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IMPROVEMENT IN LONG TERM STABILITY OF THE LONG RANGE AFMS

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Abstract – Long range AFMs suffer mainly from drift due to the relative long measurement times and from tip wear due to the large measured distances. After investigations of the long term stability of the AFMs, in this work, the recommendations for planning and realization of the long range measurements are proposed.

Keywords: AFM, long range AFM, nanopositioning and nanomeasuring machine, NPM machine, long distance measurements

1. INTRODUCTION

Atomic force microscopy (AFM) is the special technique for investigation, imaging and measurement of the surface topography and properties with nanometre resolution within the small range of a few hundreds micrometer [1]. Current trends in micro-, nanotechnology, precision engineering and further modern fabrication technologies demands beside accurate dimensional measurements, the long scanning ranges.

In order to meet the described requirements, a nanopositioning and nanomeasuring machine (NPM machine) has been developed at the Institute of Process Measurement and Sensor Technology of the Technische Universität Ilmenau with a resolution of 0.1 nm over a positioning and measuring range of 25 mm × 25 mm × 5 mm [2].

Due to its structure different probe systems, including detailed explained in several publications [3, 4 and 5] interferometer-based AFM head (fig. 1) and SMENA type scanning measurement head from NT-MDT Co. [6] (fig. 2), has been successfully integrated into the NPM machine.

The feature of the AFM head is the metrological deflection detection system that combines a beam deflection and a homodyne interferometer, and therefore enables simultaneous measurement of the bending, torsion and position of the cantilever. AFM head includes an additional high-speed piezoelectric drive for control the cantilever deflection during the fast surface scan [5]. The movement of this piezo is interferometrically measured directly on the cantilever using the deflection detection system.

The NT-MDT head is a commercially available AFM with good metrological characteristics, with the beam deflection detection system. This AFM is the stand alone scanning probe system with the capacitive sensors for measurements of the movement of the piezoscanner.

The NPM machine with these probe systems operates as the long range AFM.

Long range AFM measurements are afflicted with the drift due to the long measurement times and from the tip wear due to the large measured distances. The goal of this work is minimising drift, tip wear and therefore the measurement uncertainties in long range AFMs. This paper deals with the investigation and improvement of the long term

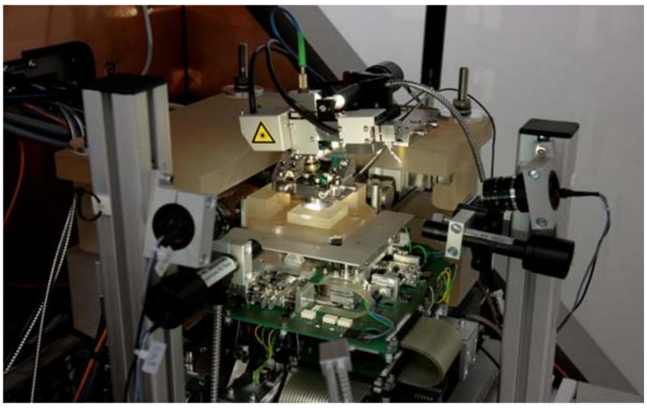


Fig. 1. Interferometer-based AFM head.

Fig. 2. SMENA type scanning measurement head (NT-MDT Co.).

stability of the AFMs as well as with the practical determination of the tip wear during the scan. In this work the authors will present the recommendations for planning and realization of the long range measurements.

2. INVESTIGATION OF THE DRIFT BEHAVIOR

Long-term length measurements are especially susceptible to the environmental effects and particularly to temperature variations [7]. Therefore the long range AFM (NPM machine with the probe sensor) is enclosed within an acoustically isolated chamber with a temperature control using refrigerated & heating circulator with a temperature stability of 10 mK. For humidity fluctuation minimization inside the environment chamber silica gel was used as the humidity puffer. The temperature control system with temperature, pressure und humidity sensors is the parts of the environment server that provides continuous environmental data acquisition.

The experimental procedure for drift evaluation presented by Marinello et al [8] was based on repeated measurements on a structured reference grating. Sometimes it is difficult to separate the drift of the probe system from the drift of the sample and from the tip wear. Another way to investigate the long term stability of the long range AFMs would be realized by continuous recording of the signals of the AFM head. Particularly important are deflection (bending or oscillation amplitude) and position value of the cantilever. Drift of the deflection signal induce to the changes in the probing forces as well as of the position signal cause to the changes in the height information during the scan.

