A Procedure for SPM Tip Recovery

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Abstract

AFM-SEPM probe tips and cantilevers contaminated by adhering sample constituents are known to produce a variety of image artifacts, especially "ghosts". In the case of particulate contaminants, a simple and effective procedure for probe recovery is ultrasonic cleaning, using a short exposure (5 min) of the soiled probes to 90 watts of 40 kHz ultrasound within a cleaning bath filled with bi-distilled, de-ionized water. Tip cleaning and recovery are demonstrated using two criteria: the cantilever frequency spectrum and the elimination of ghosts from the images of well-known samples. In some cases, the cleaning procedure has to be repeated, to achieve satisfactory results. This procedure allowed the recovery of many tens of tips without causing probe damage, in this laboratory

Keywords: AFM, SEPM, probe cleaning, ultrasonic cleaning, image artifacts, contaminating particles.

Introduction

The geometry of the probe tips used in scanning probe microscopies has a decisive role in the overall quality of the images, specially concerning their lateral resolution. Even when the surface is scanned at a constant distance, eventual tip collisions with steep features or loose particles, due to inadequate feedback adjustment, may lead to probe tip contamination. This is particularly important when soft, adherent polymer samples are under examination. For instance, we found in this laboratory that Pt-coated Si tips are easily contaminated with poly (styrene-co-acrylamide) (PS-AAM)[1] latex or colloidal polymer particles, thus creating image artifacts (ghosts) in the non-contact AFM and SEPM images. Usual probe cleaning procedures are based on UV-light abrasion, heating and Ar-ion sputtering.[4] These methods are effective for the removal of thin organic layers but not for thicker or particulate contaminants. We have recently found an effective procedure for the removal of particulate contaminants from the probe tips, which is described in this work.

Materials and Methods

Procedures for AFM and SEPM image acquisition were described in previous papers from this laboratory [2,3]. A Topometrix Discoverer TMX 2010 instrument was used throughout.Poorly-performing used tips were collected and examined in a high-resolution field-emission scanning electron microscope (FESEM), JEOL JSM-6340F. Representative images are in Figs. 1-3.

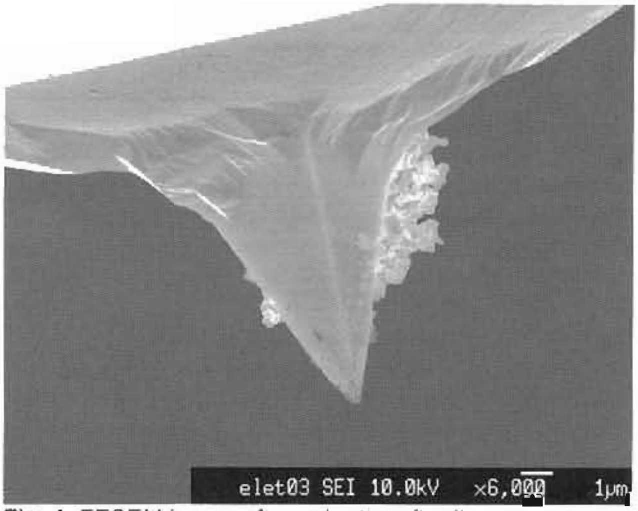

Fig. 1. FESEM image of a probe tip, after its use on a polystyrene latex sample. Note the large number of small particles adhering to the tip sides.

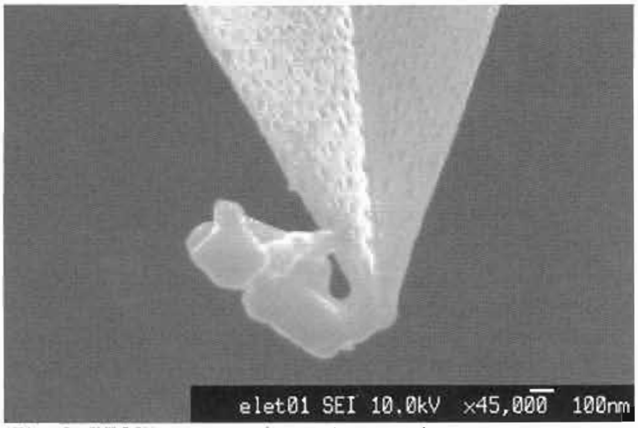

Fig. 2. FESEM image of a probe tip, after its use on a polystyrene latex sample The contaminating particles are accumulated at the tip, changing its geometry.

