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LARGE–AREA 3D SURFACE STANDARDS WITH POLYIMIDE FOR CALIBRATION OF STYLUS AND OPTICAL PROFILOMETERS

Ladislav Matay * — Robert Andok * — Ivan Kostič *
Gian Bartolo Picotto ** — Marco Pisani **

At present, it is recognized for both research and applications that there is a need of accurate calibration methods and suitable test structures as well as improvements of the instrumentation for better positioning and stability of the probe. In the meantime, for surface thickness measurements of wafers, layers and films, for step height and groove depth made by capacitive instruments, as well as for stylus and optical profilometers, there is a need of multistep height standards that could be used for calibration of various magnifications of such instruments as well as for calibration of the vertical magnification of the stylus and optical profilometers. The overall objectives of the work described herein are to develop new microtechnological processes leading to fabrication of 3D standards with polyimide (PI) for scanning probe microscopy and step-height measurements.

K e y w o r d s: 3D standards, polyimide, profilometers, calibration, RIE, wet etching

1 INTRODUCTION

Measurement instruments such as stylus, optical and other profilometers, have matured to a level where they can offer indispensable information about the geometry and structure of various surfaces [1]. To minimize the errors induced by these measurement instruments one must calibrate them at various magnifications. This calibration should be made by means of a number of step-height standards ranging from tens of nanometers up to tens of micrometers [2].

For the calibration of profilometers and scanning probe microscopes to be used at various vertical magnifications, the proposed multistep standards in this work may replace a number of samples having normally a single step [3]. Polyimide covered by a suitable metallic layer has shown to be a suitable solution to the problem described above.

These multistep height standards have been designed to have a platinum surface with a number of wide and flat steps of heights ranging from tens of micrometers down to hundreds of nanometers. The standards have been made on silicon wafers as well as on hard-glass using highly-stable polyimide combined with thick film preparation technique, deposition of a thin metal film and plasma dry etching (RIE) through the contact mask. As such surfaces are metalized at the end of their process flow, their step-height can be measured by interferometry, optical profilometry, or stylus-probe profilometry techniques.

Such a proposed project of the 3D standards preparation aims to:

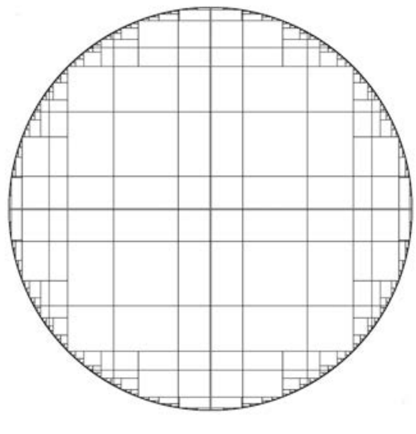

Fig. 1. Partitioning of one of the circles for 3D surface standards to orthogonal rectangles using the NINI algorithm.

- fabrication of photomasks for large-area 3D surface standards,
- fabrication of suitable structures on silicon and hard-glass consisting of large-area pyramidal steps of defined heights,
- improvements of the existing probe scanning devices equipped with capacitive and inter-ferometric control of displacements, including the error analysis and optimization of the scanner.

* Institute of Informatics, Slovak Academy of Sciences, Bratislava, Slovakia. E-mail: Ladislav.Matay@savba.sk
** CNR — Instituto di Metrologia "G. Colonnetti", Torino, Italy. E-mail: g.picotto@imgc.to.cnr.it

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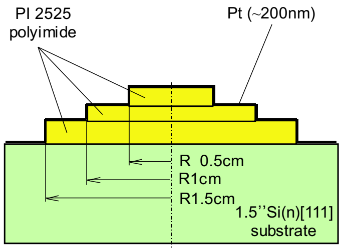

Fig. 2. Schematic of the PI standard prepared by wet chemical etching.

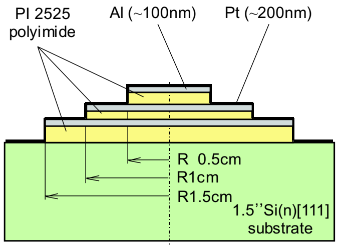

Fig. 3. Schematic of the polyimide standard prepared by the RIE.

