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SHORT TECHNICAL NOTE

A convenient and rapid sample repositioning approach for atomic force microscopy

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Summary

A novel repositioning approach is described for repeated observations of a specimen at a close proximal location in the atomic force microscope. The approach is similar to keystone architecture, whereby the repositioning is achieved by forming a male structured base for the specimen, and a corresponding female counterpart as the frame. For the combination of an acrylic acid frame and a metal base, 90% translation shifts are less than 10 μm, and almost all angular disorientations are within +3° to −3°. Nanometre-scale surface features can be relocated easily and reliably even after 40 imaging–removal–imaging cycles, dipping the specimen in solutions or heating up to 500 °C.

Introduction

The evolution of structure under temporal, spatial or thermo-mechanical treatments is an integral aspect of materials development. In such undertakings, which require some form of microscopy for imaging/analysis, site-specific specimen 'registration' is fundamental and critical, to match the images taken at a different time or after different treatments (Brown, 1992; Markiewicz & Goh, 1997; Romer *et al*., 2000; Lin & Goh, 2002; Mantooth *et al*., 2002). The advanced applications of an atomic force microscope (AFM) in localized fabrication, and mechanical, chemical and physical characterization often require a high order of registration to image the same area. In most cases, such repositioning issues involve some of the largest challenges, requiring considerable efforts and patience. This is because the x- and y-positions and the overall orientation of the sample relative to the AFM tip are often altered when the sample is put back on the AFM stage to obtain the next set of images, and there is little position and orientation control in most modern AFM set-ups.

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Here we describe a simple and efficient repositioning approach that provides good accuracy and reproducibility. The system has a moveable base and a rigid frame, which are fabricated in such a way to possess complementary structural features that allow geometrical matching only in one defined position and orientation (Fig. 1). The method is so effective that even when a sample is repeatedly processed for 40 cycles (imaging–removal–return imaging), immersed in solutions or heated to 500 °C, it is easy to find the same general location in a few seconds.

Set-up and results

Acrylic acid polymer is used as the frame and a piece of metal (copper) is used as the moveable base. The copper block is encapsulated in the polymer (Lapmaster International). After complete solidification of the polymer blend (30 min), the polymer solid is mechanically polished to expose the metal base. Subsequent releasing of the metal base yields a polymer frame that has complementary structure with the metal base. As a result, there is only one position and orientation for the base to fit the frame. Proof-of-concept experiments were performed using silicon dioxide substrate on a ThermoMicroscope CP AFM and a Nanoscope III AFM. The AFM sample is mounted on the base by double-sided tape, a polymer adhesive (Crystalbond, 509-S, Ted Pella) or a colloidal silver liquid (Pelco, Ted Pella). The polymer frame is fixed on top of the piezotube of the AFM. Figure 2(A) shows the optical image of a repositioning assembly, with a sample sitting on top of a copper base, and the base sitting on the polymer frame. The size of the assembly can be accommodated by most current AFMs.

The shifts of image centres in the x- and y-directions, and the relative rotation of two successive images are obtained. The shift in the z-direction will not influence the results. Figure 2(B) shows the distribution of Δx and Δy for 90 processes, where 90% of the data fall into or on a circle with diameter 10 μm. Figure 2(C) shows the distribution of $\Delta\theta$ for 90

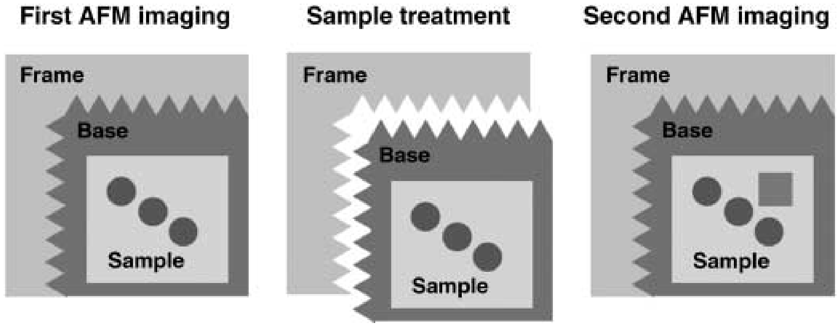

Fig. 1. Scheme of the sample repositioning system.

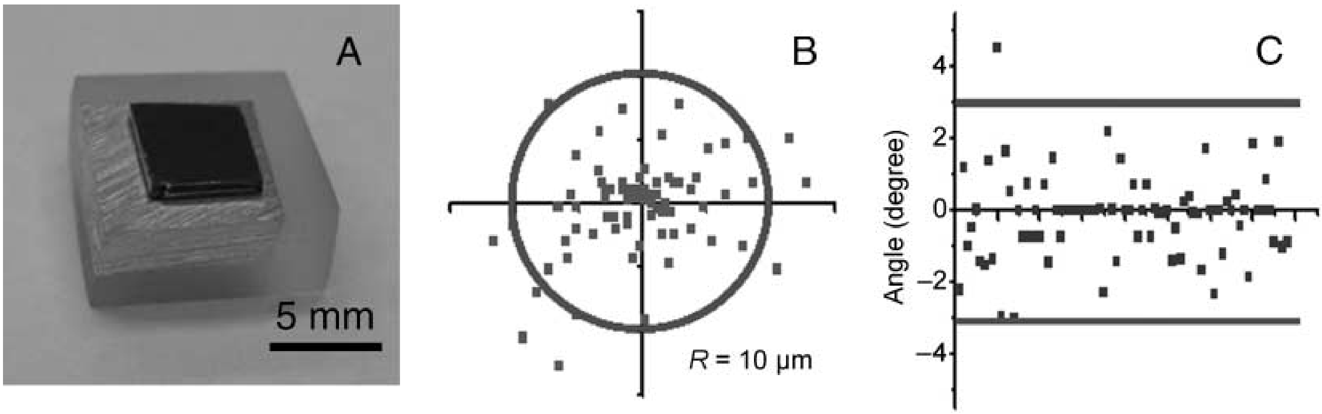

Fig. 2. (A) Optical image of the whole repositioning assembly; (B) distribution of the shifts in the *x*- and *y*-directions in successive AFM images; (C) the angular disorientation of two successive AFM images.

