February 2000

Materials Letters 42 (2000) 339–344

MATERIALS LETTERS

www.elsevier.com/locate/matlet

Scanning near field infrared microscopy using chalcogenide fiber tips

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Received 19 August 1999; accepted 24 August 1999

Abstract

Chalcogenide glass optical fibers were fabricated into functional apertured probes for near field scanning infrared microscopy. Probe fiber tips were chemically etched and aluminum coated for the purpose of simultaneously collecting near field shear force and optical signals. Surface topography and infrared optical reflectivity data were obtained using the tips in a scanning near field microscope while illuminating an integrated microcircuit with the output from a free electron laser operating at a λ of 4.7 μm. Approximately 25 nm topographical and 100 nm optical lateral resolution were observed.

Keywords: Microscopy; Near-field; Infrared microscopy; Optical fiber; Chalcogenide; Arsenic; Sulfide; Selenide; Free electron laser; Refractive index

1. Introduction

Scanning near-field optical microscopy (SNOM) has been used for imaging and analysis of various biological and nonbiological material surfaces in the visible [1–5]. Scanning near field infrared microscopy (SNIM), an extrapolation of SNOM, has a potential advantage over SNOM since almost all molecules have characteristic absorptions in the infrared. Because of the lack of good quality IR fiber and fiber tips, use of apertured infrared transmitting fiber optics has not been as widespread as compared with silica fibers, hence there are not many references in this area. Hong et al. have used commercially available chalcogenide fiber to obtain near field images in the mid-IR with a reported spatial resolution of 1 μm [6]. We have previously made sulfide fiber tips by a thermal pulling process where

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PII: S0167-577X(99)00201-3

80–100 nm spatial resolution and $\lambda/15$ optical resolution was observed [7]. Recently Unger et al. [8] devised a method for chemically etching chalcogenide fibers. Sulfide and selenide fibers, both of which are chalcogenides, are known to transmit to beyond 6 μm and 10 μm, respectively, and are therefore attractive materials for spectroscopy [9]. In this paper we report the fabrication of chalcogenide near field microscopy fiber tips using an effective and more simple chemical etching process, and, the first use of selenide fiber in near field microscopy to scan an integrated microcircuit surface.

2. Experimental

The Naval Research Laboratory has demonstrated how to make chalcogenide fiber [9]. One to two meter long acrylate-jacketed arsenic sulfide and arsenic selenide fibers having a 250 μm clad diameter and 140 μm core diameter were used with small sections of the jacket removed on the ends using methylene chloride. A SMA adapter was then fitted on one fiber end using phenyl salicylate as support to keep the fiber firmly in place. This was done by melting the phenyl salicylate with a hot plate and applying the liquid state to the adapter opening while holding the fiber in place. Likewise, phenyl salicylate was applied around the fiber projecting from the SMA. This fiber end was polished down (using water as a lubricant) to a smooth clean surface with 12-, 3-, and 0.3-μm polishing discs (Buehler). The endface was then checked with a fiberscope to examine the effectiveness of the polishing. The other end was chemically etched into a fiber tip using a protective layer etching system shown in Fig. 1. At room temperature, the arsenic sulfide tip was etched with KOH and the arsenic selenide tip was etched using a piranha solution (H_2O_2, H_2SO_4). The time-to-completion for the etching process depended on etchant concentration, temperature, and fiber diameter, and typically ranges from 15 min to 1 h. An optical microscope was used to monitor the etching progress.

The tips were later coated with aluminum using a Varian thermal evaporative deposition system at a pressure of 10^{-6} Torr. To create an aperture, the tips were angled 25° to 30° from the line of sight of the evaporation point source. Then, the fiber tips were

