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Application of the nuclear tracks methodology for the validation of atomic force microscope probe tips

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Atomic force microscopy (AFM) is a well-established technique for studying materials at nanoscale dimensions. The resolution of this instrument strongly relies on the sharpness of its tip, which can become compromised through wear and breakage, often due to contamination at the AFM tip apex. To evaluate the condition of both new and used tips, scanning electron microscopy (SEM) inspection is commonly employed. However, SEM services may not always be readily available or in high demand. In this study, we present an AFM tip calibration device that utilizes a pattern of etched tracks on CR-39 material. To construct this device, you will require a radioactive alpha particle source, typically Americium-241, as well as a controlled temperature bath set at 60 degrees Celsius, which contains a 6.25 M KOH solution. This endeavor serves as another intriguing and practical application of the nuclear tracks methodology.

Keywords: AFM tip checking; nuclear tracks.

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1. Introduction

When attempting to image a new sample using an AFM, it can be challenging to determine whether we have achieved an accurate representation of the surface [1]. It remains essential to possess an independent method for assessing the impact of the probe tip on the resulting image. The utilization and interaction between the sample and the AFM tip may result in tip breakage, abrasive wear, or contamination. A damaged or misshapen probe tip can result in inaccurate rendering of samples, [2]. Hence, there is a significant need for a straightforward and convenient method to pre-screen the available AFM tips. Such a method can save time, effort, and prevent frustration. In our study, we present a straightforward and efficient approach to pre-screening tips using CR-39 polycarbonate and the nuclear track methodology (NTM). This work represents an enhancement of our previous article published in 2005 [1]. It's worth noting that other devices for tip checking are available in the market. For instance, several commercially available devices can be used to check the sharpness and quality of Atomic Force Microscopy (AFM) tips. One such device is the "Nano-Observer" by CSInstruments [3]. Another possibility is the use of standard samples [4,5]. However, the CR-39 device proposed in our work stands out due to its cost-effectiveness, simplicity, and versatility. Unlike some other options, it is not restricted to a specific AFM mode and can be applied to all atomic force microscopy modes.

2. Methodology

In this section, we outline the methodology employed for our study. We used a Lantrack CR-39 polycarbonate sheet, which was 600 μm thick [6]. This sheet was prepared with a topographic pattern consisting of an array of conical holes, with dimensions tailored to match those of the probe tip. The creation of this pattern involved utilizing the Nuclear Tracks Methodology (NTM) and its associated procedures. We utilized $\hat{2}$41Am as the radiation source, emitting alpha particles with an energy of 5.49 MeV. Previous research [7] has demonstrated that alpha particles with energies slightly below 2 MeV are suitable for AFM scanning, providing precise profiles consistent with the cone pattern required to verify AFM tip dimensions. To attain the necessary cone dimensions, we conducted an exposure process with the alpha source positioned 3 cm away from the CR-39 surface, in ambient air, at standard room temperature and pressure. To ensure nearly perpendicular incidence of the alpha particles, we inserted a 0.5 mm thick aluminum foil with circular apertures between the radiation source and the polycarbonate detector, serving as a collimator. The chemical etching of the prepared pattern was carried out in a 6.25 M KOH solution at a controlled temperature of $60 \pm 1^{\circ}$C [7]. A one-step chemical etching

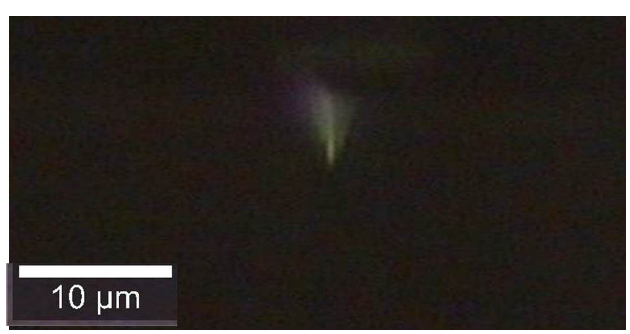

FIGURE 1. Optical micrograph of a fresh AFM probe tip.

