



Singlemode chalcogenide fiber infrared SNOM probes

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Abstract

We have fabricated and tested infrared scanning near-field optical microscope (IR-SNOM) probe tips made from singlemode chalcogenide fiber. The process used to create the tips was similar to conventional micropipette-puller techniques, with some modifications to allow for the lower melting temperature and tensile strength of the chalcogenide fiber. SEM micrographs, showing tips with sub-micrometer physical dimensions, demonstrate the feasibility of this process. Topographical data obtained using a shear-force near-field microscope exhibits spatial resolution in the range 80–100 nm. Optical data in the infrared (near 3.5 μm), using the probe tips in collection mode, indicates an optical spatial resolution approximately $\lambda/15$. © 1999 Published by Elsevier Science B.V. All rights reserved.

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The utility of scanning near-field optical microscopy (SNOM) for applications such as imaging and analysis of semiconductor devices [1–3], microstructural spectroscopy of organic films [4,5], and biological research [6,7], as well as numerous others, has been well documented in the literature. Yet little work has been done in the mid- to long-infrared (from 2–11 μm),¹ largely because the combination of fibers which transmit in this region,

expertise in tapering and making devices from these fibers, expertise in near-field microscopy, and a high-power tunable mid- to long-IR source has been somewhat uncommon. To our knowledge, there has been only one other collaboration of this type: Hong et al. [9] have used commercial multi-mode chalcogenide fiber to obtain near-field images in the mid-IR using a free electron laser (FEL), with spatial resolution approaching 1 μm or about $\lambda/6$. In this letter, we report on the fabrication of SNOM probes from singlemode chalcogenide fiber, and the performance of those probes in measurements using the Vanderbilt FEL, where optical images with resolution approaching $\lambda/35$ were obtained.

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¹ Though some work has been done at 10.6 mm, using CO₂ lasers, see Ref. [8].

These infrared SNOM (IR-SNOM) probes were made from singlemode arsenic sulfide fiber (with outside diameter 80–140 μm and core approximately 10 μm), fabricated and tested at the Naval Research Laboratory as described in Ref. [10]. The tips were made using a standard micropipette puller (Sutter P-2000), using a heated filament to soften the fiber. The laser source supplied with the pipette puller would not work in this case, since the chalcogenide fiber is transparent to CO_2 radiation. In addition, the 160 g static tension of the micropipette puller was often enough to break the thin-diameter chalcogenide fiber, so a counterbalance system had to be devised to relieve some of the static load. As described below, the detection of fiber velocity plays a crucial role in making microtips in this way, so care was taken to use the minimum amount of weight for the counterbalancing.

Variations on standard silica fiber tip geometry from the parameters of velocity, delay, and pull strength available through this pipette puller's interface have been described at length in other reports [11,12], so we will simply state that we used a standard matrix-experimentation approach when searching this parameter space for suitable chalcogenide settings. It should be noted that our parameter set also included variability in laser/heater intensity and counterweighting.

To fabricate the tips using a thermal source, small wound heaters of approximately 6 mm inside diameter were constructed from standard nichrome wire, similar to a heating system we have described previously for making chalcogenide fiber couplers [13]. Counterweighting was adjusted to inhibit static breakage of the fiber, which was completely stripped of its coating and cleaned with solvents.

The most significant influence on tip formation was found to be the heater current, controlling both the heating rate and the ultimate furnace temperature. We observed the usual balance between change in viscosity ($\partial\eta/\partial T$) and the static load, where the current must be optimized to allow accurate sensing of the viscosity trip point which will yield the desired tip shape. After the heater setting, the next biggest influence on tip formation was the velocity parameter of the puller, essentially the viscosity trip-point where the harder solenoid pull would be initiated. With appropriate velocity and

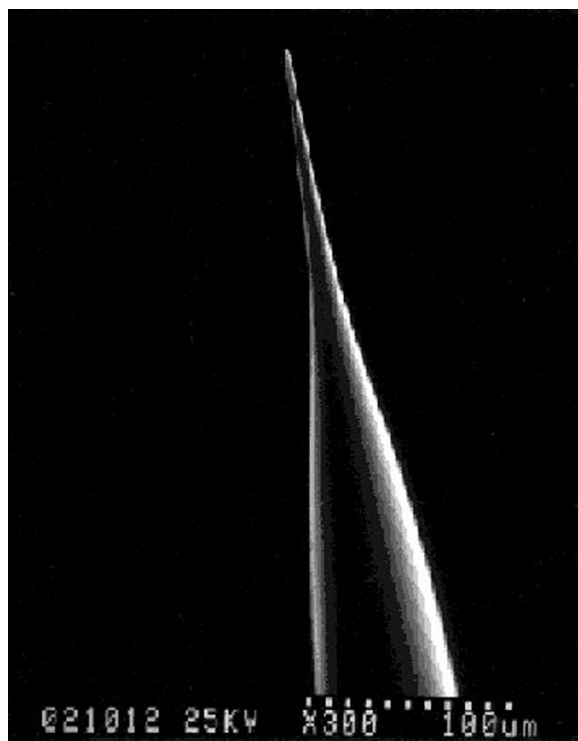


Fig. 1. Thermally produced microtip in singlemode chalcogenide fiber. The tip diameter is approximately 150 nm.

heat settings, the strength of the solenoid pull could be adjusted over a relatively large range to give good results. The delay parameter had little or no observed effect.

