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Research paper

High aspect ratio metal microcasting by hot embossing for X-ray optics fabrication

L. Romano [a,b,c,*], J. Vila-Comamala [b,c], M. Kagias [b,c], K. Vogelsang [b], H. Schift [b], M. Stampanoni [b,c], K. Jefimovs [b,c]

[a] Department of Physics and CNR-IMM, University of Catania, 64 via S. Sofia, Catania, Italy
[b] Paul Scherrer Institut, 5232 Villigen PSI, Switzerland
[c] Institute for Biomedical Engineering, University and ETH Zürich, 8092 Zürich, Switzerland

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ABSTRACT

Metal microstructured optical elements for grating-based X-ray phase-contrast interferometry were fabricated by using an innovative approach of microcasting: hot embossing technology with low melting temperature (280 °C) metal alloy foils and silicon etched templates. A gold-tin alloy (80 wt% Au/20 wt% Sn) was used to cast micro-gratings with pitch sizes in the range of 2 to20 μm and depth of the structures up to 80 μm. The metal filling of the silicon template strongly depends on the wetting properties of the liquid metal on the groove surface. A thin metal wetting layer (20 nm of Ir or Au) was deposited before the casting in order to turn the template surface into hydrophilic with respect of the melted metal alloy. Temperature and pressure of the hot embossing process were optimized for a complete filling of the cavities in a low viscosity regime of the liquid metal, and for minimizing the shear force that might damage the silicon structures for small pitch grating. The new method has relevant advantages, such as being a low cost technique, fast and easily scalable to large area fabrication.

1. Introduction

Metal microstructures are nowadays of great interest for sensors and electromechanical devices [1], manufacturing molds for nanoimprint lithography (NIL) [2,3], and for many optics applications, such as plasmonic devices, photonic crystals [4] and X-ray optics [5]. In particular, interferometric X-ray phase contrast imaging based on diffraction gratings requires the fabrication of high aspect ratio (HAR) metal microstructures [6]. Grating-based X-ray phase-contrast Interferometry has shown to provide a much higher image contrast than conventional absorption-based X-ray radiography imaging [5]. This has a high application impact in material science and medicine for imaging of weakly absorbing (low Z) materials and soft tissues [5]. The essential part of the traditional interferometer consists of one phase and two absorption gratings. The main challenge is the fabrication of the absorption gratings, whose quality and aspect ratio (AR) strongly affect the quality of the generated images. There is the need to fabricate gratings with i) high aspect ratio (in the range of 100:1, structural width in the micrometer range); ii) large area (mammography, e.g., asks for a field of view of 200×200 mm^2) and iii) good uniformity (no distortions and change in the period and height over the whole grating area). Absorption gratings are usually fabricated by metal electroplating (typically of gold, which is one of the highly absorbing material for X-rays), into high aspect ratio grating templates produced by LIGA [7] or deep silicon (Si) etching [8].

These existing processes and materials lead to high fabrication cost and low yield, limiting the broad utilization of X-ray grating interferometry for industrial and medical applications. Therefore, the mass production of HAR and large area absorption gratings with metal microstructures through a simple process is essential for commercialization of GI based X-ray imaging systems.

Metal microstructures are typically manufactured using forging [9], electroplating [8], micro powder injection molding [1] or casting [2, 10]. Forging can produce small-scale structures in ductile metals [4]; the depth is usually limited to the micrometer range [4,9]. Microscale metal electroplating is less expensive than forging, but is usually slower. Micro powder injection molding is faster than electroplating, but it is currently limited to 20 μm structure width sizes. Metal nanostructures have been produced in thin metal film by using NIL [4,11,12]. While metal microstructures can be manufactured using casting, the direct imprint in metal has not been deeply investigated in comparison to the other approaches. Microcasting for X-ray absorption gratings has been developed by using molten bismuth (melting temperature 271 °C) via capillary action and surface tension [13,14]. However, the low density (9.78 g/cm^3, atomic number 83) of bismuth requires much higher (factor of 1.7 at 30 keV) AR structures to get an absorption level comparable to that of gold (density 19.32 g/cm^3, atomic number 79). Gold microcasting can be very expensive and not easy to scale up on large

* Corresponding author at: Paul Scherrer Institut, 5232 Villigen PSI, Switzerland.
E-mail address: lucia.romano@psi.ch (L. Romano).

