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Scanning near-field optical microscopy by near-field reflectance enhancement: a versatile and valid technique

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ABSTRACT: The validity of reflection-back-to-the-fiber SNOM (scanning near-field optical microscopy) has been unduely questioned by an erratic approach curve that disputed the enhancement of near-field reflectance. It is shown now that only truncated (broken) tips without metal coating do not experience the enhancement when approached close to shear-force distance. However, sharp uncoated tips continue to show up the near-field enhancement, and chemical contrast on rough surfaces continues to be of basic value at submicroscopic resolution. It is pointed out how a good tip may be immediately differentiated from a broken one. Copyright © 1999 John Wiley & Sons, Ltd.

KEYWORDS: near-field reflectance; sharp tips; broken tip artefacts; scanning near-field optical microscopy; submicroscopic resolution; chemical contrast

INTRODUCTION

Scanning near-field optical microscopy (SNOM) has been shown to provide submicroscopic resolution with tapered uncoated tips that pick up reflected light with increased intensity when approached to constant shear-force distance above organic[1,2] and inorganic[3] surfaces. The phenomenon of increased near-field reflectivity was first described by Courjon *et al.*[4] However, in contrast to the well established facts, an intriguing paper by Sandoghdar *et al.*[5] claimed a decrease of the reflectivities upon shear-force approach due to interference effects and disputed the sudden discontinuity in the reflection signal as one approaches the tip to the sample. Unfortunately, that paper[5] confused the issue without providing relevant experimental details. In view of numerous approach curves that clearly validate (i) the near-field enhancement on various surfaces[6,7] and (ii) the repeatable chemical contrast determinations on rough organic surfaces, we address the issue with some of our common approach records and evaluate the possible errors in the failure of Sandoghdar *et al.*[5] to observe ‘the sudden increase.’

EXPERIMENTAL

Tip characterization

Multi-mode and single-mode (SMC-A0515B) fibers of 125 nm from Spectran were pulled as sharp as possible using a Sutter Instruments P-2000 laser-based fiber puller for our experiments at 488 nm. At optimized pulling parameter settings (Heat 235, Filament 0, Velocity 5, Delay 120, Pull 160), the pulling time was about 120 ms. Microscopic inspection indicated a total probe length of about 0.5 mm. Scanning electron micrographs gave radii of curvature of the probe tips of about 15 nm and taper angles of typically 18–20°. Resonance frequencies were about 150 kHz and *Q*-factors ranged from 200 to 300 (see Ref. 7 for further details). The broken tips were judged under a microscope at 400-fold magnification. The force setting for automatic coarse and close approach at a DME Rasterscope 4000 SNOM was 0.017 nN for a force constant of 1.024 N m^{-1} in these experiments. The level of parasitic light was measured to be 14% of the background intensity by disconnecting the unengaged illuminated fiber.

RESULTS

When an illuminated multi-mode tip (488 nm, total exit intensity 200 μW) was coarse-approached to a polyvinylpyrrolidone layer on glass at an initial recorder reading of 180 mV, a considerable increase in reflected light intensity was obtained upon a short touch of the set-point for shear-force damping. From there the tip was forced to move to the fixed close approach start position (Fig. 1). The scan of the chemically uniform surface after close approach gave a recorder reading of 860 mV, more than 4.7 times the total background (Fig. 1). With the

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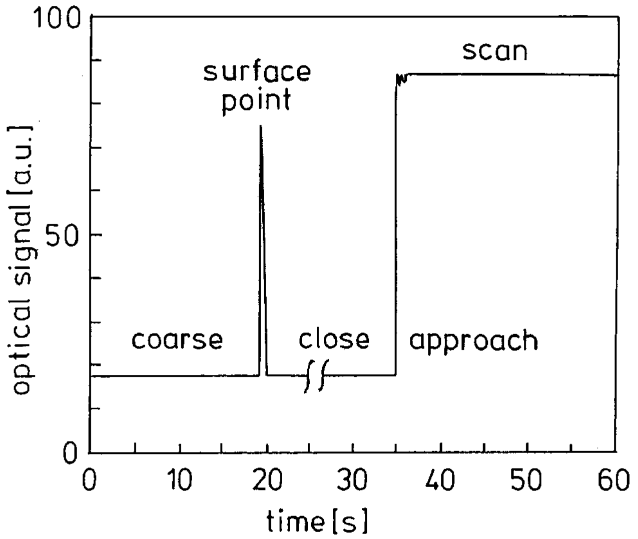

