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Verification of thickness and surface roughness of a thin film transparent coating

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Abstract

Thin film coatings are extremely interesting for industries, where there is a need to protect a highly accurate surface which has tight dimensional tolerances. The topic is important both in the production of new metallic tools and repair applications. In both applications it is vital to have a specific knowledge about coating thickness and roughness. In the present paper a novel application of a transparent HSQ coating is presented. Furthermore the thickness and roughness of the transparent coating with nominal thickness of 1 µm is measured in the presence of surface roughness of the substrate. Measurements were done using AFM and a precision 3D mechanical stylus instrument.

1 Introduction

Polishing of metal tools and parts is a manual process with many drawbacks and risks, including being detrimental to worker health. Therefore it is relevant to investigate alternative methods for polishing, such as methods relying on coatings. Thin film Hydrogen Silsesquioxane (HSQ) is commonly used in the semiconductor industry in the manufacture of integrated circuits (ICs), both as a low-k dielectric and as a planarization material to fill in the gaps between metal wires and spatially separated components [1]. HSQ is an unstable, cage-like silane hydrolyzate, which cures to form a solid, amorphous quartz layer. It can be obtained as a pure material or in liquid form prepared for IC manufacture, which can be applied via typical means of coating, leading normally to reductions in the surface roughness [2]. The focus of this paper is the geometric characterisation of the thin HSQ coating applied on a flat surface of steel.

2 Measurement of coating thickness

A flat surface of a gage block made of steel has been used as the base surface for application of the HSQ coating. The HSQ coating was applied using an ultrasonic spray nozzle, and then it was cured at 450°C for 1 hour. For the sake of height comparison after coating, a portion of the surface was masked before coating. So a step was created which was the subject of height measurement. The instrument used was Form TalySurf 50 inductive stylus profiler with 0.6 nm height resolution, 250 nm lateral resolution and 2 µm tip radius capable of 3D movement. The stylus covered a length of 1.5 mm with a width of 50 µm. Figure 1 shows the result of measurement after applying a Gaussian filter (λs = 2,5 µm) and plane correction in software SPIP, version 6.0.13 (Image Metrology A/S, Horsholm, Denmark).

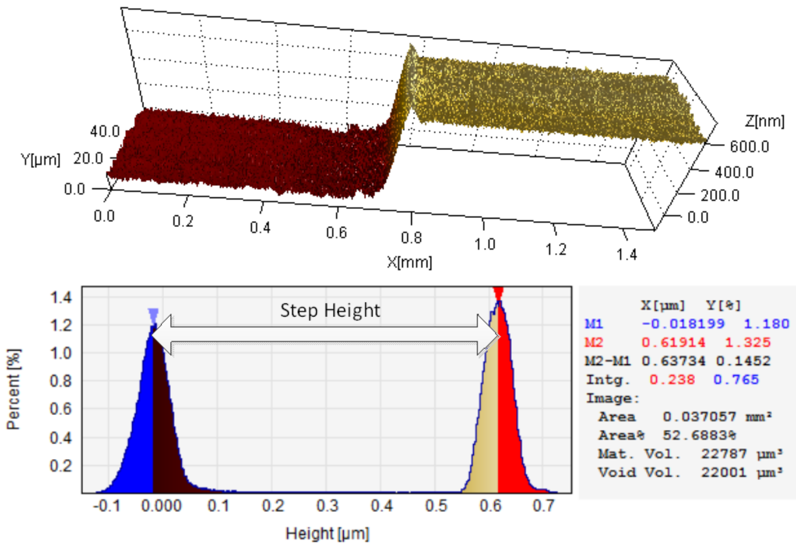

Figure 1: Height measurement of HSQ layer by 3D stylus including the height histogram

An area in the middle of the step (about 200 µm length in each side) shows some shape irregularities which are mainly due to the reflow phenomenon created on the step. But the remaining area which is sufficient for measurement (about 500 µm in each side) covered in this test shows a quite homogeneous height distribution of 0.6 ± 0.07 µm with respect to base surface (left distribution in the histogram) which itself exhibits ±0.12 µm height deviation. An improvement in surface roughness after coating is demonstrated by comparison of different distributions although mechanical stylus might not be the proper instrument in this range of roughness.

An additional effort to support coating thickness measurement has been performed to get a cross sectional information about the coating layer through Jeol 5900 scanning electron microscope (Fig.2). In order to recognize the HSQ layer, a thin layer of Nickel (400 nm) was deposited onto surface of the work piece using Physical vapour deposition (Metallux-ML18). Compared to the large coverage of the stylus (1500 µm), the 1:1 SEM picture covers only a very small portion on the surface (40 µm). The coating thickness variation in the presence of very high lateral resolution of SEM gives a very different distribution although the nominal value is still in conformity to what is measured by mechanical stylus.