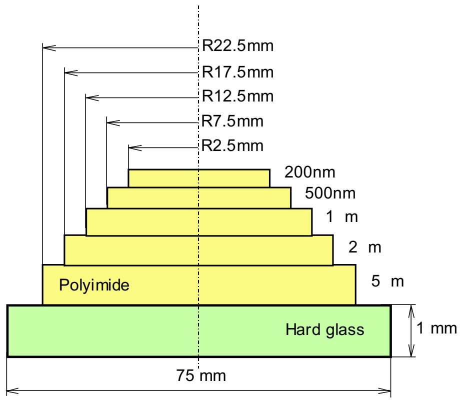

Fig. 4. Cross-section of the pyramidal structure of the 3D standards prepared on the hard glass.

2 MATERIAL AND EXPERIMENTAL PROCEDURES

Five different photomasks were fabricated for the large 3D surface standards: 3″ borosilicate glass ("HOYA") was used for masks fabrication (all the masks consisted of a Cr layer and an antireflective coating); Electron Beam Lithography (EBL) direct writing technique was used to fabricate these photomasks; and the negative-tone e-beam resist NS-125 (Microimage Technologies, Inc.) was used for direct writing of the masks. Figure 1 is showing the design of the mask for such a 3D standard. The maximal roughness of the circle boundaries was set to 5 microns. The whole area of the circle was decomposed into orthogonal rectangles using the NINI [4] algorithm technique. Such rectangular areas were then directly exposed by the electron-beam pattern generator ZBA 10/1 (Carl-Zeiss).

In our experiments the PI2525 polyimide [5] (DuPont) has been used and tested for its excellent thermo-mechanical and other properties. After the polyimide was deposited onto the substrate by standard spin-coating technique, it was soft baked (85 °C/5 min) and subsequently hard-baked to reach its required properties for the use as a large-area surface standard. Hard baking was carried out on a hot-plate in nitrogen atmosphere, at temperatures from approx. 80 °C up to 350 °C. The temperature rise was set to be max. 0.8 °C per minute to reach good hard-baking conditions. Then the samples were cooled down to ambient temperature. In the next step, patterning of the polyimide samples took place. Two different approaches have been chosen to fabricate 3D surface standards: wet chemical etch process and the RIE process. Figure 2 is showing in cross-section an example of the completed PI (3-layer) standard prepared by the wet chemical etching technique [6]. The thickness of respective PI layers varies according to requirements and is attained by appropriate spin-spinning.

A series of samples with 3D surface standards have been fabricated using both wet chemical and RIE etching techniques to work out the best sample process flow, sufficient for our requirements. The main advantage of the wet chemical process flow was that it required less technological steps and did not need any additional metallic (Al) layers (used as hardmasks) comparing to the RIE process flow. However, the main advantage of the RIE process flow in as much as the wet chemical process flow was in its cleanliness and high anisotropy. That was also the reason why RIE has been chosen as the chief technology in 3D standards fabrication. Figure 3 is showing in cross-section a schematic of such a PI standard prepared by the RIE etching technique in O_2 plasma.

For RIE etching, the Microsys 350 (Roth&Rau) RIE equipment was used. For process evaluation, visible-light microscopy as well as Scanning Electron Microscopy (SEM) were used, completed by profilometric and ellipsometric measurements of the 3D standards. For process quality evaluation, high-resolution Field Emission SEM (Hitachi S-800) was used. The samples were further examined by the Taylor-Hobson Talystep profilometer in order to obtain basic information of the quality and homogeneity of the samples. More precise surface quality measurements take place at our partner's laboratories (CNR, Italy) and are part of further investigation.