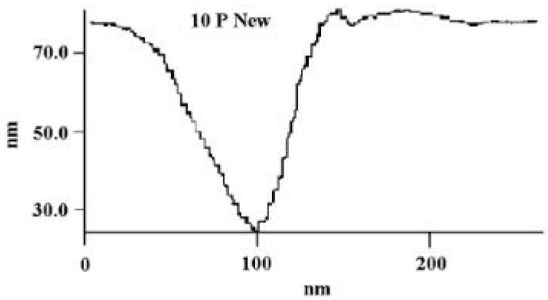

FIGURE 2. AFM topographic profile of an etched nuclear track, obtained with the probe tip shown in Fig. 1.

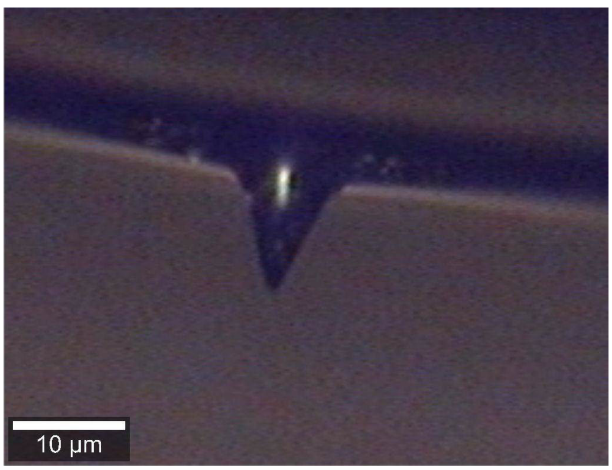

FIGURE 3. Optical microscopy image of a worn out probe tip.

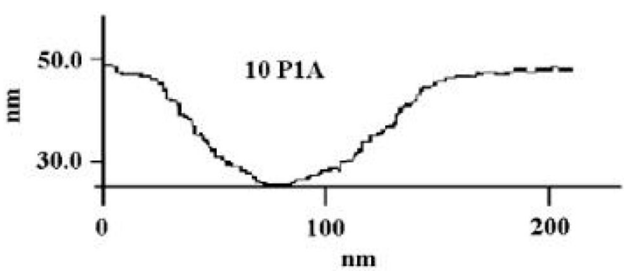

FIGURE 4. AFM topographical profile obtained with the probe tip of Fig. 3.

process was applied for 30 minutes. In Fig. 1, we present an image of a fresh probe tip obtained using a metallographic optical microscope, with objective lens of 50 x. This particular probe tip corresponds to the Digital model Ultralever, with a height of 10 μm and a radius of curvature of 5 nm. These types of tips are capable of generating images that reveal topographical profiles of the etched nuclear tracks, as illustrated in Fig. 2.

To assess the efficacy of the proposed tip gauge method, we will employ a worn AFM probe tip for testing. This tip is depicted in Fig. 3. Using this particular tip, we obtained an AFM image of the etched tracks, and the resulting topographical profile is displayed in Fig. 4. When we compare this image with Fig. 2, it becomes evident that there is a noticeable reduction in resolution.

3. The radius of curvature of the AFM probe tip

The radius of curvature can vary depending on the specific type of AFM tip being used. It can range from a few nanometers to tens of nanometers or more, depending on the manufacturer and the application. A smaller radius of curvature typically allows for higher resolution imaging and more precise measurements. Furthermore, it's important to note that this parameter can increase as the probe tip undergoes abrasive wear, which can, in turn, negatively impact the resolution and performance of the AFM instrument over time. Monitoring and maintaining the quality of the probe tip is crucial for obtaining reliable AFM results. Thus, the tip probe can be represented by a sphere whose radius matches the radius of curvature (R) of the probe. In Fig. 5, we provide a simulation illustrating the topographical profile of an etched track alongside the probe tip [1].

