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Development of calibration artefacts for x-ray radiographic and micro-CT metrology

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Abstract

This paper presents an artefact manufactured for the purpose of calibrating x-ray micro-CT metrology systems. The artefact proposed here is manufactured using photolithography and reactive-ion processing tools. Features on the artefacts are comprised of closed boundaries with material removed to a fixed depth within the boundary. Artefacts can have one of two feature sets of patterns categorized as first-degree and multi-degree. The first-degree are polygonal shapes and the multi-degree have a boundary edge following a quadratic or cubic curve. Scanning white light interferometry is used to map the artefacts and the data is analysed using a custom MATLAB algorithm to calculate the dimensions with associated uncertainties. For volumetric CT measurements, a three dimensional artefact has been made by bonding multiple planar artefacts. The stacked layers are placed in a specific order with orientation and bonded at 1000 °C to create a solid cube. To evaluate interface adhesion, samples were subjected to mechanical testing to determine the durability of the bonds. The work has resulted in both planar (4 mm x 4 mm) and cube (4 mm x 4 mm x 3.5 mm) x-ray calibration artefacts that will be evaluated in future studies.

Keywords: Metrology, x-ray, CT, micro-CT, radiography, calibration, artefact, bonding, microfabrication, precision

1. Introduction

Interest in using x-ray to perform metrology has quickly grown in popularity as computer processing power has increased. With most metrology instruments, some method to calibrate these systems comes in the form of a physical artefact. For instance, coordinate measuring machines (CMM) and micro-CMM's often use ball bars, ball plates, spheres, precision step gages, and more [1]. Some of these CMM artefacts have been adapted for calibrating large volume x-ray CT systems, however when the feature sizes reach sub-millimetre, new artefacts must be specifically designed with micro-CT in mind. One such artefact called a resolution coil plate uses different densities objects (Shelley Medical Imaging Technologies) [2] while another produced by QRM Gmbh uses two small etched silicon chips placed inside a protective capsule [3]. This works describes the development of two etched silicon planar artefacts, one with first-degree features (i.e. polygons such as rectangles and triangles) and another with second and third-degree polynomial features (i.e. at least one side of the feature is constructed using a quadratic or cubic function). Finally, we explore bonding multiple planar artefacts to construct cube artefact with internal voids that were measured beforehand.

2. Manufacturing and Metrology

The production of small x-ray artefacts borrows from the semiconductor industry by using photolithography with dry etching to manufacture microscopic features onto silicon wafers. This project can be broken into three stages: planar artefact manufacturing, metrology, and cube artefact bonding.

2.1. Planar Artefact Manufacturing

Planar artefact manufacturing starts with cleaning multiple double sided polished silicon wafers using a 3:1 solution of sulfuric acid and hydrogen peroxide, commonly referred to as a piranha solution. The piranha solution is monitored for 20 minutes and maintained at 90 °C with a hotplate. The wafers are rinsed using deionized water and dried with nitrogen before spin coating Shipley 1813 photoresist. The photoresist is soft baked, exposed with UV through a photomask, and developed to remove the exposed photoresist. Next, the wafers are etched with a STS Advanced Silicon Etcher (ASE) running the Bosch process to create vertical sidewalls. The last two processes involve removal of the remaining photoresist with ozone and cutting the wafer to create the individual artefacts with a dicing saw.

2.2. Artefact Metrology

Prior to performing dimensional metrology of an artefact, the artefact is cleaned with a piranha solution, rinsed with deionized water, and dried with nitrogen. To remove water spots still present on the surface, the artefact is rinsed with isopropyl alcohol and dried again with nitrogen. After cleaning, the artefact is securely held with silicon elastomer in a sealed container for storage and transfer. A scanning white light interferometer is used to measure the entire artefact with a 20× objective by stitching approximately 180 sections with each section containing a 20 % overlap of the adjacent maps. The resulting file is saved and imported into MATLAB where a script locates and saves every feature as an individual CSV file. Another script loads a CSV, creates a binary mask of the feature to find the feature's border, locates feature corners and edges, and finally performs dimensional analysis with uncertainties.