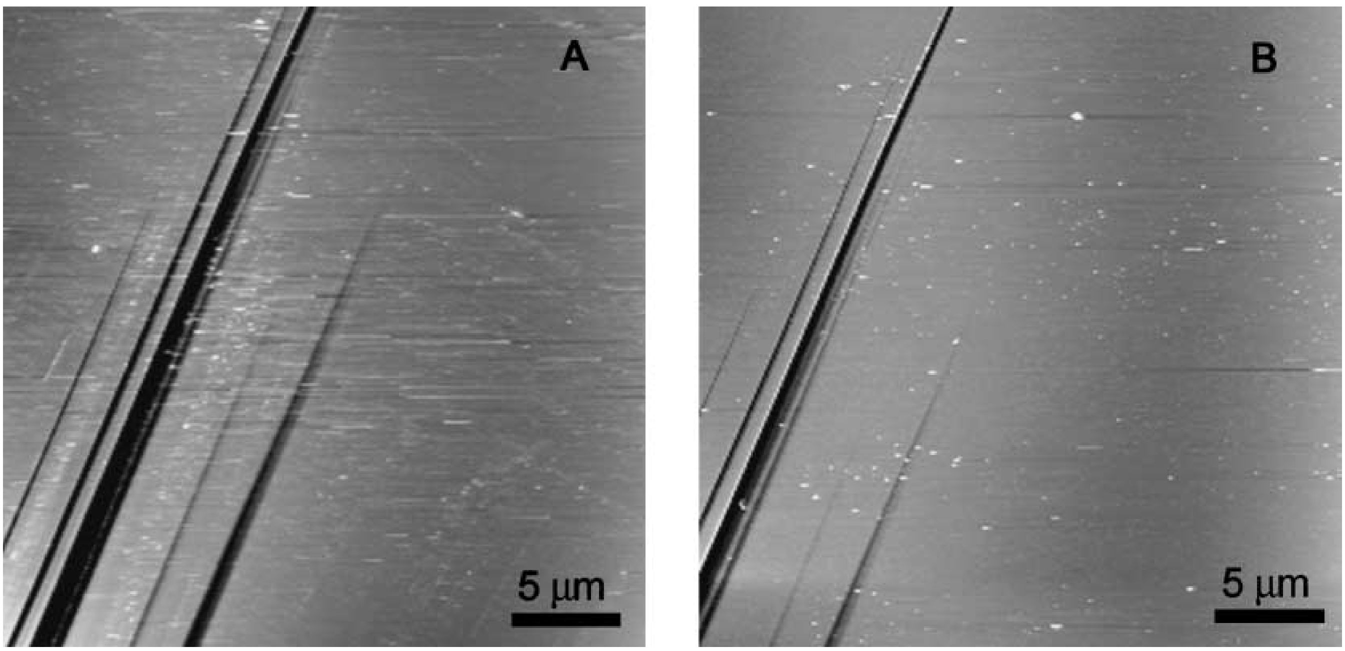

Fig. 3. (A) AFM image of an area before heating; (B) the same area is imaged after heating to 500 °C.

processes; almost all the angles are between +3° and −3°, and some successive images do not show any disorientation. In addition to good repositioning abilities, this simple system shows good long-term stability and reliability: the same area can be found easily after 40 imaging–removal–imaging cycles.

Colloidal silver liquid is used to fix the sample on the metal base to sustain moderately high temperature. Figure 3(A) shows a preheating AFM image of the scratched silicon dioxide substrate attached on a copper base by silver colloid. Figure 3(B) shows an AFM image of the sample after being heated to 500 °C for 1 h. The shifts of image centre are 5.6 and 6.7 μm in the *x*- and *y*-direction, respectively. Rotation of the sample relative to the previous position is 1.07°. This means expansion or shrinkage of the silver glue does not significantly affect the accuracy of the system, and deformation of the metal base at this temperature is not appreciable. On the other hand, the crystalbond mounting adhesive can sustain most solvents and water, and thus is ideal to attach a sample in solution (except acetone and its stripper). We can locate the same area on a sample that was fixed by the adhesive and dipped into a 0.5 M aqueous solution of nitric acid (data not shown). The *x*- and *y*-direction shifts are 10.7 and 8.6 μm, and the rotation is 6.2°.

Conclusions

The novel sample repositioning approach described here enables the site-specific study of surface structures using a AFM in a convenient and rapid fashion. The simple set-up, which consists of a polymer frame and a metal or ceramic base, allows sample treatment in solution phase and at high temperature. Repositioning accuracy is appropriate for most daily practices and could be useful for research in the field of nanotechnology and surface science, in which the morphology of the same location needs to be checked after each sample processing.

Acknowledgements

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