SCANNING PROBE MICROSCOPY: MEASURING ON HARD SURFACES

Milan MATĚJKA a, Michal URBÁNEK a, Vladimír KOLAŘÍK a

a Institute of Scientific Instruments of the ASCR, v.v.i., Královopolská 147, 612 64 Brno, Czech Republic, mmatejka@isibrno.cz

Abstract

During a measurement by scanning probe microscopy (SPM) an image artifacts can appear in a measurement data. The source of image artifacts during an SPM measurement could be in parts of the SPM tool: mechanical system, piezoelectric crystal, scanner electronic. However, the main source of image artifact is the probe tip geometry and properties of the sample. For example, probe wearing, which occurs during the contact measurement on a sample with a hard surface, could result in heavy probe shape change, causing probe-related image artifacts. Measurement could appear problematic on a sample with periodical relief structure (e.g. gratings with sub 10 µm periodicity) prepared in hard materials (e.g. silicon), when the structure height is greater than about 500 nm. In this case, probe can easily get struck during the scanning, on the hard surface as well as at the high aspect ratio relief structure, causing image artifact thus reducing measurement quality. This contribution describes a methodology for hard surface sample treatment, so the probe-related artifacts should be minimized. We used a thin polymer film coatings and the SPM image artifact analysis of such modified samples with emphasis on the measurement quality and the probe wearing.

Keywords: scanning probe microscopy, atomic force microscopy, hard surface samples measuring, image artifacts, polymer coatings.

1. INTRODUCTION

The scanning probe microscopy (SPM) is one of basic and universal methodology for nano and microtechnology research [1,2]. It allows imaging of surface topography down to atomic level, also offers a wide range of information about investigated material (mechanical, tribological properties). This technique is based on interaction of sharp tip (probe) on cantilever with the sample surface during scanning. The surface information is transmitted by means of deflection of the cantilever. During measurement, the probe tip is the direct contact (contact mode) with the sample material and it wears. The possible wear mechanisms of probe tip include adhesive wear, abrasive wear low cycle fatigue and tribochemical wear [3,4]. Probe wear may increase the tip radius thus reduce the image resolution. In some case when a measured sample contain sharp features with size smaller or comparable to probe tip radius, the measured topography image of the sample then consist of combination of the tip shape and the sample surface, referred as tip-related artifacts [5,6]. When the probe is on contact with a hard surface (e.g. silicon) there is increased wearing of the probe tip mainly due to low cycle fatigue and abrasive wear (depending on the measurement condition). Then the SPM measurement on a sample with high aspect ratio micro features prepared in hard materials could be even more problematic, due to probe tip wear. On top of that sample with complex microstructures require series of measurement on different part of the sample to fulfill its analysis, which means a few tip landing procedures (low cycle fatigue wear) and scans (abrasion wear). All this can increase probe wearing greatly and will consequently introduce image artifacts, thus reducing measurement performance over time [7]. There is different approach how to reduce probe wearing as is changing mechanical properties of the scanning probe tip e.g. coating probe tips with DLC layers, by use of tip made of monolithic ultrananocrystalline diamond (UNCD) [8], or by use of different type of lubricant coatings as PFPE, PFPTES [9]. We have tried different approach of reducing probe tip mechanical wearing. The methodology arisen from practical experience with a contact SPM measurement on the thin organic layers (such as
polymethylmetacrylate - PMMA). The idea is based on sample treating with organic polymer coating so the probe related image artifact and probe wearing should be minimized.

2. EXPERIMENT

2.1 Test Samples

For experiment the sample containing different types of microstructure patterns (such as diffraction grating, blazed grating ...) was prepared. The base material for the sample was chosen monocrystalline n-type silicon crystallographic orientation (100). The micro features on the sample were prepared by the CF₄ based plasmatic etching of the silicon through soft etching mask made of PMMA layer. The remaining etching mask was removed using chemical solvents (Chloroform, Acetone) and to remove residues the oxygen plasma cleaning was used. The depth of prepared relief micro features in silicon was 1190 nm. This value was obtained using the profilometry device (Taylor-Hobson taylstep).