At the beginning of our experiments, only 3-layer technique was used to eliminate the necessary technological steps of the polyimide layer preparation. These test samples were prepared on 1.5″ silicon substrates. In the future steps the silicon substrates were replaced by a plan-parallel optical window of 75 mm diameter and 1 mm thickness, made of BK7 hard glass, to meet quality requirements. This design modification is seen in Figure 4. In this modification of the pyramidal 3D structures the largest of the circles was chosen to be of a diameter of 45 mm and thickness of several microns, ending with the

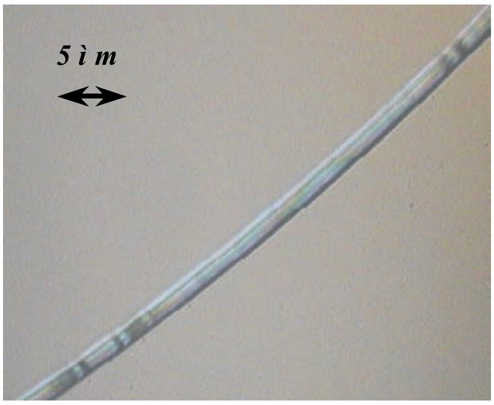

Fig. 5. Boundary step between two PI layers (obtained by the optical microscope through a polarizing filter).

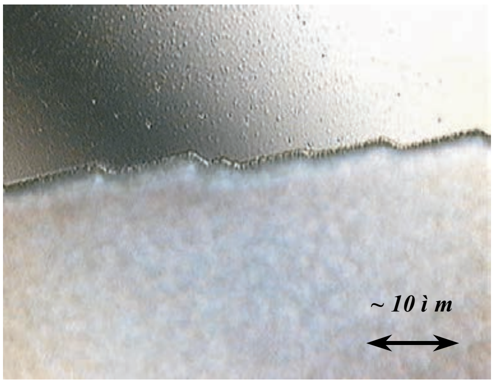

Fig. 6. Boundary between the Si substrate and the first (bottom) PI layer, obtained by optical microscopy.

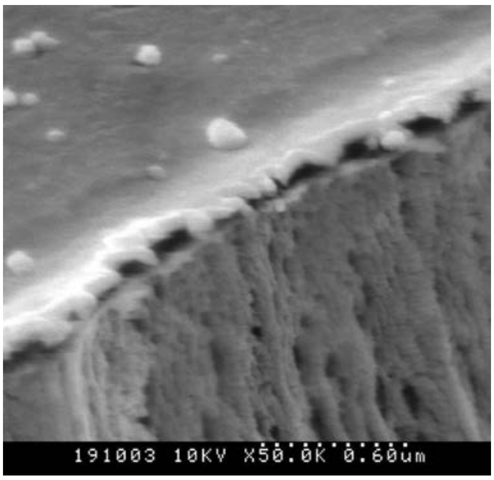

Fig. 7. SEM picture of the Pt/Al/PI boundary. The small grainy particles (biggest of them of diameter of about 300 nm) above the two metals may possibly come from the RIE process. These particles are negligible for application but can be further eliminated by setting appropriate plasma conditions.

smallest structure with diameter of 5 mm and thickness of approx. 200 nm, that is altogether 5 structures placed on the top of each other.

3 RESULTS AND DISCUSSION

3.1 Results from the wet chemical etching

The wet etch process flow of the 3D standards accounted several differences when compared to that of the Reactive Ion Etching (RIE) process flow. However, the results obtained from these two processes are shown to be quite similar. The main disadvantage of the wet chemical etching appeared in that it is an isotropic process, a less clean process and, that it often underetched the uncovered polyimide layers from their side. Figure 5 is showing the boundary step between two PI layers prepared by wet-chemical etching process flow technique.

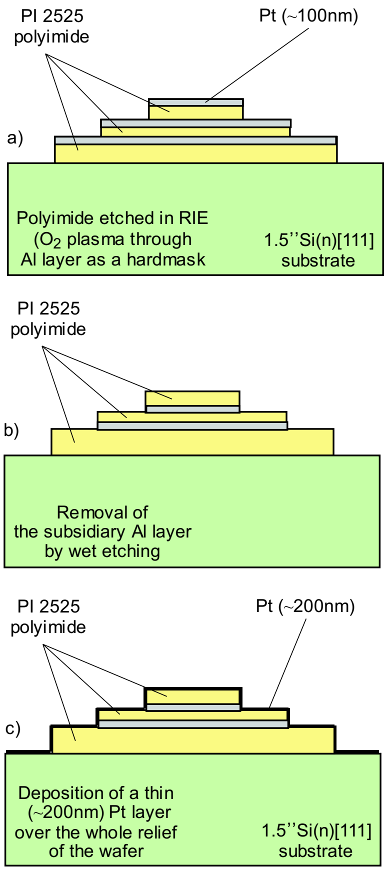

Fig. 8. Schematic of the improved RIE process flow with removal of the subsidiary Al layers before Pt deposition took place.