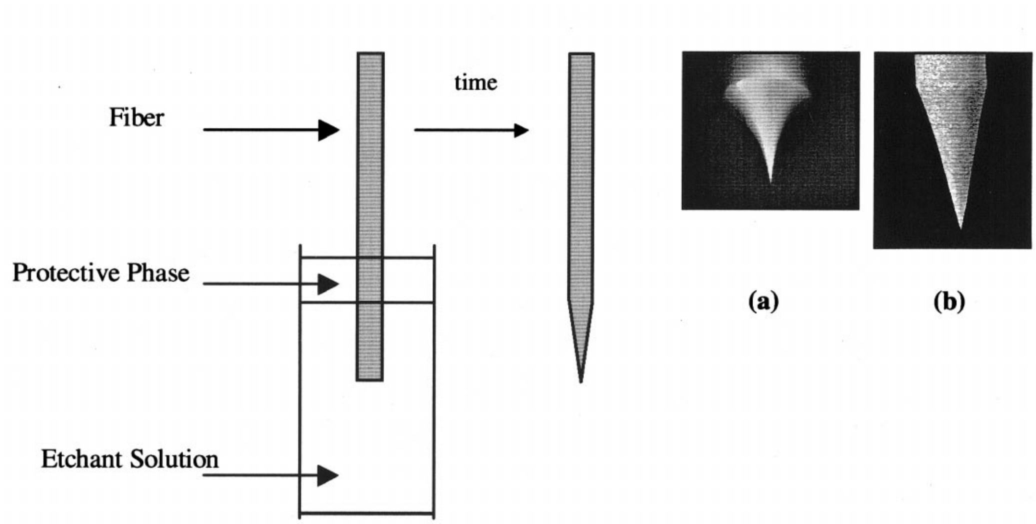

Fig. 1. Etching method used for making fiber tips for near field microscopy. The protective phase used was typically tetramethylpentadecane. An XYZ translation stage was used for positioning the fiber. The SEM image shown on the left (a) is an arsenic selenide tip and on the right (b) is arsenic sulfide.

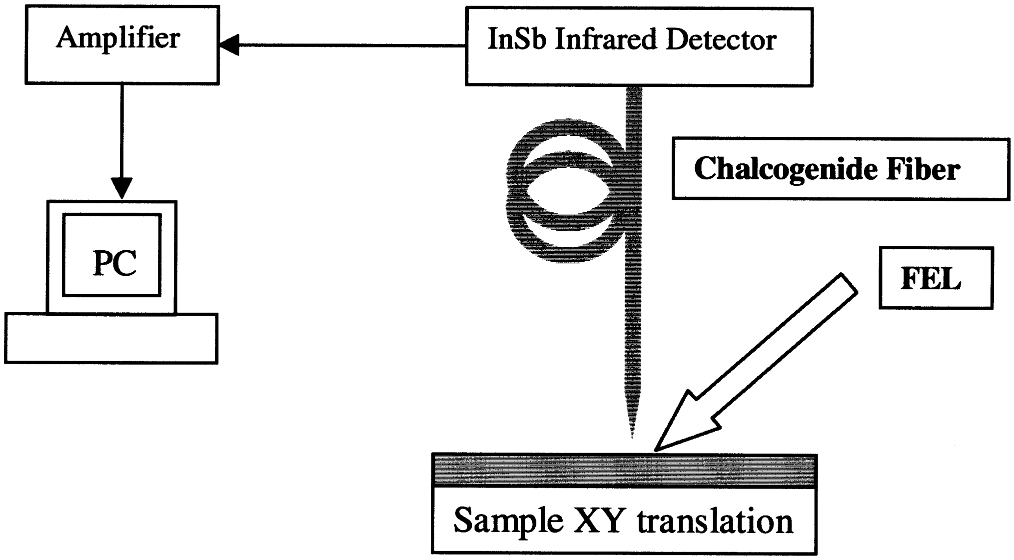

Fig. 2. Schematic of near field apparatus showing the role of the fiber tip as a signal collection tool. The fiber tip was held at a 10-nm *z*-distance from the sample surface by a shear force feedback circuit.

rotated and the coating repeated to apply an even coating. An approximate 100-nm coating thickness was applied.

Near field microscopy was performed at Vanderbilt University using the Vanderbilt Free Election Laser (FEL). The near field microscope [10] rested on an optical bench as part of an optical network where the laser beam could be transmitted and focused onto the sample stage of the microscope (Fig. 2). A fiber tip was then installed into the microscope with the output end lens-coupled to a liquid nitrogen-cooled indium antimonide infrared detector. The sample scanned was an integrated microcircuit (i.e., semiconductor material). Reflectivity images and topography images were simultaneously obtained while scanning over the sample while illuminated by the infrared FEL light. Scanning of the sample took place using a wavelength of 4.7 μm. Scan area capability ranged from 1×1 μm to 17×17 μm. Relative to the scanning area, the 1-mm diameter spot size of the FEL beam was broad and insured good sample illumination. The average energy level of the FEL was approximately 30 mJ/macropulse (4–5 μs pulse-width) at a 30-Hz frequency. Reflectivity was collected using a standard lock-in technique. Topography images were obtained by measuring the shear force signal by synchronous detection using a piezoelectric oscillator and AC/DC converter.