2.3. Cube Artefact Manufacturing

To manufacture a cube artefact, multiple planar artefacts that have been measured are returned to the clean room. A PTFE holder was machined to organize and allow for cleaning. Two first-degree artefacts, two multi-degree artefacts, and three blank pieces are loaded into the holder and submerged in piranha solution. After 20 minutes, the holder is removed and placed into a beaker of deionized water with continuously running water. The water is dumped after two minutes and this is repeated three times. While the holder and artefacts are submerged, the cube artefact is assembled by alternating between artefact and blank, with artefacts facing outwards as shown in Figure 1. The reason for placing the two distal planar artefacts outwards is to enable the cube artefact to still be measured with other line-of-sight non-contact instruments for direct comparison to the original measurement data.

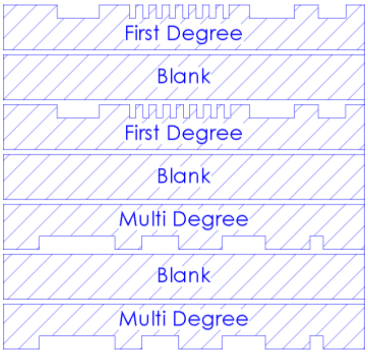

Figure 1. Stacking order and direction the planar artefacts face for construction of the cube artefact.

Once stacking is finished, the artefact stack is removed from the water, placed on a quartz disk, and aligned using custom manufactured tweezers. The quartz disk and stack are moved to the vacuum furnace where a second quartz disk with an attached tungsten mass are carefully added to the top of the artefact stack to apply constant force throughout the heating process. Once aligned and balanced, the vacuum furnace is closed set to 1000 °C for 1.5 hour. Finally, the cube is cleaned with deionized water, isopropyl alcohol, and nitrogen before final inspection of the outer facing features.

3. White Light Interferometer and Cube Bonding Results

After optimizing the parameters of the deep reactive ion etch, two artefact patterned wafers were etched depths of approximately 9 µm and 18 µm. After being diced and cleaned, first and multi-degree artefacts from the 9 µm group were picked out and transferred to the scanning white light interferometer. Images of two artefacts are shown in Figure 2.

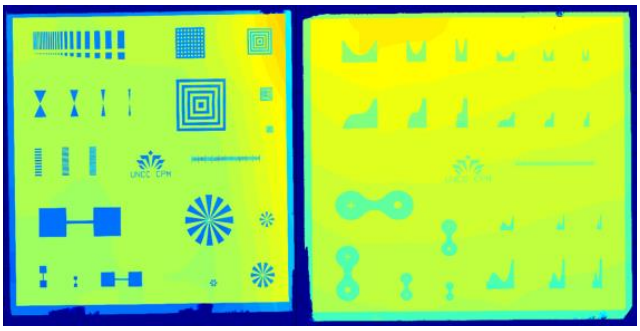

Figure 2. First and multi-degree planar artefacts measured by a white light interferometer with a lateral resolution of 407 nm.

To test the bonding process, different pieces were chosen, cleaned, and heated to 1000 °C for 1.5 hours. Once cooled, the cubes was removed from the vacuum furnace, visually inspected, and subjected to light force from prying and being dropped from various heights. Two of the test cubes are shown in Figure 3.

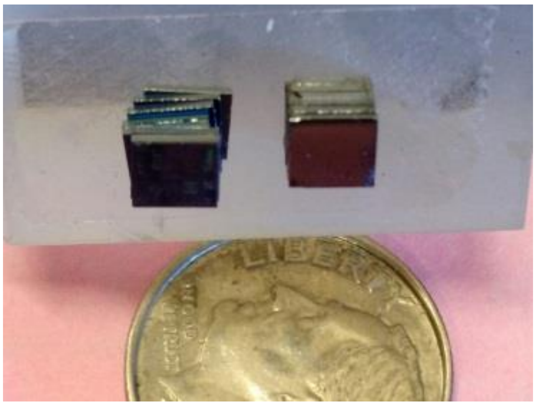

Figure 3. Two bonded cubes used for testing. (Left) Alignment problems occurred when the furnace shield closed. (Right) Cube strongly bonded 6 of the 7 pieces, while the 7^{th} piece failed during the pry test.

4. Conclusions and Future Work

Through the use of semiconductor fabrication, two types of artefacts to be used in x-ray metrology were successfully manufactured. The planar artefact was designed for radiographic imaging, but can be used for calibration of other optical systems while stacking planar artefacts together yields a cube artefact designed for micro-CT metrology. The benefit this cube artefact has over current micro-CT artefacts is that it is composed of only silicon with internal voids that have been measured. Some future work will be developing an improved method that both provides clamping force and retains the alignment during the bonding process, and finally, comparison of micro-CT data to the data obtain using a white light interferometer.

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