3.2 Results from the RIE etching

The main advantage of the RIE process flow comparing to that of wet chemical etching is that it is a clean and highly anisotropic process. To etch all the polyimide layers for 3D surface standards, the following RIE plasma parameters have been used: 100 W RF power, and 20 sccm

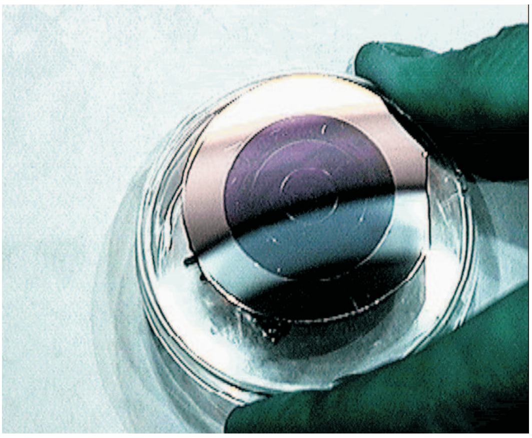

Fig. 9. Completed 3D standard on Si substrate with 3 PI layers.

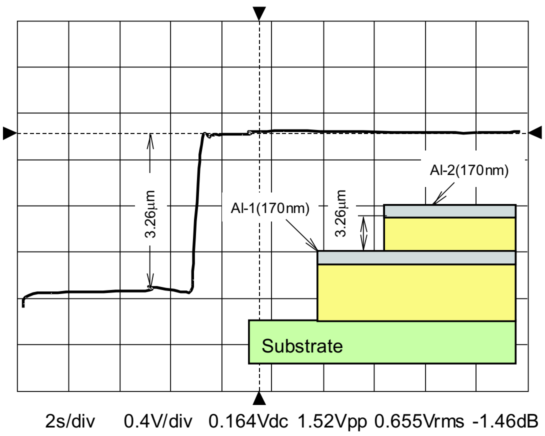

Fig. 10. Profilometric curve showing thickness of the bottom PI layer.

of O2 flow. Figure 6 shows the boundary between the silicon substrate and the polyimide layer.

Inspecting in more detail the area of the Pt/Al/PI boundary (Fig. 7) one can see that this metal-to-PI junction tends to split at the edges of the etched polyimide layer. There have been several interpretations why this happens: One explanation may be due to the mechanical stress between the two metals (Pt, Al) and the PI layer (or, in general, due to the stress of the multilayer). However, as suggested later, the stress should not play such a significant role in this case. This splitting of metal-to-PI junction is supposed to be due to the RIE process when in O_2 plasma not only polyimide but also the Al hardmask layer is bombarded by ions (for Al the etch rate is $\sim$ 0.8 nm/min). Thus the Al layer protecting the polyimide becomes underetched on its edges with polyimide while this effect is most redoubled between two adjacent polyimide layers of the 3D standard (Fig. 7).

In order to eliminate the herein mentioned problem, removal of redundant Al layers was proposed to take place before deposition of the final Pt layer over the whole relief of the wafer. Figure 8 well illustrates this process flow. Aluminum that was left below the PI layers is well planarized by the polyimide layer.

Out of a series of samples prepared during the first stage of the experiments, the thickness for all three layers of the 3D standards was in the range of 5.2 to 5.4 μm. For future work, photomasks for large-area 3D standards were prepared in agreement with the modified structures proposal (Fig. 4). Figure 9 is a picture of a completed 3D standard on Si substrate with 3 PI layers before it was covered by a Pt layer.

