Fabrication of high aspect ratio tungsten nanostructures on large area ultrathin c-Si membranes for X rays applications

F. Delachat, J. Margot
Université de Montréal, C.P. 6128, Montréal, Canada QC H3C 3J7
florian.delachat@umontreal.ca
C. Constancias, C. Cadoux, P. Gergaud
CEA-LETI – Univ. Grenoble Alpes -17 rue des Martyrs F-38054, France
B. Le Drogoff, S. Delprat, M. Chaker
INRS – EMT, 1650 Boul. Lionel-Boulet, Varennes, Québec J3X 1S2, Canada

Fabrication of highly reproducible nanostructured membranes is a major asset for applications in nanoscience while silicon is highly desired for transmission X-rays optics because of its extensive use in microelectronics and of its very high transparency (*e.g.* Si is the preferred choice for soft X-rays / Extreme UltraViolet membranes @ 13.5 nm)[1]. In this work, we demonstrate a full fabrication process of high aspect ratio (up to 8.5) diffraction gratings made of tungsten on flat (planarity <10nm), ultrathin (100 nm and 50 nm) and large area (1 x 1 mm^2) silicon membranes using pseudo-Bosch etching innovatively applied to tungsten. First, we report a state-of-the-art process for the fabrication of ultra-thin Si membranes, based on a new hydrophobic direct-bonding process (involving thermal difference between a silicon-on-insulator wafer and a perforated wafer). This method enabled us to produce large area (several square millimeters) flat membranes with circular/square designs as reported on Fig.1.A. Afterwards, the silicon membrane is coated with a few hundred nanometers (100 to 300 nm) of stress-controlled tungsten and a very thin chromium layer deposited by sputtering. The line/space gratings were then written in a thin resist layer by electron beam lithography (VB6 Vistec-Raith) and transferred onto tungsten via the consecutive Induced Coupled Plasma (ICP) etching of the Cr hard mask and the W layer. In literature, the etching of high resolution gratings in tungsten was successfully demonstrated through the use of ICP etching with accurate substrate cryogenic temperature control.[2] However, cryogenic temperature control is challenging for large area membranes as the accuracy of the temperature control is deteriorated by the restricted thermal exchange imposed by the suspended membrane. Therefore, the use of a large temperature window process is essential in order to achieve highly anisotropic patterning of tungsten over large area Si membranes. The pseudo-Bosch tungsten etching developed in this work enabled us to fit these requirements with the simultaneous injection of SF_6 and C_4F_8 gases near room temperature (15-25°C). As reported on Fig.1.B, a high anisotropic tungsten etching process was achieved at room temperature. Finally, the homogeneity of gratings and the diffraction efficiency were demonstrated with SEM and Small Angle X-ray Scattering using synchrotron radiation. The experimental scattering intensity achieved on the 50 nm half-pitch grating is reported on Fig.2 with the associated top-view SEM image. This whole fabrication process is extremely promising because it is fully compatible with standard industrial semiconductor technology.

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1 B.L. Henke, E.M. Gullikson, J.C. Davis, *Atomic Data and Nuclear Data Tables* **1993,** 54, 2.
2 L. Goodyear, S. Mackenzie,D.-L. Olynick, E. H. Anderson, *JVST. B* **2000,** 18, 3471.

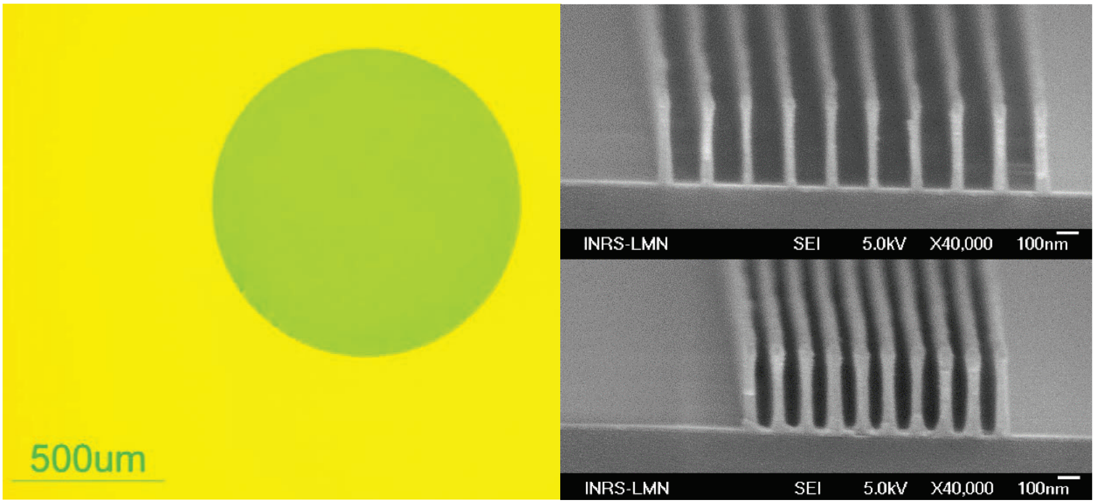
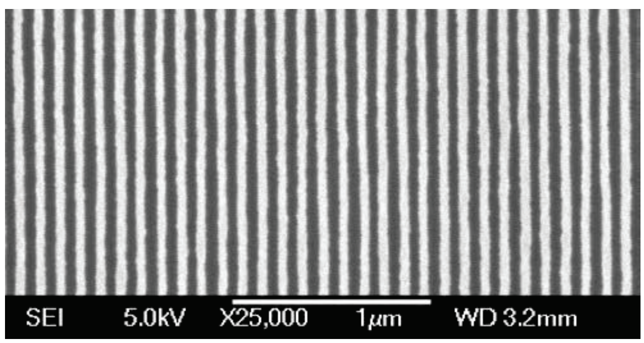


Figure 1.A. *Photography of the suspended ultrathin (100 nm) monocrystalline Si membrane. The c-Si membrane has a diameter of 1mm and is secured on a 2×2 cm² frame.* ***B.*** *Cross sectional views of line-space grating on c-Si bulk achieved using sulfur hexafluoride (SF_6) and octafluoro-cyclobutane (C_4F_8) gas mixture: (a) line (35 nm), space (145 nm) and aspect ratio equal to 10; (b) line (60 nm), space (60 nm) and aspect ratio equal to 6.*



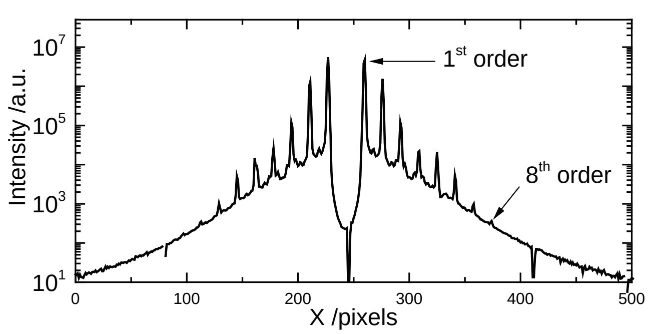


Figure 2. *SEM top view imaging of the 50 nm half pitch tungsten grating with an aspect ratio equal to 6 achieved on a 1 mm ultrathin (100 nm) Si membrane. Small Angle X-ray Scattering of the high resolution tungsten grating measured using synchrotron radiation at 17keV. The first eight diffraction orders of the 50 nm half pitch tungsten gratings are distinctively measurable.*