For the practical measurements the information about the waiting time after cantilever replacement and switch-on of the temperature control plus the knowledge about the long term stability and drift behavior of the cantilever deflection and position are especially crucial.

The waiting time (stabilization time) for achievement of ≤ 20 mK temperature stability after sample/probe adjustment/replacement and turning-on of the temperature control as well as the total drift (during the stabilization time) were determined and presented in figures 3 and 4.

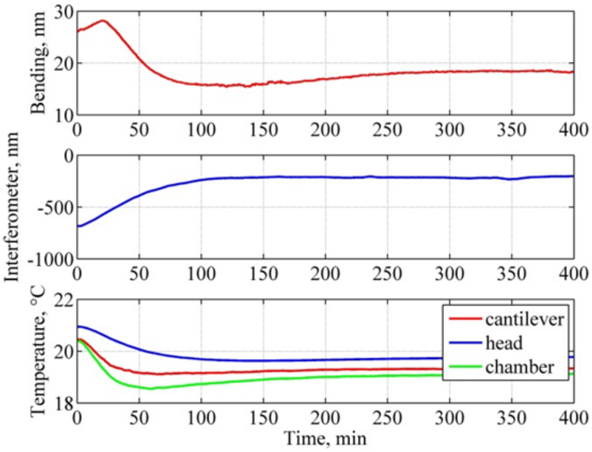


Fig. 3. Interferometer-based AFM head, behaviour after the cantilever replacement/adjustment.

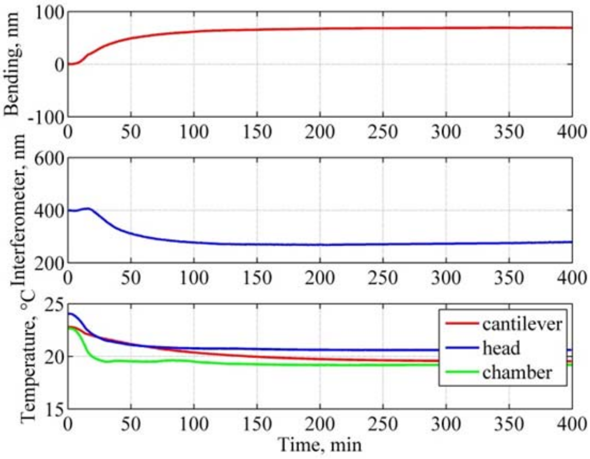


Fig. 4. SMENA AFM head, behaviour after the cantilever replacement/adjustment.

The waiting period last respectively approx. 2 hours for Interferometer-based AFM and 3.5 hours for SMENA AFM.

Furthermore the long term stability of the AFM signals after the stabilization time and at high environmental stability over several hours were determined. The long term stability can be presented as the peak to valley (pv) and the standard deviation (std) of the probe system signals:

Table 1. Long term stability over 25 hours.

	Bending		Position	
	pv, nm	std, nm	pv, nm	std, nm
Interferometer-based AFM head	1.6	0.3	24	7
SMENA AFM head	2	0.4	120	35

Generally, the parameter determined above can fractionally differ when using another cantilever (probe).

3. INVESTIGATIONS OF THE WEAR OF THE CANTILEVER TIP

The finite size of the AFM tip and the changes in its shape due to the wear during the scan induce lowering of the lateral resolution and accuracy of the AFM measurements. Because each measured image of the sample surface corresponds to the convolution (dilatation) of the cantilever tip with the real surface [9]. It is important to know the shape of the tip and its wear behavior according to the measured distance and different operational, scanning and surface parameters.

3.1. Practically possibilities of determination of the AFM-tip shape

Established possibilities to determine the shape of the AFM tip are via imaging by scanning electron microscope (SEM) as well as by scanning the tip characterizer with special structures and subsequent tip reconstruction ("in-situ" methods). This are tip-characterizers with the different pattern types such as TGT1 array of sharp tips [6], (square) pillars with sharp undercut edges as example TGX1 [6] or different sharp-edged line-space structures with vertical sidewalls [10] as well as PA01 with hard sharp pyramidal random nanostructures [11] (cf. fig. 5).

