Mapping surface properties of sinusoidal roughness standards by TPM

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Abstract. We report our investigation on the surface properties of sinusoidal roughness standards made from pure electroformed nickel. Two specimens having a sinusoidal profile with nominal Ra of 0.36 μm and a peak spacing of 25 μm are chosen for this investigation. One specimen is further treated with a hard protective coating of nickel-boron. The surface topography, friction, hardness and Young’s modulus of the specimens were measured by a novel instrument, the multi-function Tribological Probe Microscope (TPM). The results show that hardness of these two specimens is 14.1 GPa for uncoated specimen and 25.7 GPa for the coated one, while the Young’s modulus is 188 GPa and 225 GPa, respectively. The ramping force was set to 3 mN for both the specimens and the effect of the tip penetration was investigated by comparing the topography measurements before and after hardness mapping. It has been found out that there is no significant change in the averaged profiles over the scanned area, which indicates the topography distortion seen in the multi-function mapping, is recoverable. Cross correlation between topography and its corresponding hardness/Young’s modulus has been carried out and the result will be discussed in the paper.

1. Introduction

There is a growing requirement in high precision engineering, bioengineering and nanotechnology, for surface measurement of not only the surface geometrical features but also of such surface properties as friction, contact stiffness, hardness, and adhesion etc. It has been widely recognised that the surface of material, particularly the topmost layer, plays an important role in its function and durability. Most materials at light loads exhibit different properties from those expected of the bulk materials. This difference can be even enhanced by various techniques such as plating, anodising, plasma nitriding, and carburising, etc [1]. At Warwick University, a novel instrument, the multi-function tribological probe microscope (TPM) has been developed to provide property mapping for topography, friction, hardness and Young’s modulus of a surface. As the measurement is based upon point-by-point scanning, the measured four functions are linked in space and time. This capability enables us to carry out a cross correlation between these functions, so as to identify how one function affects another and ultimately to be able to optimise the function/performance of a component. The capability of TPM has been demonstrated on a wide range of surface materials and details can be referred to early reports [2-5]. Roughness standards are widely used in industries for instrument calibration and validation, workshop comparison, inspection, and quality control. Roughness standards are of different types of
profiles and topography and different ranges from micrometer down to nanometre scales of roughness height. In this paper we report our investigation on the properties of sinusoidal roughness standards by using TPM to see how the coated and uncoated specimens react under a different contact load.

2. Standard specimen preparation
The roughness specimens used in this investigation are made from pure electroformed nickel, as replicas of an original master specimen which was itself produced as an interferographic pattern in a thin layer of photoresist on a polished glass plate. The photoresist is first covered with a PVD layer of silver to make it electrically conductive, the whole glass plate is then connected to the negative power supply (-6V), and then immersed in an electrolyte bath containing a nickel salt solution. The bath also contains a solid nickel anode connected to the positive power supply (+6V). In the process, a layer of nickel will gradually be electrodeposited onto the silver, and it can be removed from the silver provided that there has been suitable chemical passivation of the silver surface prior to immersion. This first replica of the silver surface will be a faithful but negative (inverted) copy of the original surface profile. This negative replica is then immersed in the electrolyte bath in exactly the same way as the silvered glass plate, and the replica that then forms on its surface will be an identical positive replica of the original master. The positive replica is the specimen used in the tests.

The interferographic technique involves exposing the thin layer of photoresist to a pattern of laser-produced interference fringes, which consist of sinusoidal variations of light intensity. The molecules of the photoresist are made more soluble by the action of light that breaks down large polymer chains into smaller molecules. By doing this, the variations of light intensity is converted to the variations of solubility, thus the photoresist is developed in a form of a corrugated surface profile. The spacing of the sinusoidal corrugation pattern is the same as that of the interference fringes. After silver deposition and electroforming as described above, the sinusoidal corrugations are reproduced in nickel. Using this technique with laser light of blue and near UV wavelengths, sinusoidal roughness specimens with spacing from 25 μm down to 0.25 μm can be produced reliably.

Two specimens labeled as EL and XEL are chosen for this investigation and they both have a nominal roughness of Ra = 0.36mm and a spacing of 25mm. Specimen EL has a hard top layer of nickel-boron, the other specimen XEL is pure electroformed nickel.