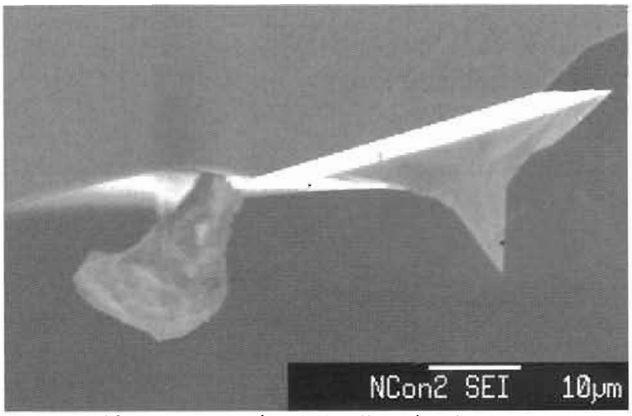

Fig. 3. FESEM image of a probe tip, after its use on a polystyrene latex sample. In this case, de contaminating particle accumulated at the cantilever.

The large particles observed in Figs. 1-3 would certainly require long exposure times for removal by sputtering, oxidation or thermal methods. On the other hand, they are very well suited for removal by shearing or cavitation techniques, such as ultrasonic cleaning.

The tips was thus sonicated within a 50 mL beaker filled with deionized water, partly immersed in a 3L-rectangular water bath, powered by 90 watts of 40 kHz ultrasound, for 5 minutes. Due to its strongly damaging action on many adhesive joints, water is a suitable liquid for cleaning by ultrasonic cavitation, dispensing with the use of any other cleansing agent[5,6], and it is recommended for the removal of particles in the micrometer size range, from solid surfaces.[7]

The fast periodic compression and decompression of a high-surface tension liquid such as water produces a myriad of micro bubbles bursting within the liquid, especially at the existing solid-liquid interfaces. The resulting pressure gradients are sufficient to dislodge the particles, but they can also produce geometrical deformations at the surfaces.

Figure 4 shows a diagram of the set-up used in this work.

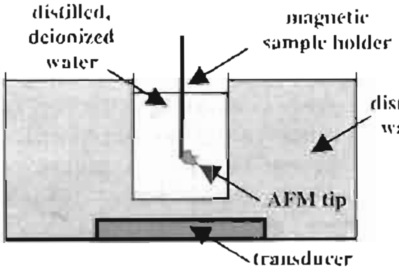

Fig. 4. Schematics of the sample mounting for cleaning

Results

A couple of low-quality, strongly convoluted AFM and SEPM images is shown in Fig. 5. These images show strong "ghosts" of the objects, what can be due to the existence of a double-ended tip.

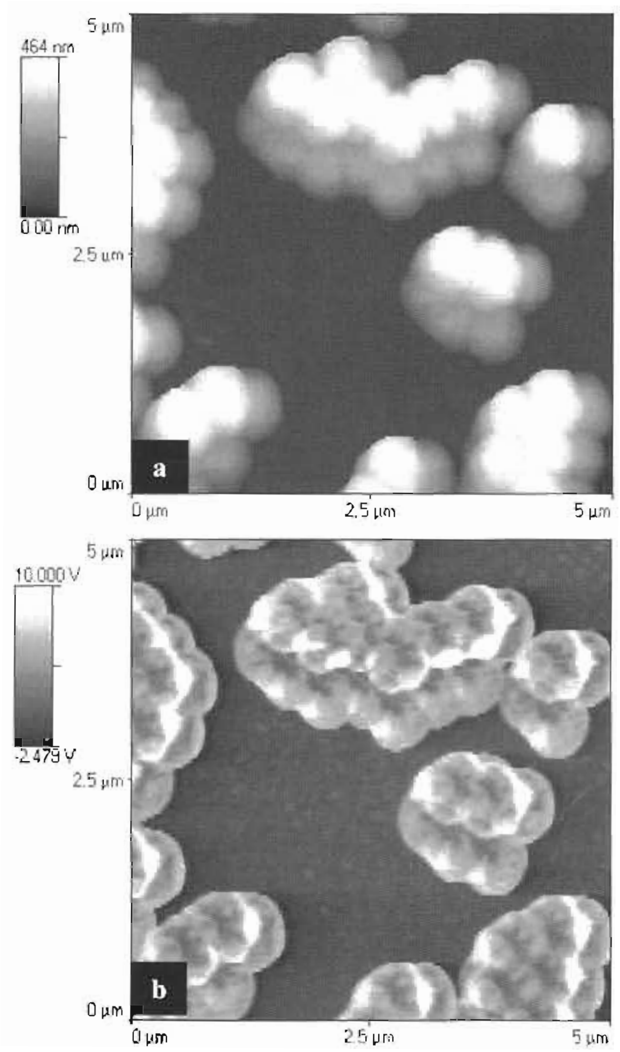

Fig. 5: (a) Convolution artifacts or ghosts in non-contact AFM and (b) SEPM images

The probe used in the acquisition of these poor images was examined in the FESEM, and the micrograph in Fig. 6 shows that it was contaminated by two adhering particles, one of which is at the very probe tip.