3. Results and discussion

Both fiber materials etched and produced responsive tips although there were some differences. The KOH etching system is less preferred since it attacks the protective phase more aggressively than the piranha solution. Differences in etching were most apparent in tip geometry and surface roughness. The etching process for the selenide material gave a shorter, more sharply tapered tip shape as well as good surface smoothness. The tip shape obtained with the sulfide fiber was longer and gradually tapered, and the surface was considerably rougher (Fig. 3) where surface pits can be on the order of a micrometer or more. The selenide tip geometry is preferred for two reasons: (1) there is little difference

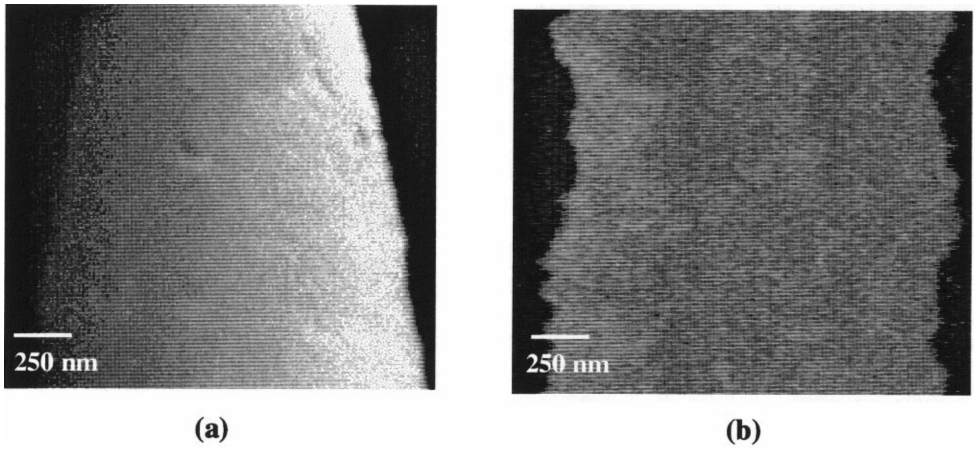

Fig. 3. SEM images of etched arsenic selenide and arsenic sulfide fiber. Arsenic selenide fiber (a) etches more smoothly than an arsenic sulfide fiber (b).

in the resonant frequency at the tip and where the piezoelectric oscillation initiates, and (2) the distance the light must travel to the cladded region is shorter so signal is better conserved.

Differences in the aluminum coating were visually inspected using an optical microscope and showed the coating procedure performed better on the selenide tip compared to the sulfide tip. It is certainly plausible there are adhesion force differences between the etched selenide and sulfide tips, however, the main difference in coating outcome was likely due to inadvertant differences in deposition procedure. For example, the mechanical placement of the fiber tip inside the evaporation chamber differed.

Both the selenide and sulfide fiber tips gave shear mode response and optical signal coupling and transmission once installed into the microscope instru-

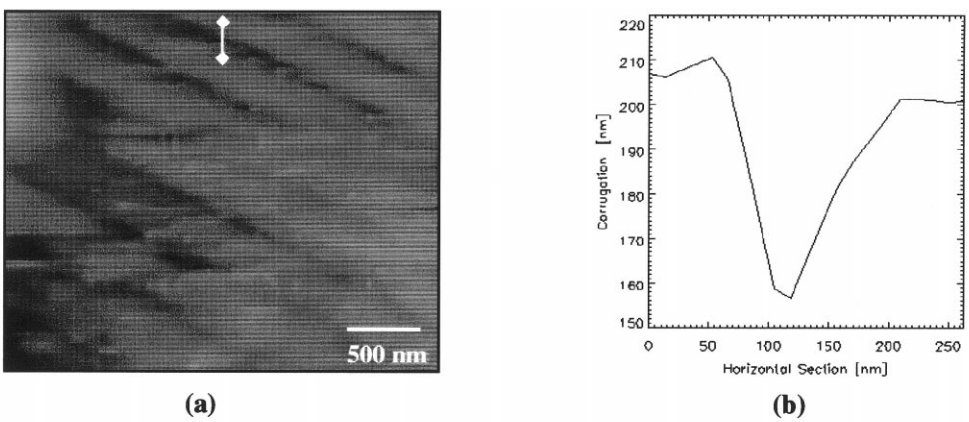

Fig. 4. (a) Topography image obtained with a sulfide tip. Vertical resolution is 5–10 nm. (b) The graphical data corresponds to the cross-sectional data (white line) in the image and shows lateral resolution of 25 nm.