3. TPM system
Fig. 1 shows a photograph of the mechanical system of the TPM. It consists of a sensor probe attached to Z-positioner, a closed loop controlled X-Y stage, and an electronic drive and control system. The specifications of the TPM are:
- **Scan area**: 100 × 100 μm².
- **Force range**: 0.01 ~ 30 mN.
- **Height range**: 15 μm with 0.1 nm resolution.
- **Tip size**: 0.1μm Berkovich diamond tip.

Other specifications and details of the instrumentation and calibration can be found in reference [4].

4. Results and discussions
The specimens were first measured by using a commercial optical profiler WYKO NT2000 for surface topography. The scanned area was set to be 93×112μm² in order to match the TPM’s scanning area. The results are shown in Table1 with parameters of central line average Rₐ, root-mean-square Rₛ, peak to valley Rₚ, and average spacing Sₘ. The multi-function mapping was then carried out by TPM.
Table 1 Measured results by WYKO NT2000 and TPM on two specimens.

| Sample | $R_a$ (µm) | $R_q$ (µm) | $S_m$ (µm) | $H$ (GPa) | $E$ (GPa) | Comments                        |
|--------|------------|------------|------------|-----------|-----------|--------------------------------|
| XEL    | 0.361      | 0.407      | 1.51       | 25.6      |           | WYKO NT2000                     |
|        | 0.334      | 0.384      | 1.18       |           |           | TPM topography only             |
|        | 0.354      | 0.402      | 1.20       | 14.1      | 188.4     | TPM Multi-function              |
|        | 0.331      | 0.376      | 1.19       |           |           | After indentation                |
| EL     | 0.353      | 0.399      | 1.60       | 25.3      |           | WYKO NT2000                     |
|        | 0.324      | 0.371      | 1.14       |           |           | TPM topography only             |
|        | 0.358      | 0.409      | 1.16       | 25.7      | 225.3     | TPM Multi-function              |
|        | 0.323      | 0.370      | 1.15       |           |           | After indentation                |

with a scanning force of 0.1mN and a ramping force of 3mN over an area of 100×100µm². Fig. 2 shows the mapped images of topography, hardness and Young’s modulus for both the specimens. The averaged parameters are listed in Table 1. The nickel-boron coated specimen shows a large increase in both the hardness and Young’s modulus, over 80% increase in hardness and about 20% in Young’s modulus. From Fig. 1, for the uncoated XEL, there seems no correlation between the topography and hardness or Young’s modulus. However, for the coated specimen, three images show some kind of link between them. A cross correlation was carried out between these functions for both the specimens. The results show that a high correlation of about 0.9 was found between hardness and Young’s modulus and a negative correlation about -0.3 is between topography and hardness/Young’s modulus for the coated specimen. This negative correlation between topography and hardness or Young’s modulus has been found on other type of surfaces [6]. Fig. 3 shows the detailed profiles of the averaged topography and hardness for the coated specimen in order to demonstrate the effect on its hardness. It seems that the slope of the profile modulates the hardness. As the topography was measured at the same time as the hardness and Young’s modulus, there is certain degree of distortion, as shown in Fig. 2. In order to see if the distortion was made permanently by TPM during the indentation mapping, each specimen was scanned for topography only before and after the multi-function measurement. As the scanning stage is feed loop controlled, the relocation repeatability is better than the tip resolution of 0.1µm. Thus a comparison between these images can be made directly.

Fig. 2 Multi-function mapping for topography (a1, a2), hardness (b1, b2) and Young’s modulus (c1, c2). The subscript 1 represents for uncoated specimen (XEL) and subscript 2 for the coated one (EL).
Fig. 3 Averaged profiles of topography and hardness for Specimen EL.

Fig. 4 Comparison of topography profiles obtained before, during and after the force ramping on specimen XEL.

Fig. 5 Topography images obtained (a) before and (b) after the force ramping mode from Specimen XEL.

Fig. 4 shows the topography profiles obtained from the uncoated specimen by TPM before, during and after the multi-function measurement (force ramping mode). The distortion made during the force mapping is clearly shown in the plot. The corresponding parameters listed in Table 1 show that roughness parameters obtained from the force ramping mode are slightly higher than those taken with only topography. However, it is interesting to note that this distortion was significantly recovered by comparing the before and after profiles. The corresponding images for the two profiles (before and after) are shown in Fig. 5.

In conclusion, we have demonstrated that the TPM is capable of measuring the geometrical and mechanical behaviours of the chosen roughness standard specimens under a controlled condition.

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