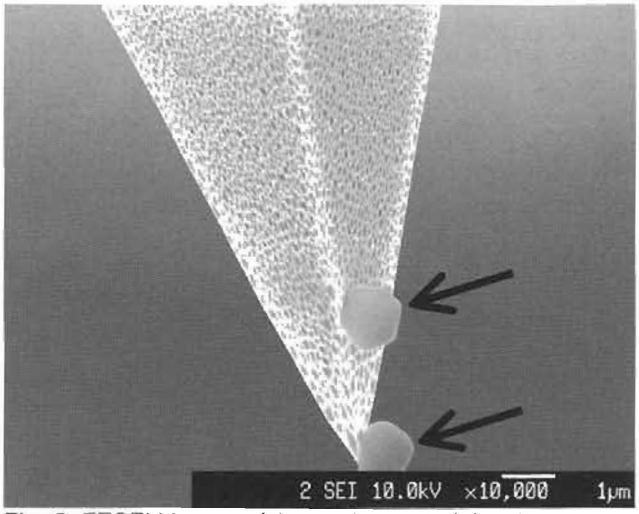

Fig. 6: FESEM image of the contaminated tip, showing two adhering particles.

This tip was cleaned following the procedure described in this work, and the removal of the contaminating particles was complete, as shown in the micrograph in Fig 7.

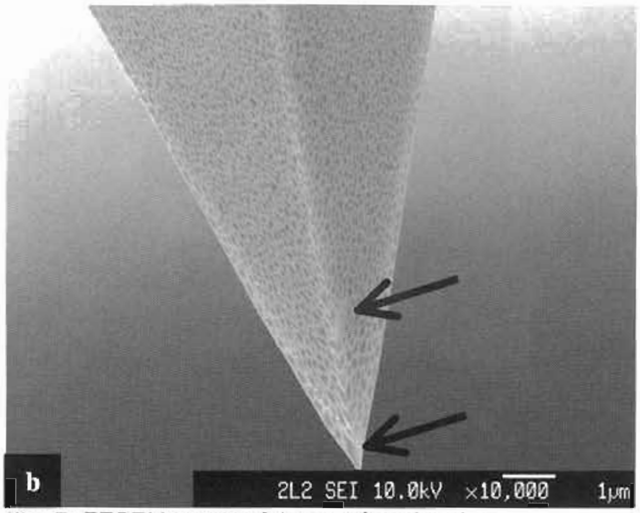

Fig. 7: FESEM image of the tip after cleaning

After tip cleaning and FESEM image acquisition, the tip shown in Fig. 7 was used again to acquire AFM and SEPM images, and these are shown in Fig. 8a e 8b.

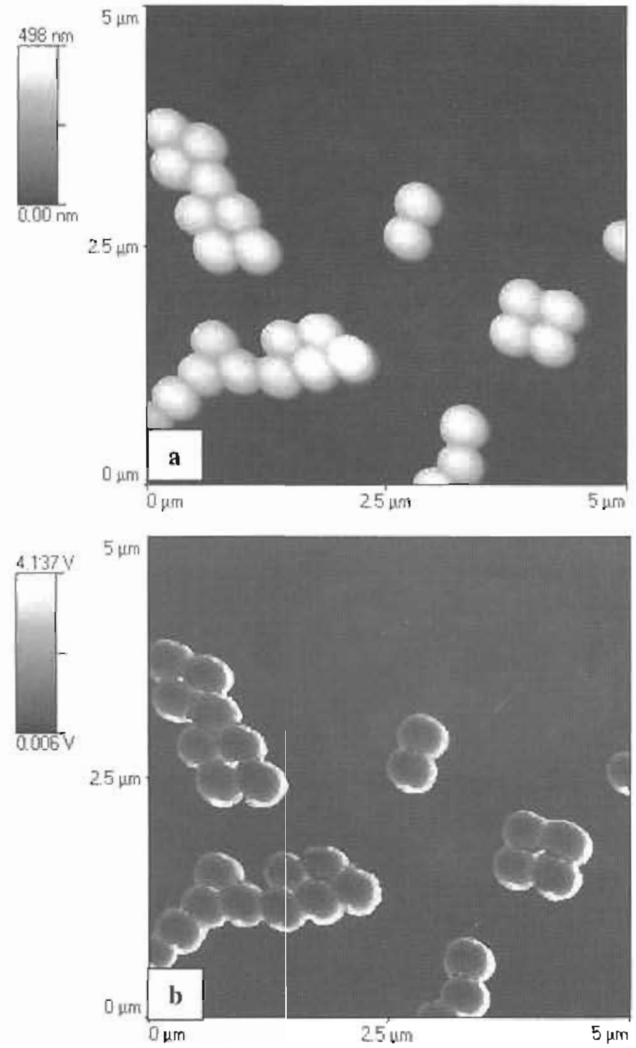

Fig. 8: (a) Non-contact AFM and (b) SEPM images of PS-AAM particles on mica, acquired with the same probe, but after cleaning.