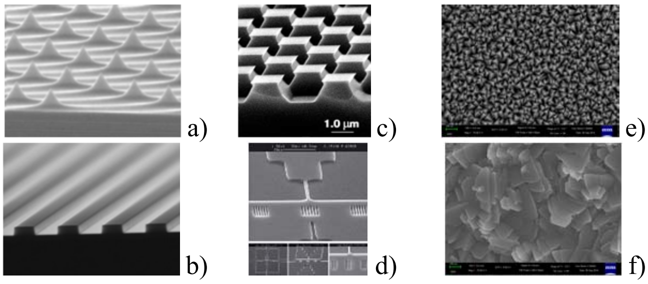


Fig. 5. Tip-characterizer: a) TGT1 [6], b) TGZ [6], c) TGX1 [6], d) AFM-tip characterizer [10] and rough surfaces with sharp-edged structures with small curvature radii: e) PA01 characterizer [11], f) Ti-structure.

In order to select the most appropriative, particularly with regard to technical feasibility, accuracy and repeatability, for wear investigation method of the tip shape determination all prementioned characterizers were tested and results compared with each other as well as with the SEM images of the tip.

The tip-characterizer with line-space structures (cf. fig. 5 (b, d)) and sharp undercut edges (cf. fig. 5 (c)) allows the determination of the tip shape on the basis of few scanned lines over the structure. With the precondition that width of the lines are exactly known and the tip-characterizer is perfectly perpendicularly aligned to the scan direction.

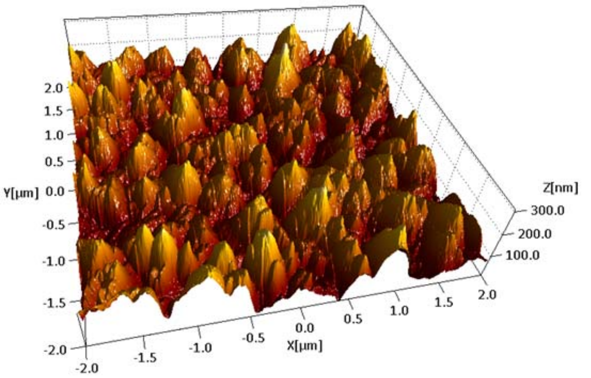


Fig. 6. Falsified mapped topography of the rough surface with sharp nanostructures (cf. fig. 5 (f))

The shape of the AFM tip can be also reconstructed from the falsified mapped topography of the tip-characterizer [12, 13 and 14] (cf. fig. 6). The approach of this blind-tip-estimation (BTE) method is based on the fact that the image of the tip shape is the component of the scanned image of the sample. This technique enables determination of the tip shape without exactly information about the structure of the tip-characterizer. Whereupon this method is sensitive to the scanning range, signal noise, vibrations and oscillations of the control loop. The stochastically rough test structures with sharp-edged isotropic arranged structures with high local inclinations and small curvature radii (cf. fig. 5 (e, f)) are particularly suitable for determination of the tip shape. The BTE method was tested using Gwyddion Software [15] from Department of Nanometrology, Czech Metrology Institute and Software Scanning Probe Image Processor (SPIP) [16] from the Image Metrology Company; thereby both software yielded almost identical results. As the measurement samples were used Ti-structure and PA01 characterizer [11]. But the specifically sharp-edged Ti-structure leads to the wear of the tip during the scan. Especially fine and compact structure of the PA01 characterizer allow the determination of the shape of the tip only to the deepness until 50 – 60 nm. The determination of the blunted tip is therefore impossible.

Particularly suitable tip-characterizer with regards to long term AFM measurements is the TGT1 [6]. The scanned image over the TGT1 tip directly shows the superposition of the sample and the AFM tips. Therefore it is possible to determine the shape of the AFM tip from the measuring data. The scanning range of 1 µm × 1 µm and point distance of 2 nm are sufficient. The determined tip size is larger than it actually is. Advantageous is the combination of previous described BTE method with the measurement of the tip-characterizer TGT1. That provides the best coincidence with the images recorded by the SEM (cf. fig. 7).