2.2 Measurement

The measurement of testing pattern went on the atomic force microscope (AFM) brand Pacific nanotechnology. Contact mode Single-crystal silicon probes with symmetric tip (SICON-W, AppNano) were used for study. According to the manufacturer AppNano comp. the probe were made of 0,01-0,025 Ωcm N-type Si (100). The length of cantilever on the probe is 450 µm. Tip height is 14 - 16 µm. The nominal tip radius is less than 10nm. In the first part of the experiment took place a measurement on the uncoated sample. Before stress measurement on the test sample, the probe tip shape was characterised, on for this purpose designated testing pattern TGG1 see Fig.1b (NT-MDT, Moscow) [10]. This was followed by the stress measurement on five different features on the testing sample with micro-relief structures etched in Si. After stress measurement went characterization of probe tip shape on testing pattern TGG1 and TGT1 (NT-MDT, Moscow, see Fig.1a,b) [10]. The entire measurement procedure was repeated with new probe for the testing sample coated with layer of polymethylmetacrylate (PMMA). NanoRule+ software (Pacific nanotechnology) was used to characterize tip and evaluate the tip radius.

2.3 Sample coating

Polymer coating was prepared by the spin coating from the 1% wt. solution of the PMMA (Mw = 950k) in Anisole. The choice of PMMA for sample treatment was based on good experiences with AFM measurement on its layers. Its part played also its easy availability and some experience of its thin layer deposition.

Coating parameters were as follows:

- Spin speed: 8000 rpm
- Spin acceleration: 1200 rps²
- Spin time: 20 s
- Temp: : 24°C

![Fig. 1 The SPM probe shape characterization tests a) TGT1; b) TGG1.](image-url)
Immediately after coating, the sample was placed on a hot plate for drying and hardening of the coated layer (150°C; 300s).

3. CONCLUSIONS

It can be concluded from the results of measurement and its evaluation that the layer of polymer film applied to the sample with micro relief structure has a positive effect on the measuring probe wearing (see Table 1.). The wearing of the measuring probe is about five-times lower when the PMMA layer is applied on the sample than if it is not (see Fig.2). This fact can be explained by reduced probability of probe breaking when the tip approach to the sample surface treated with polymer layer. The reason is quite simple because the polymer layer, even so it is very thin, is much softer and pliable then the hard surface of silicon, so the probe won't break so often. During the scanning the probe is again in contact with polymer interlayer so the probe wearing is greatly reduced. With regard to resolution and conservation of information about the original sample relief structure, the relief height is maintained at a very good rate as well as information about a lateral dimension of relief structures. However, at the bottom of relief structures some small deformation might appear, this could cause some troubles especially when measured elements are of smaller sizes (>1 µm). It should be also realized that by the sample coating we give up the opportunities of obtaining some certain information, such as the original surface roughness, material contrast etc. If is necessary to maintain the sample conductive for certain types of measurement, polymer coating such as PMMA is not appropriate as well. The advantage is that the layer of PMMA is relatively easy to remove by chemical or plasmatic way and the original structure and material can be restored.

Table 1 Evaluation of the AFM measurement

<table>
<thead>
<tr>
<th>Sample</th>
<th>Tip radius * [nm]</th>
<th>a [µm]</th>
<th>b [µm]</th>
<th>h [nm]</th>
<th>Tip radius ** [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>without polymer coating</td>
<td>&gt; 20</td>
<td>3.040</td>
<td>3.074</td>
<td>1185</td>
<td>747</td>
</tr>
<tr>
<td>with polymer coating</td>
<td>&gt; 20</td>
<td>2.437</td>
<td>3.605</td>
<td>1203</td>
<td>148</td>
</tr>
</tbody>
</table>

* probe shape before measurement
** probe shape after measurement

Fig. 2 Image of TGG1 testing pattern showing the probe tip shape changes after a sets of measurements on a) uncoated sample; b) polymer coated sample

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LITERATURE