In Fig. 6, we establish the geometric parameters for both the etched track and the probe tip.

Y_o represents the depth of the nuclear track, D signifies the diameter, and θ denotes the angle formed between the axis and the cone surface. These parameters are determined based on the etching conditions specified in the methodology. It's worth noting that due to the finite size of the tip, it cannot reach the bottom of the cone. The minimum distance between the center of the sphere representing the tip and the vertex of the cone is calculated as $R \csc \theta$. This value, along with the apparent depth observed through AFM (Y_{oa}), is interconnected with the geometric dimensions of the cone in the following manner:

$$Y_o + R - Y_{oa} = R \csc(\theta). \tag{1}$$

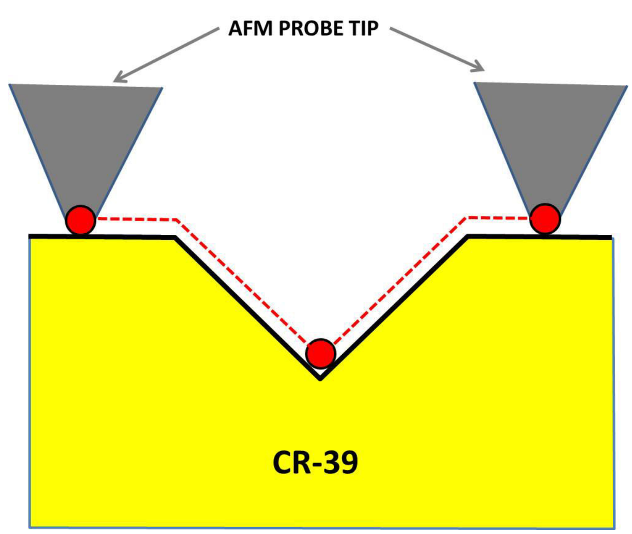

FIGURE 5. The trajectory of the AFM tip along the etched track profile. The red line is the profile obtained with the AFM.

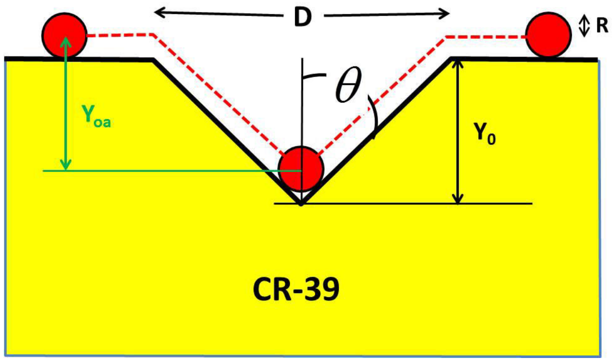

FIGURE 6. Geometrical parameters of the etched nuclear track.

Following this, we can calculate the radius of curvature R using the following equation:

$$R = (Y_o - Y_{oa})/(\csc(\theta) - 1). \quad (2)$$

From Fig. 2 and the average values obtained from real tracks using fresh tips, we have fixed parameters as follows: $\theta = 55$ degrees and $Y_o = 61$ nm. For the image profile obtained with the damaged tip (Fig. 4), $Y_{oa} = 16$ nm. Using Eq. (2) in this case, we calculate R as $R = 195$ nm, which is significantly larger when compared to the R value for the fresh tip (5 nm).

4. Conclusions

This work has introduced a novel method for assessing AFM probe tips. The method relies on the preparation of etched nuclear tracks on CR-39, with carefully calibrated geometric parameters. Experimental testing of this methodology in AFM operations has demonstrated several advantages over other methods, including simplicity, reduced analysis time, applicability to all AFM modes, and cost-effectiveness. This represents a promising new application of solid-state nuclear tracks methodology.

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