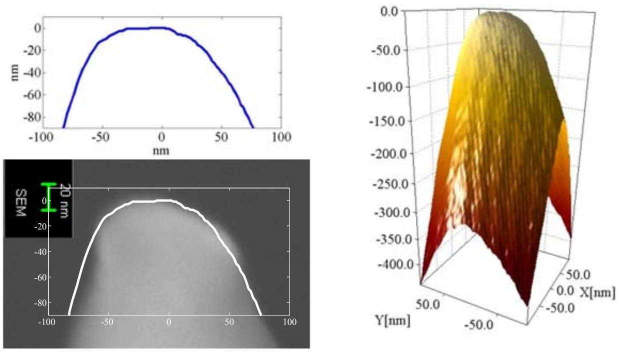


Fig. 7. Determination of the tip shape.

The great advantages of the SEM are the real images of the AFM tip and not only the approximations of it are given. However, a disadvantage is that none "in-situ" characterization is possible and the cantilever must be expanded for investigation from the AFM and reassembled in the SEM. The SEM imaging can complete previous discussed "in-situ" methods.

3.2. Experimental determination of the tip wear

The experimental procedure for wear investigation based on the successively scan of the tip characterizer TGT1 (with the subsequent tip shape determination using BTE) and the measurement sample, at the end of measurements for verification of the tip reconstruction the SEM images of the tip are used (fig. 7).

Due to the non-linear wear behaviour with respect to the scanning distances the tip shape are several times determined. The intervals for tip characterization were experimental decided. The changes of calculated tip form and tip radius serve as the rate of the wear. Because the radius of a new tip at the beginning is least, the estimated wear, due to the resulting large Hertz's contact pressure [17], is greatest. For this reason, the scan distances between the tip characterizations at the beginning was selected as small and then gradually enlarged. The scan distances for wearing of the tip was limited by 1.5 m, because this is common measured distance by long range measurements [18] as well as the test measurement indicates that approx. up this scanning length only hardly changes of the tip radius were detected.

Besides to the scanning distance the careless approach motion (or respectively landing of the tip to the sample surface) can affect the wear of the tip and was investigated. The landing procedure was reiterated. The increased enlargement of the tip radius up to the first few landings was determined (cf. fig. 8). The reduced probing force and slow landing speed (of approx. 200 nm/s) also leads to lesser enlargement of the tip radius. However unnecessary landings during the measurements should be avoided.

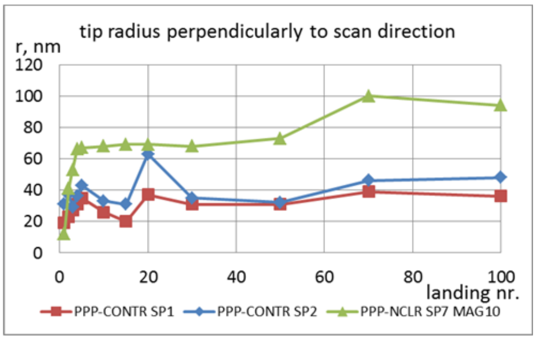
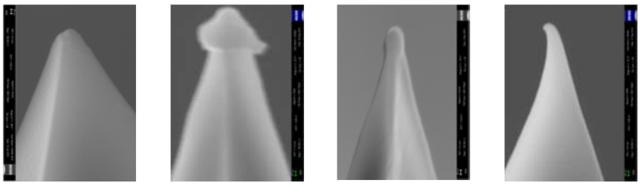


Fig. 8. Landing. Determined tip radius in xz-plane, perpendicularly to the scan direction. SP1 – 1 nA or 125 nm, SP2 – 2 nA or 250 nm (CM), SP7 MAG10 – 70% of the 10 nA free oscillation amplitude (IM).

The changes of the tip form as well as the tip radius are caused not only from wear itself but also by bending, buckling and addition of the particles (cf. fig. 9).

