Fabrication of high aspect ratio metal gratings for X-ray phase contrast interferometry

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Interferometric X-ray phase contrast imaging[1] (see Fig.1) has a high application impact in material science and medicine for imaging of weakly absorbing (low Z) materials and soft tissues. The main challenge is the fabrication of the absorbing gratings, which are periodic high aspect ratio microstructures of highly absorbing material, such as gold, whose quality and aspect ratio strongly affect the contrast of the generated images. In this paper, we review the grating fabrication strategy with a special focus on a novel approach using hot embossing for metal casting into silicon grating templates[2]. The Si gratings were produced by deep reactive ion etching or metal assisted chemical etching with line pitches in the range of 2 – 23 μm and depths in the range of 10 – 100 μm.

While metal microstructures can be manufactured using casting, little work has been published on this method compared to the other approaches (forging, electroplating, micro powder injection molding etc). Here, we used metal alloys with low melting point (<320 °C), such as Sn- and Pb-based alloys, for casting into silicon trenches. The wetting properties of the liquid metal both on top and on the trench wall surface were extremely important for uniformly filling the Si grooves (see Fig.2). When the liquid metal did not uniformly wet the available Si surface, the metal was unequally distributed over the grooves filling only some of them. A thin layer (20 nm) of Ir was deposited by Atomic Layer Deposition (ALD) to conformally coat the Si surface and thus, improve the wettability of the liquid metal on the template during the casting process.

The metal casting was performed in a Jenoptik HEX 03 hot embossing tool by pressing the metal foil in contact with the grating surface at the melting temperature. The process, similar to nanoimprint lithography, requires particular adjusting efforts because of the sharp transition between solid and liquid state, and the low viscosity of the molten metal. A typical experiment is reported in the schematic of Fig.2. Figure 3 shows the Si trenches fully filled with Pb-In alloys. The quality of the metal grating was investigated by scanning electron microscopy and tested in X-ray grating interferometry. The obtained performance of the gratings is comparable to that of electroplated gold but the process is much faster, cheaper and scalable to large area. We demonstrated fabrication of 75×75 mm^2 gratings on <100> wafer with aspect ratio higher than 20:1.

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1 T. Weitkamp, A. Diaz, C. David, F. Pfeiffer, M. Stampanoni, P. Cloetens, and E. Ziegler, Optics Express **13,** 6296 (2005).

2 L. Romano, J. Vila-Comamala, M. Kagias, K. Vogelsang, H. Schift, M. Stampanoni, and K. Jefimovs, Microelectronic Engineering **176,** 6 (2017).

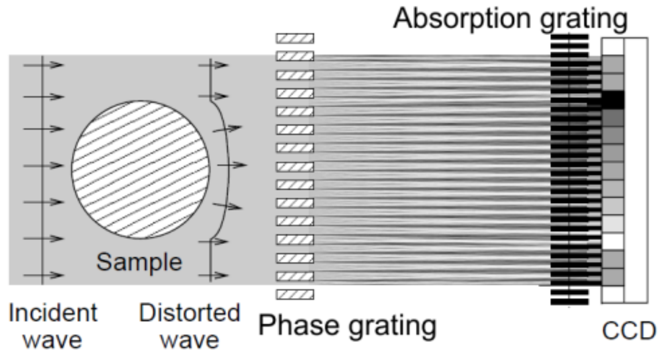

Figure 1. Schematic of X-ray phase contrast interferometry (adapted from ref. 1).

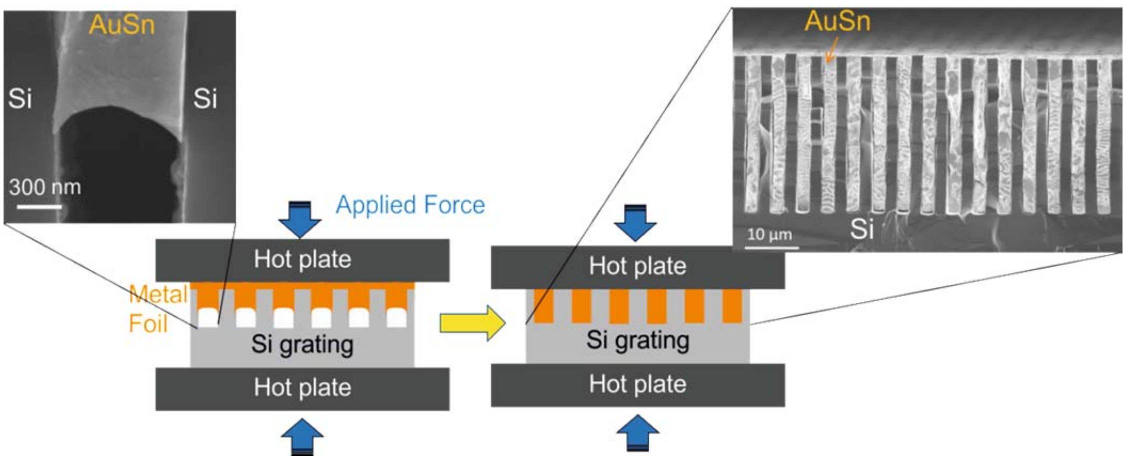

Figure 2. Schematic of hot-embossing with Au-Sn alloy foil and Si grating. SEM images in cross-section show a detail of the wetting property (left side) and an example of metal filled grating (right side) with pitch size of 4.8 µm and depth of 40 µm (adapted from ref. 2).

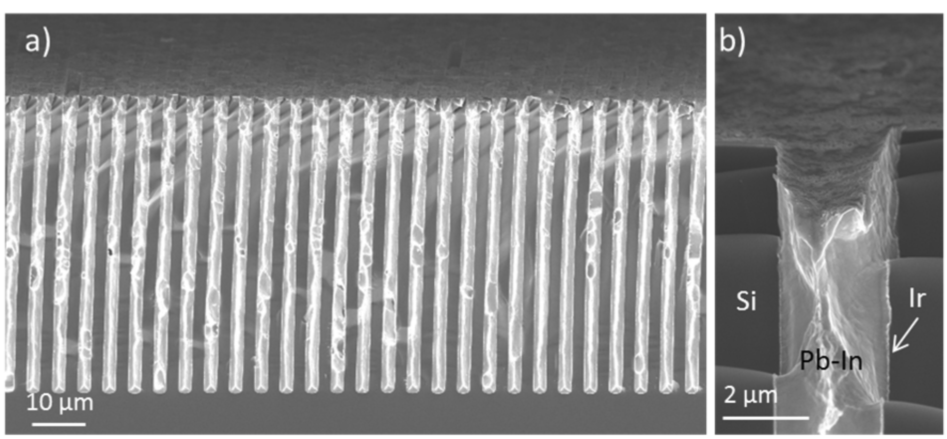

Figure 3. SEM image in cross-section of Si grating (pitch 4.8 µm and depth 55 µm) filled with Pb-In alloy (a). High mag SEM (b): Ir layer by ALD is indicated by the white arrow; Si and Pb-In alloy are also indicated. The liquid metal is completely filling the template with a residual top layer of few nanometers.