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area, not only for the required bulk quantity of liquid metal but also for the high melting temperature of gold (1064 °C). There are some other metal alloys which have the benefits of low temperature liquid phase and high X-ray absorption. For example, several tin alloys have been developed for metal bonding technology [15,16], like gold-tin and lead-tin alloys. These materials are commercially available in a wide range of foil thicknesses from few tens to hundreds of micrometers.

In this paper, we propose the use of hot embossing with eutectic gold-tin (Au 80 wt%/Sn 20 wt%) foils into Si templates. The low melting point at 280 °C, its relatively high density 14.7 g/cm^3, make Au-Sn alloy an attractive material for casting X-ray absorption gratings. For example, it requires only a factor of 1.23 higher structures than pure gold at 30 keV to get the same absorption. Moreover, the hot embossing technique is a well-known technique for large area silicon processing being largely used as a tool for NIL technology [17]. In this publication, due to narrow process window for the phase transition between solid and liquid phase, we characterize the physical process as microcasting but the technical method as hot embossing.

2. Material and methods

Fig. 1a shows the schematic process flow for preparing the metal microstructures into Si templates. First, a pattern was realized by conventional UV photolithography in a positive photoresist (MicroChem S1805) Si gratings with duty cycle 0.5, pitch in the range of 2–20 μm, and depth in the range of 25–80 μm were fabricated by deep reactive ion etching [18] with the so-called Bosch process [19] or by Metal Assisted Chemical Etching (MACE) [20,21]. The 100 mm diameter Si wafers were 〈100〉 n-type (0.005–0.01 Ωcm) and p-type (1–30 Ωcm) for Bosch etch and MACE, respectively. An example of Si grating produced by the Bosch process is reported in Fig. 1b, the grating was cleaved in order to obtain a cross section image by Scanning Electron Microscopy (SEM). Seedless electroplating [22] was used to grow a conformal thin layer of Au on the Bosch etched grating (see Fig. 1c) to improve the wettability of the liquid metal on the template during the casting process. Due to the high resistivity of the wafer, the seedless electroplating is not possible on MACE gratings, so Atomic Layer Deposition (ALD) was used to realize a conformal coating of 20 nm of iridium (Ir). Afterwards square gratings of 20 × 20 mm^2 up to 70 × 70 mm^2 were cut out from the wafer. The metal casting was performed in a Jenoptik HEX 03 hot embossing tool, in vacuum (pressure in the range 100–500 Pa), by pressing a metal foil with same size as the grating in contact with the grating surface at the melting temperature. The pressure was varied from 1 MPa to 12 MPa. The temperature was controlled with a precision of 2 °C and the maximum temperature for this system is 320 °C, therefore metal foils of material with melting point lower than 320 °C can be used. We used a metal foil of eutectic Au-Sn (80 wt% Au/20 wt% Sn with eutectic temperature of 280 °C) alloy [23] from Ametek with thickness of 25 and 50 μm. The thickness of the Au-Sn alloy foil was chosen depending on the profile depth, matching the cavity volume of the grating and minimizing the excess of material. For example, for a duty cycle of 0.5, the thicknesses of the foil should typically be a half the depth of the grooves.

