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Short communication

Preparation of surfaces of composite samples for tip based micro-analyses using ion beam milling

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ABSTRACT

Ion beam milling, as a method of surface design for tip analytical techniques, was explored. A sample of clay, embedded in a resin, was treated by the ion beam and allowed AFM (a typical tip technique) to be successfully applied. The method is suitable for advanced tip analyses based on AFM, like TERS or SNOM, and for samples that are not possible to prepare by standard mechanical methods. The approach can be useful for characterisation of the surfaces of many different types of materials in versatile applications such as catalysis, corrosion science or advanced material characterisation.

1. Introduction

Knowing the chemistry and physics of material interfaces and surfaces is essential for understanding of many processes occurring in the various areas like catalysis or corrosion. For the purpose of surface characterisation, especially of the layered structures, tip methods like Atomic Force Microscopy (AFM) are frequently deployed. AFM is suited for morphological characterisation of a surface but also for measuring of nanoscale contacts, atomic bonding, Van der Waals forces or Casimir forces via force spectroscopy (Hinterdorfer and Dufrene, 2006). Advanced tip methods like tip-enhanced Raman spectroscopy (TERS) or scanning near field optical microscopy (SNOM) further reveal maps of chemical composition of the surface what may be crucial for material characterisation on the nanoscale level (Kumar et al., 2015; Langelueddecke et al., 2015; Zaleski et al., 2016). Usage of the tip methods requires the surface of the studied object should be sufficiently flat. Simple polishing by standard mechanical methods may be problematic because of possibility of modification of the surface layer or tarnishing of the parallel layers over themselves, effectively preventing proper characterisation of the surface. Similarly, removing of the surface layer by chemical etching may be questionable because of selective leaching of different components or possible unintentional corrosion. Although current mechanical polishing methods may be upgraded to e.g. electrochemical-mechanical polishing (Tiley et al., 2010), these methods are usually suitable only for metal surfaces.

Polyester resins are largely used as a holding matrix for almost all types of samples and therefore samples prepared by this way are especially suitable for validation of the suggested methods. Ion beam systems are largely used for surface treatment of e.g. nanosystems (Erdmanis et al., 2014; Morita et al., 2013), phyllosilicates (Bourdelle et al., 2012), industrial mineral microparticles (Mee et al., 2008), or glassy surfaces (Peng et al., 2014).

The ion milling method has been applied to surface processing for various electron optical methods e.g. high-resolution scanning electron microscopy (SEM), wavelength-dispersive electron probe microanalysis (EPMA), electron back scattered diffraction (EBSD) combined with energy-dispersive electron probe microanalysis (EDX), transmission electron microscopy (TEM) combined with EDX, and micro-Raman spectroscopy (Weiszburg et al., 2017) but not for tip methods. In this work, the method of surface preparation by ion milling focused to

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Table 1
Conditions of ionic polishing.

Number of beam sources	3 x argon
Acceleration voltage	2 kV
Current (each gun)	1.2 mA
Time	20 h

application of tip methods is presented. The approach promises a shift in methodology of the surface treatment of versatile samples, especially those embedded in a resin. As a typical tip method, AFM was selected for validation of the applicability of the method.

2. Experimental

A sample of Bavarian red sandy clay from the Troschenreuth deposit was covered in polyester resin Neukadur PE 45. The sample was selected due to its material properties – the red sandy clay should be excellent to point out advantages of the method in comparison with more conventional mechanical polishing. The red sandy clay tends to crumble and its colour is red, making good contrast for optical microscopy observation. After curing, the grinder LaboPol-5 from Struers GmbH was used for final shaping without polishing. Sample was modified for ion polishing by using the metallurgical saw ATM Brillant 220 and mechanical grinding. Different SiC metallographic papers (P1200, P2500, P4000) and two polishing agents were used; diamond paste with an average grain size of 1.5 µm and suspension of silicon dioxide (Eposil F) with an average grain size of 0.1 µm. In every single step, water was used as coolant. Width and height of the sample were to 7 × 7 mm^2 because of limitations imposed on the samples by the holder used in the ion process.

Ion treatment of the sample was done by using ion beam milling system Leica EM TIC 3 × . Conditions of measurement are summarized in Table 1.

The sample was placed on a special holder, where the assumed section plane was parallel to the edge of the holder and the mask. Overlapping of the sample over the mask was set to about 50 µm by using optical microscope. After the first cut, the plane of the primary incision was shifted by 5 µm and the cut was repeated with a new mask and the same conditions as in the previous case.

After and before the ion treatment, the sample was analysed by the confocal microscope Olympus Lext OLS 3100. Roughness of the surface was measured to demonstrate suitability of the method for analysing of the sample by AFM. Roughness measurement was limited to area 650 × 480 $µm^2$ to obtain more detailed information about the surface.