Smooth tapers with very small tips, typified by the one shown in Fig. 1, were obtained in this manner, as well as tapers with higher aspect ratios and slightly larger tips. No statistically significant correlation was obtained between the tip diameters and the pulling parameters, but the qualitative results, such as those shown in Fig. 2, were statistically reproducible. Several microtips were made with diameters estimated at 100–200 nm.

However, the apparent physical diameter of the tip may not be an accurate reflection of what spatial resolution it will give, and in any event, actual performance numbers are more relevant. As has been discussed often in the SNOM literature, the resolution of the probe can be highly dependent on the topography of the specimen [6]. To gauge

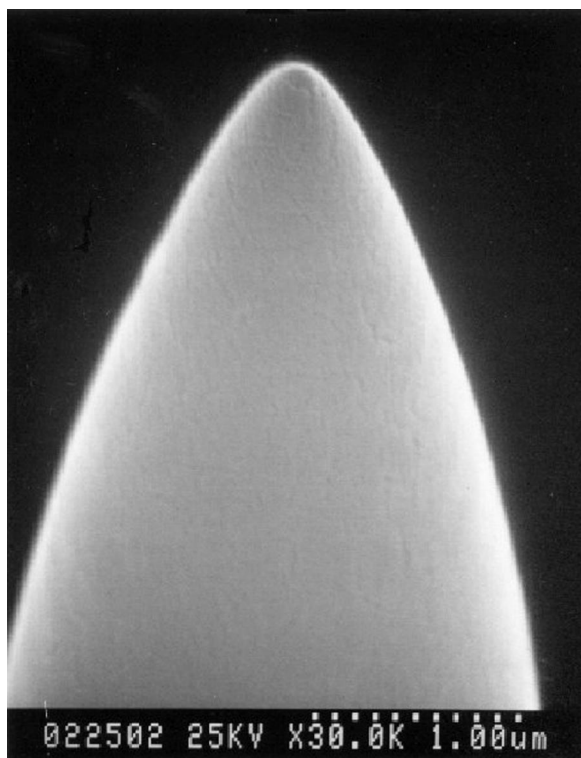


Fig. 2. High-magnification image of chalcogenide fiber SNOM tip. The variation on the surface is due to the metal coating used for SEM preparation.

the performance of our chalcogenide SNOM probes, we tested them in a microscope setup constructed at ISM-CNR [14,15] for use with the Vanderbilt FEL. The first experiments with this setup measured the local reflectivity of a PtSi/Si system between 1.2 and 2.4 μm using silica fiber tips [16]. The reflectivity in the SNOM images revealed features that were not present in the corresponding shear-force images and which were due to localized changes in the bulk properties of the sample.

The specimens examined in this study were polycrystalline diamond films prepared by plasma chemical vapor-phase deposition [17]. The SNOM was first calibrated using standard silica fiber tips, then several topographical (shear-force) images were taken with the chalcogenide tips to verify that the data from the new tips correlated with the old. As shown in Fig. 3, the chalcogenide tips were able to resolve even small features on the individual

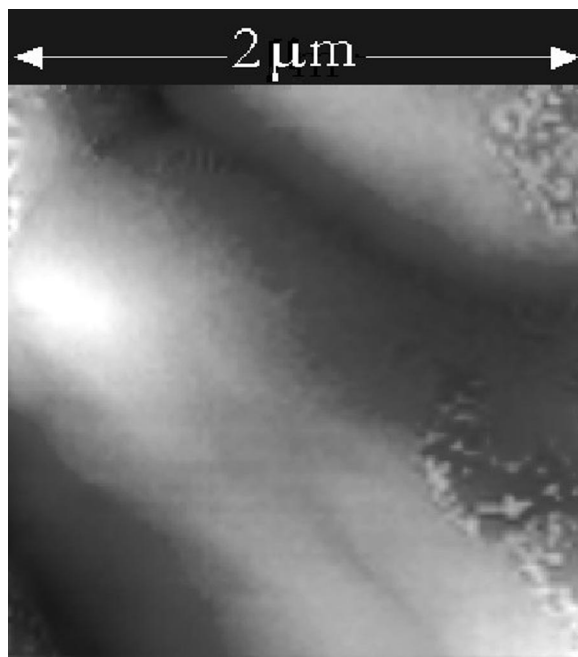


Fig. 3. Topographical (shear-force) image of diamond grains obtained with chalcogenide SNOM tip. The mottled features at the lower and upper right and upper left are most likely impurities or imperfections in the grains. Each scan step was 10 nm; the entire image is 200×200 points.

grains quite well. From these small features, we estimated the best lateral resolution of the probe (from the FWHM of diagonal cross-sections) in shear-force mode to be between 50 and 80 nm. Since the surface was quite rough (at least on a nanoscopic scale), these images indicate that the probe aperture was somewhat smaller than our physical estimates. Also, since irregularities in the probe are expected to be a large detriment to lateral resolution on rough specimens [6,18], these topographical images indicate that the probe tip itself was smoothly rounded.