A typical hot embossing experiment [24] is reported in the schematic of Fig. 2a (not in scale). A1 mm thick sheet of silicone rubber (PDMS, i.e. polydimethylsiloxane) was used as a cushion layer for pressure equilibration, it is sufficient to smoothen out any kind of unevenness, e.g. caused by substrate bow and warp or even dust particles. In order to avoid sticking of the PDMS [25], a polyimide foil was used on both sides of the PDMS as an anti-sticking layer. A Si chip with thickness of 500 μm was used as a flat surface to apply the pressure on the metal foil, a polyimide foil between the metal and the Si chip helps to easily detach the casted grating at the end of the process. Fig. 2b reports the measured force and tool temperature as a function of time in a typical process of hot embossing on a grating with size of 20 × 20 mm^2: 1) after evacuating the embossing chamber (in the range 100–500 Pa), the hot plates were heated up from room temperature and 2) a touch force of 300 N was applied while the heating took place with about 15 °C/min; 3) a force of 5 kN was applied once the substrate reached the melting temperature of the metal foil; the applied force was maintained for few minutes and then 4) the system was cooled down to room temperature and finally, 5) the force was released.

3. Results & discussion

In order to ensure that the metal does not flow sideways, the geometrical conditions need to be fulfilled, which means that the foil thickness has to match with the cavity volume of the grating. On the other hand, to achieve complete filling of the lines a wetting layer was used and the casting process was performed in vacuum. We applied different pressure and temperature ramping to optimize the casting process. The good control of pressure, temperature and speed of the system allowed to hot emboss at the melting point of the metal.

Fig. 1d shows the Si trenches filled with bubble-free gold-tin alloy in a 4.8 μm pitch grating with a size of 70 × 70 mm^2. The metal foil was 25 μm thick and the applied force during hot embossing was 20 kN (equivalent to a pressure of 8 MPa on the 70 × 70 mm^2 square and due to the 0.5 duty cycle of the grating). The hot embossed grating was inspected with optical microscopy (not shown), revealing a very uniform filling of the trenches over the full area. The quality of the metal grating was investigated by SEM. For the 4.8 μm pitch grating, the trenches were completely filled with no empty cavities. The excess material flowed sideways on the top of the grating leaving a clean top surface with a very thin residual layer of ~100 nm.

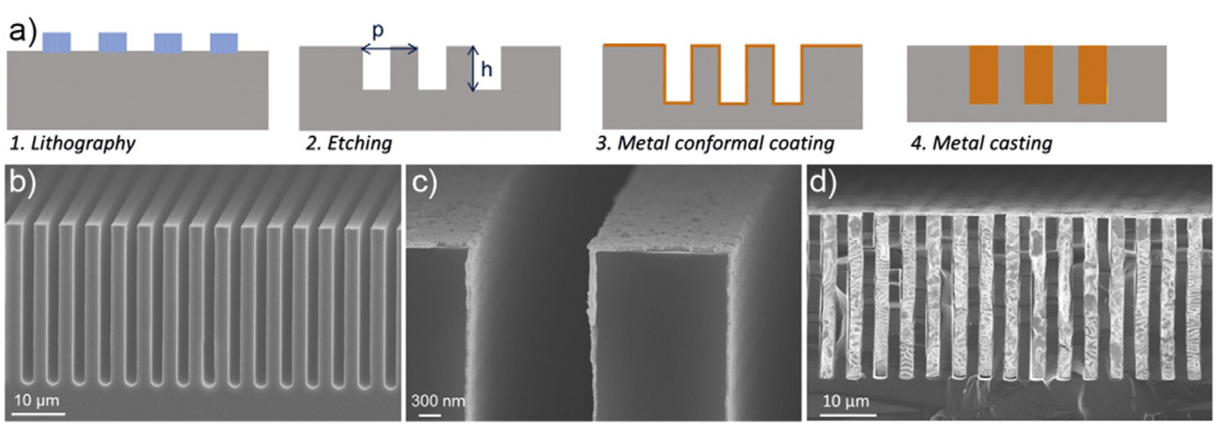

Fig. 1. a) Schematic of the full fabrication process of gratings (not to scale). The following SEM cross section images show some examples of the reported process steps: b) SEM cross section detail of 70 × 70 mm^2, 40 μm deep Si grating etched by Bosch (representative of step 2); c) seedless Au electrodeposition (20 nm Au) to produce a wetting layer on the entire Si surface (representative of step 3); d) Au-Sn alloy casting in the Si template after hot embossing (representative of step 4) realized with applied force of 20 kN on 70 × 70 mm^2 grating.