Figure 1. Approach record of a good uncoated tapered tip (radius *ca* 15 nm) to the chemically uniform organic resin surface under shear-force control, indicating the automatic search for the close approach tip position (time constant of the recorder: 0.5 s) and the increased intensity level that is collected back through the fiber upon scanning

single-mode tip under the same conditions the increase was 4.8-fold. Thus, without the necessity for assessment of the precise tip-to-sample distance, we do observe Courjon's *et al.* enhanced near-field reflectivity[4] in the approach record (Fig. 1). Similar near-field increases were obtained when organic crystal surfaces,[1] e.g. (110)

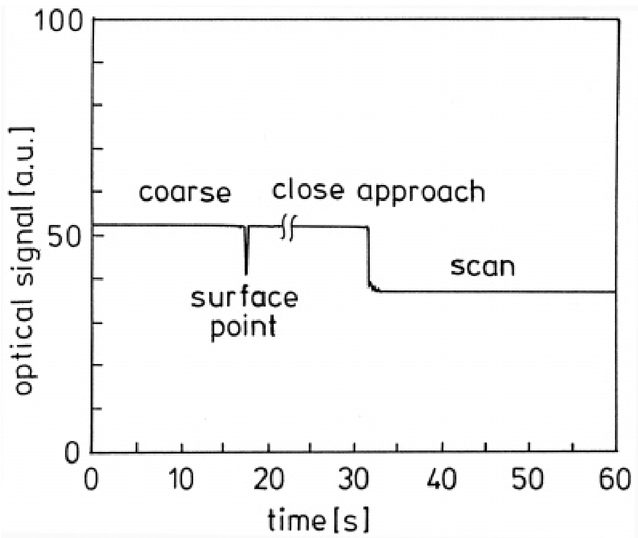

Figure 2. Approach record of a broken uncoated 'tip' with a diameter at the truncation of 2 μm to the organic resin surface under shear-force control, indicating the automatic search for the close approach tip position and the decreased scan response energy that is collected back through the broken fiber upon scanning

of anthracene, were approached with single-mode or multi-mode sharp tips.

Further, we took approach records of tips which had suffered the loss of their sharp cone end, in order to evaluate the conditions for the failure that was claimed by Sandoghdar *et al.*[5] As expected, we observed a decrease (from 520 to 365 mV recorder reading) in the reflected light intensity that came back through the truncated 'tip' (2 μm wide, Fig. 2). Hence only broken or truncated tips reproduce Sandhogdar's *et al.* claims, and such tips do produce artefacts in both the topographic and the optical image. Only sharp tips are suitable for artefact-free SNOM measurements with submicroscopic resolution.[1]

As Sandhogdar *et al.* did not report all details of their tip,[5] we also checked the response of still more heavily truncated tips. Extremely blunt truncated cones (40 μm wide) did not give any change in reflected light intensity when approached under shear-force control, at least in 10 μm scans.

CONCLUSIONS

Our results indicate that Sandhogdar *et al.*[5] either used uncoated tapered tips that were truncated or broken, or that their tips broke during their approach procedure. Broken tips are useless for the collection of enhanced near-field reflected light and give topographic artefacts but no SNOM response; any chemically and physically imposed correlations between AFM and SNOM contrasts are lost when running broken tips under shear-force control. Hence a warning is necessary, because every tip could be broken or eventually break during approach and/or scanning: *approach records similar to* Fig. 1 *must be documented in order to ensure the near-field enhancement prior to every SNOM measurement!*

Furthermore, some of the reported changes in contrast behavior during scans may be due to tip breakage. Experimentally, tip breakage is immediately recognized by a substantial decrease in the response signal. Good tips do, of course, not show a taper end under a light microscope, whereas unsuitably truncated (broken) tips do. Properly performed reflection-back-to-the-fiber SNOM continues to provide valuable and reliable chemical contrast on technically important rough surfaces at submicroscopic resolution.[1]

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