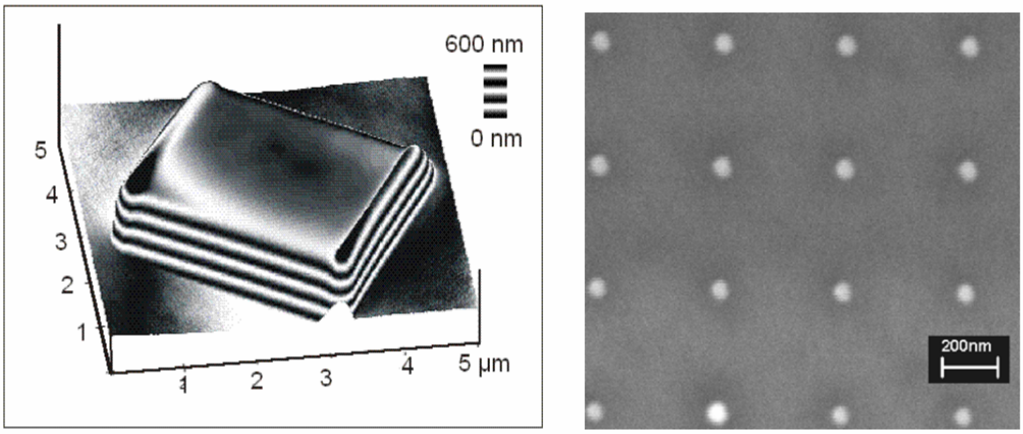

Fig. 1: Electron beam induced deposition of SiO_2 as (a) square area and (b) dot array

A dot array of silicon oxide nanoparticles (Fig. 1(b)) was deposited by deliberately setting a 500 nm wide spacing between scan pixels. Single, round-shaped nanoparticles with a diameter below 50 nm could be fabricated.

The process parameters and the gas composition had to be optimized to achieve high material quality. By oxygen addition electron induced deposition yielded contamination-free silicon oxide. The material composition as determined by energy dispersive X-ray spectroscopy (EDX) is illustrated in Fig. 2.

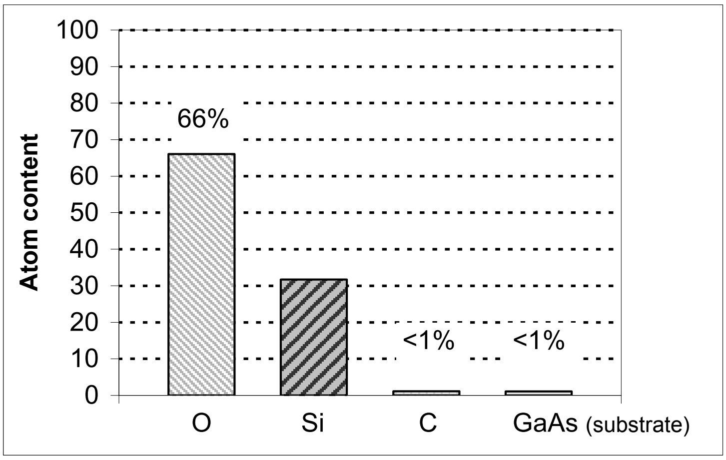

Fig. 2: Chemical composition of EBID-deposited silicon oxide measured by EDX.

Optical properties in the visual range were investigated by optical transmission in the Vis-range using a deposition made on an aperture hole (Fig. 3). With low wavelengths absorption is still considerable. In the mid visual range and with thin layers satisfactory transmission was achieved. In further investigations, thermal annealing of samples will be explored as approach to further increase the optical quality.

Fig. 3: Schematic illustration of setup for measurement of optical transmission of 120x120 micron deposition on 100x100 micron aperture in reflection layer.

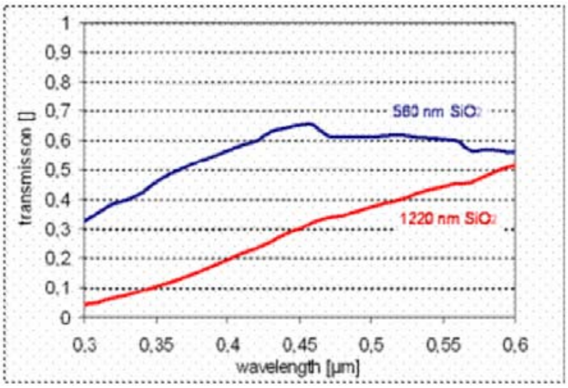

Fig. 4: Optical transmission spectrum of EBID-deposited silicon oxide.

Conclusion

With the electron beam induced deposition pure silicon oxide objects can be fabricated within a single process step. This maskless fabrication method renders a versatile prototyping and repair tool for existent and future nano optics.

References:

[1] Yang MR, Chen KS, Hsu ST, Wu TZ , Surface & Coatings Technol 123 (2-3), 204 (2000)

[2] Dominguez C, Rodriguez JA, Munoz FJ, Zine N , Vacuum 52(4), 395 (1999)

[3] Lipp S, Frey L, Lehrer C, Frank B, Demm E, Pauthner S, Ryssel H, Journal of Vacuum Science & Technology B 14(6), 3920 (1996)

[4] Bret T, Utke I, Hoffmann P, Microelectronic Engineering 78-79, 307 (2005)