Since the films had been annealed to remove residual hydrogen and water, it was of greatest interest in the optical experiment to look for absorption near 3.5 μm due to the C–H vibrational stretch mode. Topographical and optical images were taken simultaneously, with the FEL illuminating the specimen over a broad area and the SNOM probe collecting the reflected light. Fig. 4

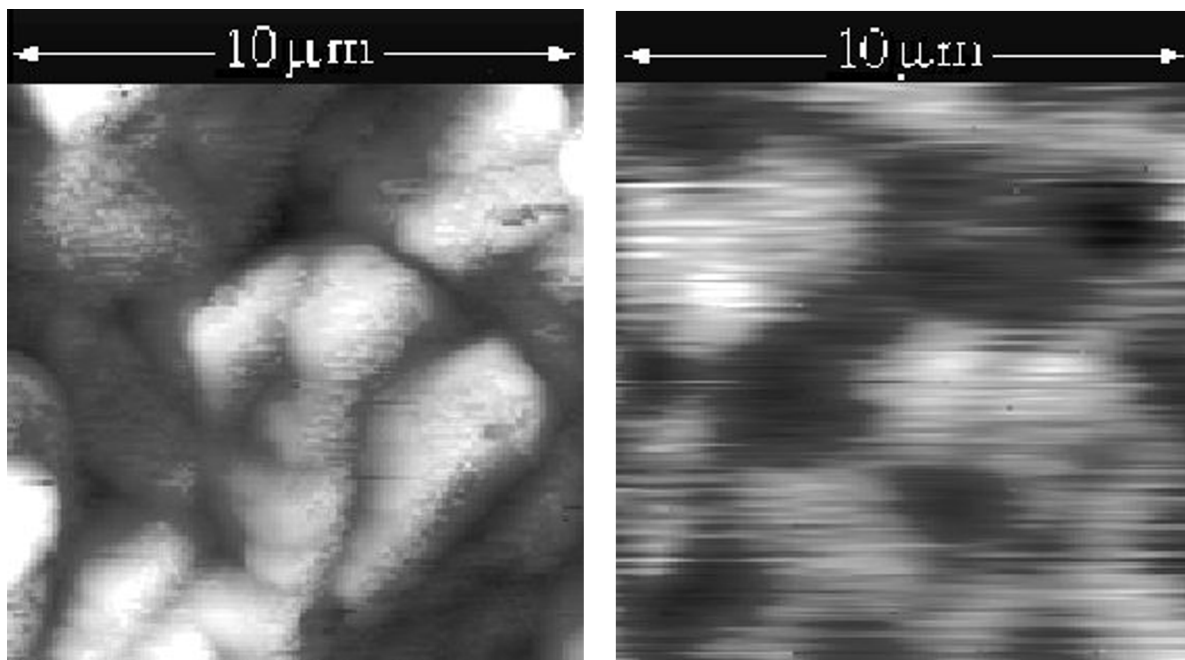


Fig. 4. Comparison of simultaneous topographical (left) and optical (right) images taken with the IR-SNOM. The dark areas in the optical image represent areas where the C–H vibrational absorption was strongest. These optical features were only evident when the FEL was tuned into the C–H absorption band around $3.5\ \mu\text{m}$.

shows a pair of images obtained in this manner, with the FEL operating at $3.5\ \mu\text{m}$. The correlation between optical data and certain features in the topographical image can be seen clearly. When the FEL was tuned outside the C–H absorption band (to 3.2 or $3.7\ \mu\text{m}$), the topographical image was reproduced, while the optical image became featureless, indicating that the dark regions in the optical image of Fig. 4 correspond to regions where the absorption was stronger, perhaps signifying residual hydrogen in the film. Though the relative intensity noise in the FEL (which could not be normalized out in the apparatus used for this report) is evident in Fig. 4, several small features and edges are still resolved. Though the small features were reproducible and would suggest an optical lateral resolution to be between 100 and $150\ \text{nm}$ (about $\lambda/25$ to $\lambda/35$), a more conservative estimate of the resolution would be $\lambda/15$.

These results demonstrate clearly that singlemode chalcogenide fiber can yield excellent results in this novel FEL-SNOM combination.

As this was the first attempt at such an experiment and many of the systems and indeed the probes themselves had not been fully optimized, we expect that these performance numbers will be surpassed in the near future. Applications for IR SNOM, especially at wavelengths longer than about $2\ \mu\text{m}$ where silica fiber does not transmit, range from studies of buried semiconductor interfaces and implants to imaging of sub-cellular features in animal tissues. As interest grows in obtaining high-resolution images of inorganic and biological specimens with distinguishing spectral features in the mid- to long-IR region, these probes could prove an indispensable research tool.

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