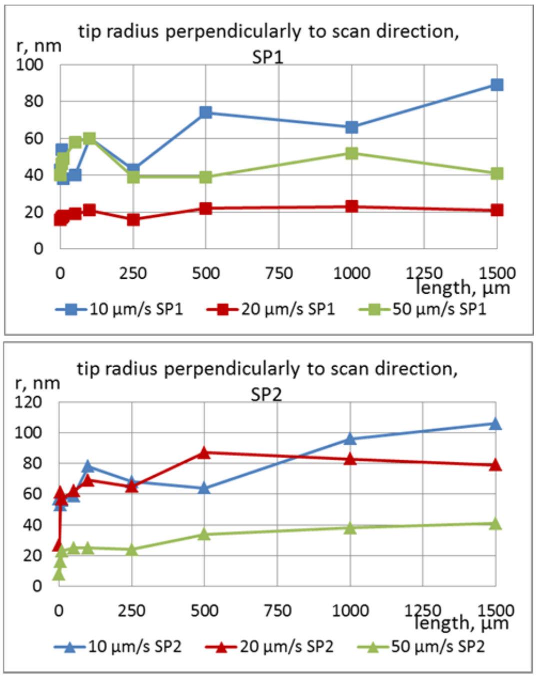


a) b) c) d)

Fig. 9. SEM images of the AFM tips: a) wearing, b) addition, c) buckling, d) bending.

3.3. *Effect of the different operating parameters on the wear of the AFM-tip*

Effect of the scanning speed, probing force, mode of operation as well as the design of the tip itself on its wear were investigated.



a)

b)

Fig. 10. Scanning speed. Determined tip radius in xz-plane, perpendicularly to the scan direction: a) SP1 – 1 nA or 125 nm, b) SP2 – 2 nA or 250 nm (CM).

The chosen scanning speed was applied to conventional for AFM measurements values from 5 µm/s to 50 µm/s and the measurements were performed in both modes of operation and with different probing forces (cf. fig. 10). Accordingly the duration of the series of measurements varied depending on the scanning speed from 8 h 20 m to 83 h 20 m. On the one hand, due to the high speed, the feedback (control loop) of the AFM can be too slow to adjust the height and therefore to remain a constant probing force when the tip hit the step. Abruptly increased forces can act on the tip and deform it. On the other hand due to the very slow speed the measurement time and therefore the action time of the probing force on the tip is increased, that could lead to wear of the tip.

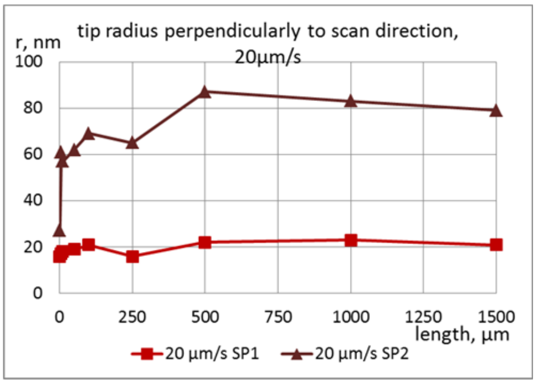


Fig. 11. Probing force. Determined tip radius in xz-plane, perpendicularly to the scan direction. CM measurement.

The probing force acting on the tip is proportional to selected setpoint value that the oscillation amplitude or deflections of the cantilever correspond. On the basis of measurement data at 10 µm/s and 20 µm/s speeds (cf. fig. 11) can be shown, that the increasing of probing force (e.g. from 1 nA to 2 nA, or respectively 125 nm and 250 nm deflection) leads to the faster increasing of the tip radius to a larger value.

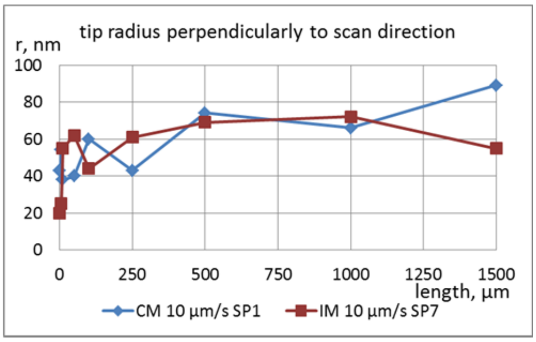


Fig. 12. Mode of operation. Determined tip radius in xz-plane, perpendicularly to the scan direction.

The different modes of operation (contact (CM) and intermittent (IM)) require also different cantilevers. For investigations of the effect of the mode of operation, cantilevers with similar tips were used: PointProbe® Plus PPP-CONTR [19] and PPP-NCLR [20] from NANOSENSORS™. Also in these investigations all parameters besides the mode of operation (and respectively probing force 1 nA in CM and 70% of the free oscillation amplitude in IM) were attempted to keep constantly. The behaviours of the transformation of the tip radii in CM and IM are similar. But in IM the tip radii run betimes to the somewhat larger values than in CM (cf. fig. 12). This can be explained by the measuring principles of the both operating modes.