The new images are now exempt of the artifacts seen in Fig. 5a e 5b, thus showing that the contaminated tip was effectively recovered. These particles have been extensively studied in this laboratory: they are ellipsoids with soft shells, formed by an electrically negative core partially enclosed by a positive half-shell.[8]

The cleaning time should be kept as short as possible, to avoid probe break-down. In some cases, the adherent particles could not be removed in a single step.

Micrographs in Fig. 9 are from a heavily soiled probe subjected to two consecutive cleaning steps.

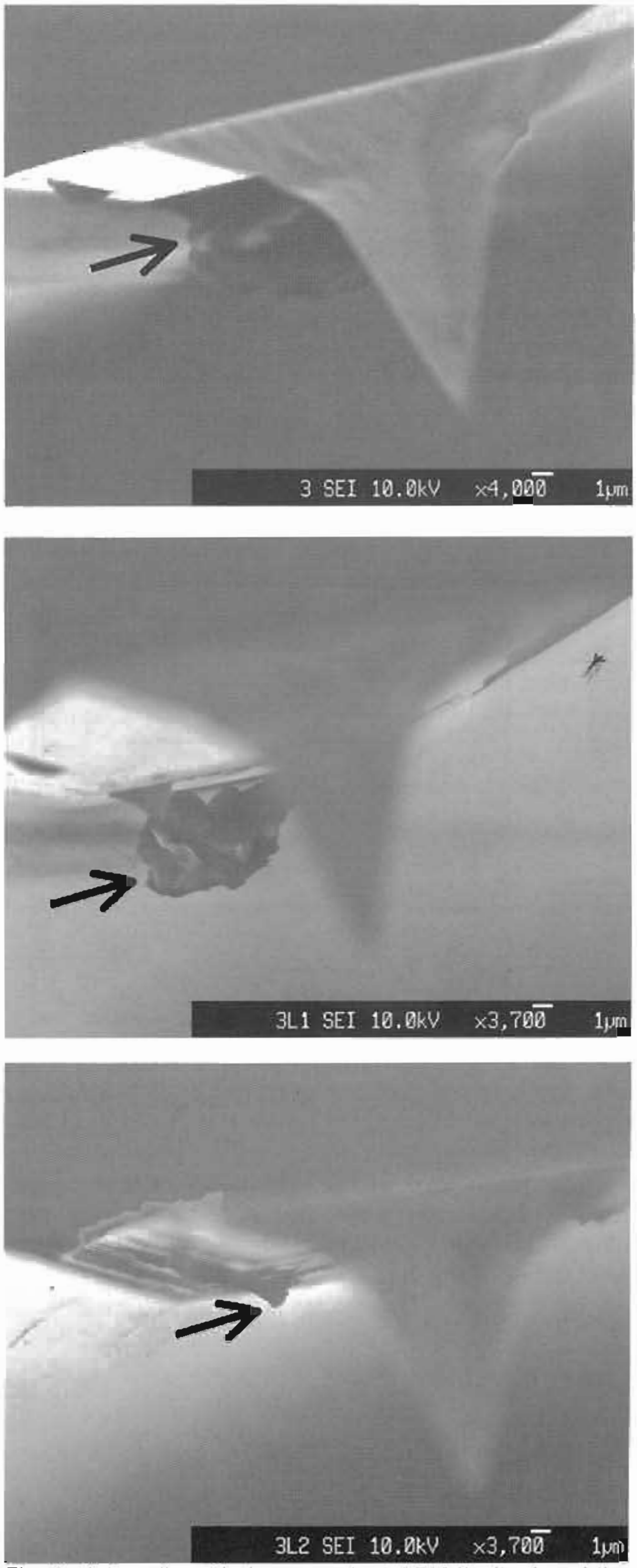

Fig. 9: A heavily soiled non-contact AFM tip (top) and the same, following the first (center) and second (bottom) cleaning steps.

In this case, the efficiency of the cleaning process was verified by using two criteria:

i) the cleaned probe had a single resonant frequency, in the 40-70 kHz range:

ii) the acquired images did not display "ghosts".

Discussion

The cases described in this work are just a few examples, among some tens of tips recovered by using this simple, fast and effective procedure. This is probably adequate for any particulate contaminants, especially those having hydrophilic surfaces. This procedure may be extended to other thinner contaminants, provided suitable cleaning agents are devised. Considering that probe replacement is often the largest single running cost factor in some types of probe microscopy experiments, the procedure described in this work are decisive for making these experiments more easily accessible, while keeping a high imaging quality.

Acknowledgments

The authors acknowledge the support of CNPq, FAPESP and Pronex/Finep/MCT. This is a contribution from the Millenium Institute for Complex Materials.

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