The Atomic Force Microscope (Bruker Innova) was used for characterisation of the prepared sample surface. The AFM images were obtained using tapping mode with frequency of 300 kHz and resolution of 1.2 nm in xy and 0.2 nm in z directions.

For comparison, the sample was also modified in direct (glow, diode) Ar^+ plasma on Balzers SCD 050 device under the following conditions: gas purity 99.997%, pressure 10 Pa, electrode distance 55 mm, and various times (2–10 min).

Mechanical polishing was selected as a standard method. The sample was ground using sand paper P4000 and polished using Eposil F with particle diameter 0.1 µm. Polishing was realized on neoprene disk. In both types of sample processing, water as coolant was used.

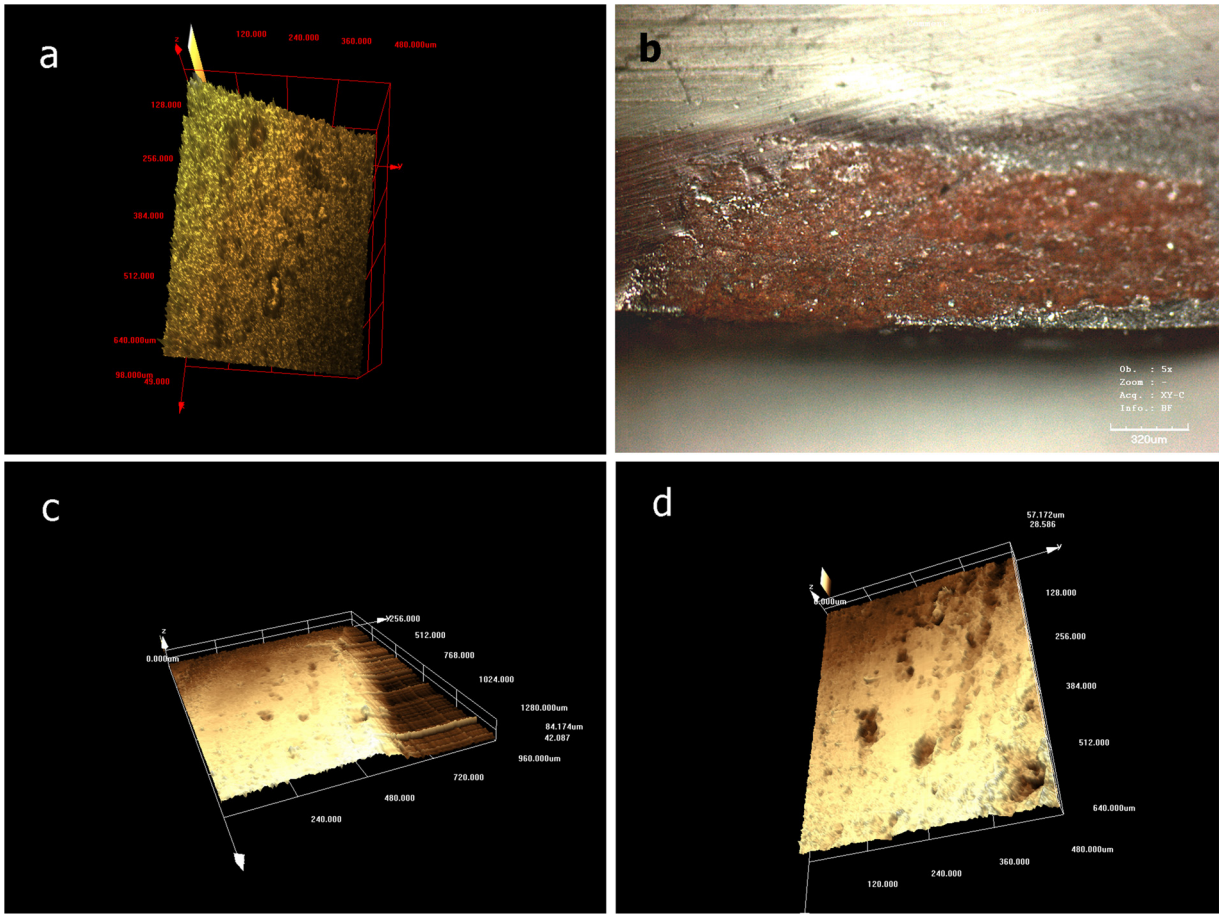

Fig. 1. a) 3D image of the sample before fabrication by ion milling, b) picture of the sample in true colours after mechanical processing before ion beam treatment, c) and d) the sample after teatment with ionic cutter.