The tip wear of the following cantilever types were tested: besides the standard PointProbe® Plus, probes with hard diamond-like-carbon DLC coating on the tip [21] and with the super sharp improved super cone SS-ISC tip [22].

The measurements were performed with a scanning speed of 20 µm/s and setpoint that correspond to 70% of the free oscillation amplitude (q.v. previously).

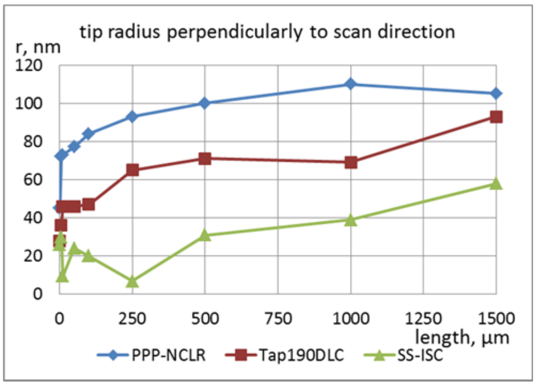


Fig. 13. AFM tip design. Determined tip radius in xz-plane, perpendicularly to the scan direction.

In figure 13 are presented, the changes of the tip radius in respect to the different cantilever types. The behaviours of the increasing of the tip radii of the Tap190DLC and PPP-NCLR cantilevers are similar. But the tip radius of the Tap190DLC cantilever run to the somewhat smaller value than of the PPP-NCLR (cf. fig. 13). This can be explained by the special wear resistant coating. In the last 500 mm of the entire test section comes to enhancement of the radius, what could be explained by the abrasion of the 15 nm thick coating. So a DLC coating increases the wear resistance and reduces wear of the tip up to approx. 60%.

The smallest radii were determined by the SS-ISC cantilever, what can be explained by the special form of the tip. Owing the special cone form of the tip its radius very slowly enlarges. The wearing of the tip lead to slow increasing of its radius. A SS-ISC cantilever allows long scans with a high lateral resolution despite normal wear of the tip.

Therefore the tip wear or increase of the tip radius strongly depends on the material and its shape.

As the last parameter, the influence of the material of the sample surface on the wear behaviour of the tip was investigated. The measurements were performed also on the glass surface, with the similar roughness as by silicon surface, in both modes of operation (CM and IM) with a scanning speed of 20 µm/s. The same results were achieved for measurements on both surfaces.

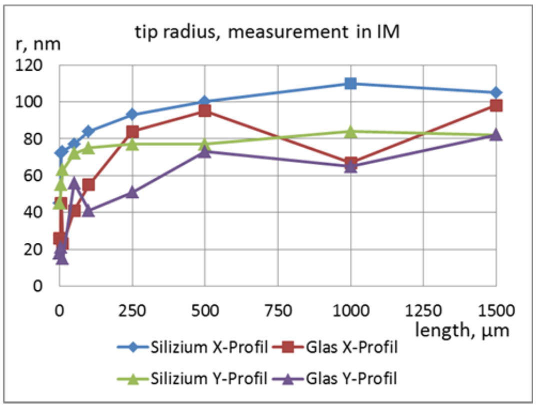


Fig. 14. Material combination. IM measurement.

In all cases the wear behaviour is fast in the beginning of scan and slower toward the end. The coated cantilever tips are more wear resistant as the standard cantilever. Furthermore super sharp tips are suitable for long distance scanning.

4. CONCLUSIONS

A good recommendation is the recording of the probe system signals and environmental data during the waiting time. With this information the start of the measurement can be decided. Furthermore, to avoid the temperature changes after beginning of the scan, the corner mirror (stage) of the NPM machine must be moved during the waiting time.

The determined values of the tip wear can help planning the long range measurements. Most changes in the tip shape happen within first millimetres of the scan. The tip shape can be any time determined (by scan on the tip characterizer) during the long distance measurement. If, during the scan, the cantilever replacement is required and measurement must be continued, the knowledge about the position of the new tip is important for with the developed fiducial marks for accurate relocation of the tip can be used [23].

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