Figure 10 is a Talystep profilometric curve for getting preliminary thickness and flatness information of the 3D standards. Here, the distance between two polyimide layers (layer 1 and 2) can be seen, which was estimated to be 3.26 μm (without Al capping metallization).

Ellipsometric measurements of the PI surface morphology of the samples have shown that two PI layers give lower mean values of surface unevennesses ($\langle\Delta_{\rm PI}\rangle$ = 16.8 nm) comparing to a single PI layer ($\langle\Delta_{\rm PI}\rangle$= 26.2 nm). This can be explained by the fact that the second PI layer planarizes the adjacent layer lying below it, leaving the resultant sample surface smoother. The maximum measured peak-to-peak difference of these unevennesses was only up to 40 nm within the specific central area of the sample, which is about 1.5 % of the whole polyimide thickness. The mean value of the surface roughness was 0.25 % of the whole polyimide thickness.

4 CONCLUSIONS

In order to evaluate the technology process, visible-light microscopy together with high-resolution SEM were used and finally completed by essential profilometric and ellipsometric measurements of the 3D standards. These have proved that the final quality of polyimide surfaces strongly depends on precise polyimide deposition as well as its subsequent thermal cure (soft- and hard bake). The required PI thickness was controlled by precise automated spin-coating technique. Also the PI surface morphology was examined and unevenness was achieved within $\sim$ 20 nm precision for the whole polyimide layer surface. Polyimide thickness accuracy and repeatability of the steps were estimated to be within about 80 % when the same spinning and curing conditions have been preserved. Precise profilometric and optical measurements are to be supplemented by the project partner during the further co-operation. RIE was chosen for etching of the PI steps for its cleanliness, minimal underetching and high anisotropy.

Acknowledgements

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Ladislav Matay (Ing) was born in Piešťany, Slovakia, in 1950. He received his masters degree in solid state physics from the Faculty of Electrical Engineering of the Slovak University of Technology, Bratislava, in 1974. Since his graduation he has worked at the Institute of Informatics of the Slovak Academy of Sciences (SAS) in Bratislava. His research activities include application of thin polyimide films in microtechnology, mainly in the field of microsystems technology and micromechanics.

Robert Andok (Ing, PhD) was born in Veľký Krtíš, Slovakia, in 1973. He received his masters degree in solid state physics from the Faculty of Electrical Engineering of the Slovak University of Technology, Bratislava, in 1997. From 1998 he was a PhD student at the Faculty of Electrical Engineering STU, in specialization of microelectronics; and since 2000 he has worked at the Institute of Informatics of the SAS in Bratislava, in the field of microsystems technology.

Ivan Kostič (BS, RNDr) was born in Rožňava, Slovakia, in 1955. He received the BS degree in physics from Moscow State University, USSR, in 1980, and his RNDr degree from Comenius University in Bratislava in 1982. He joined the Laboratory of Electron-Beam Lithography of the Institute of Technical Cybernetics at the SAS, Bratislava, where he was involved in the research and development of e-beam technology. Currently he continues his work in microlithography and microfabrication at the Institute of Informatics of the SAS.

Gian Bartolo Picotto received the degree in physics from the University of Torino, in 1987. He is currently a researcher at the CNR-Instituto di Metrologia, where he has been involved in the field of optical frequency standards and laser spectroscopy. Since 1990, he has been responsible for the research on submicron surface metrology. His main interest are in scanning probe microscopy, stylus profilometry, laser interferometry, and capacitance-based displacement transducers. He is now responsible for the CNR-IMGC of the european thematic network "SPMet - Calibration of SPMs". He is author and co-author of over 50 published papers. y **Marco Pisani**, born in Milan (Italy) in 1963, received the doctor title in Physics, and the PhD in Metrology. He worked at CERN (Geneva) mainly on silicon detectors for high energy particles, then at IMGC (Torino) on frequency stabilization of CO_2 lasers. Since 1996, as a researcher at IMGC, is engaged in the nanometrology field, namely on micro-actuators, capacitive sensors and interferometers for scanning probe microscopy.