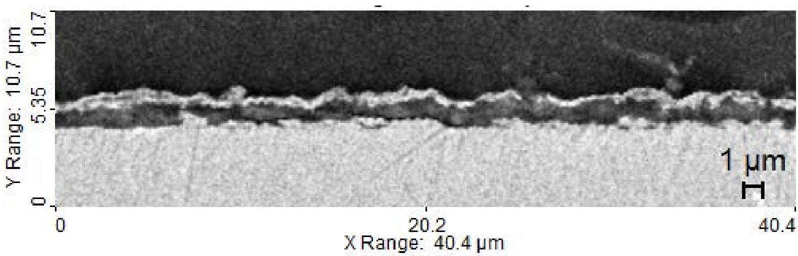

Figure 2: SEM image of the substrate (bottom) coated with HSQ (middle black layer) and an additional Ni layer (white thin layer in the middle of the picture)

3 Measurement of roughness

Roughness tests were done using atomic force microscope [3]. Scanning areas of 50X50 µm were selected on both uncoated and coated surfaces (Fig.3). The 3D visualisation of the surfaces shows a considerable improvement in surface roughness after coating (Fig.3A). The nature of the coated surface is completely different to the initially polished one. The 3D roughness parameters as well as the histogram (Fig.3B) demonstrate this change (S_a reduced from 13.7 to 4.7 nm and S_z reduced from 183.7 to 56.2 nm). The bearing curve parameters clearly show an improvement on the surface in a lower height distribution. The specific values of peak, valley and core roughness parameters are all reduced simultaneously (Fig.3C) meaning that the reduction of height is distributed evenly between peaks, core and valleys.

A parallel roughness study has been performed by a 2D mechanical stylus (2 µm tip radius) with 12 repetitions covering an evaluation length of 1.25 mm. After applying Gaussian filters (λ_s = 2,5 µm, λ_c = 250 µm) it resulted in 10.2 ± 0.4 nm R_a for the uncoated surface and 8.75 ± 0.4 nm R_a for the coated surface. An examination of the background noise of the stylus on a reference plain glass showed 4 nm R_a which makes it difficult to rely on stylus results on this range of roughness.

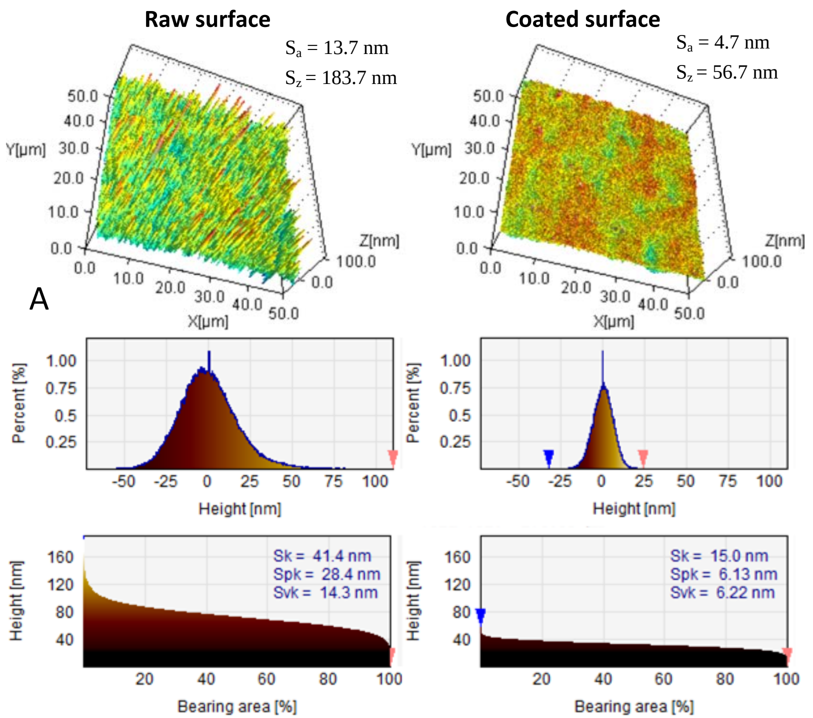

Figure 3: 3D surface characterisation of raw (left) and HSQ coated surface (right): visualisation (A), height histogram (B) and bearing curves (C)

4 Conclusion

The study toward surface characterisation of the HSQ coating in the presence of a base roughness shows that the thickness of the coating is 0.6 ± 0.07 µm based on long coverage of 3D stylus (1500 µm), while short coverage surface variation (50 µm) at each side of the step as measured by AFM gives 183 nm R_z on the uncoated surface and 56 nm R_z on the coated surface. The smoothening effect of the coating is obvious through the study which is the subject of an upcoming publication by the authors. SEM micrographs compared to AFM and stylus instruments exhibit a different view of thickness variation because of the high lateral resolution. The advantage of SEM is the possibility to observe inner and outer coating surfaces at exactly the same point on the surface which is quite valuable to study the correlation of coating roughness based on a certain substrate roughness.

Acknowledgment

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