mentation. The sulfide tip's mechanical resonant frequency was approximately 11 kHz whereas the selenide tip resonated at a lower frequency of 8.5 kHz. This difference is mostly due to differences in their stiffness (modulus) although temperature can affect this as well. Surface topography with 25-nm lateral resolution was obtained with both fiber tips. Fig. 4 shows surface topography obtained using a sulfide tip. Lateral resolution of 25 nm was determined by Fig. 4(b) assuming a minimum corrugation (z-axis tip displacement) of approximately 5–10 nm. Although optical transmission was detected through both materials only the selenide fiber gave signal with any optical contrast and resolution. The better quality reflective coating on the selenide tip improves the optical characteristics by limiting the light input to the aperture at the fiber tip. Also, the rough surface produced during the etching process, and the relatively long and gradual taper of the sulfide tip, could have contributed to less effective modal coupling through the fiber tip. Fig. 5 shows corresponding topography and reflectivity images obtained at a λ of 4.7 μm for a 1×1 μm area on the integrated circuit. The topography shows a flat surface on the order of a few nanometers while the corresponding optical image indicates a 100-nm wide dark region that is caused by a reflectivity change in a topographically smooth area. The data suggests that a subsurface defect or a small material heterogeneity may reside at this specific location which produces a band at 4.7 μm. This detected change in signal could be a change in reflectivity caused by a refractive index change and/or absorption change in the material due to unique chemical vibrational modes.

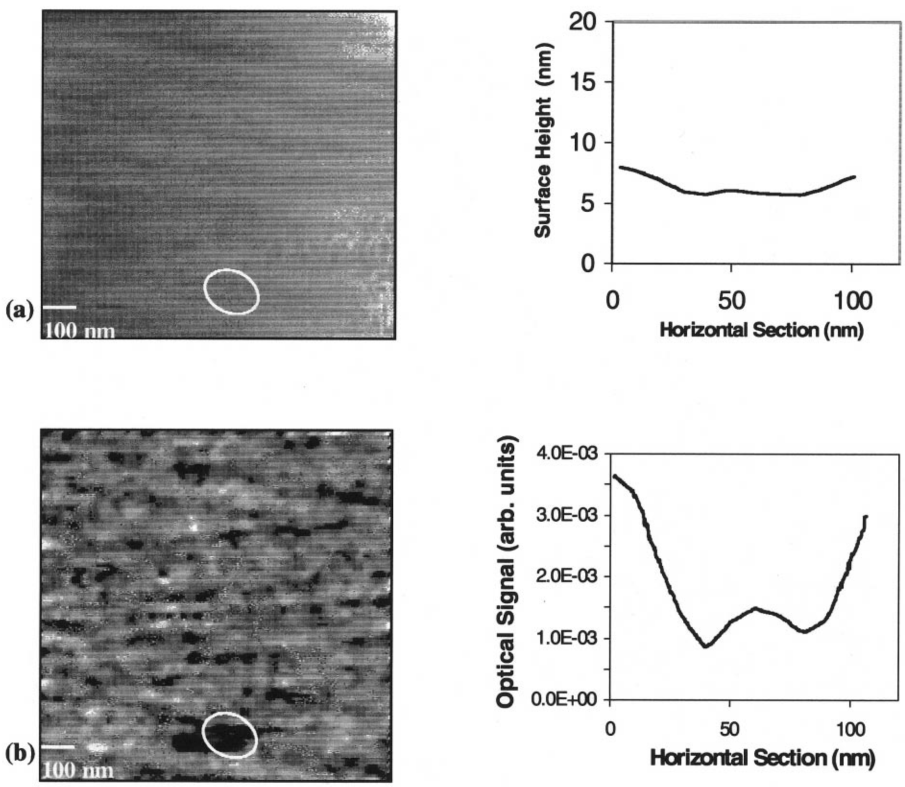

Fig. 5. (a) Topographical image using a selenide tip. The cross section taken in the circled area is very smooth and featureless in the topography. (b) Corresponding optical image at 4.7 μm using a selenide tip. There is a significant localized change ($\sim$ 100 nm) in the optical image indicating a heterogeneity.

4. Conclusion

Chalcogenide glass fibers show good suitability for making chemically etched apertured probe tips for near field infrared microscopy. These chalcogenide fiber tips allow one to topographically and spectroscopically probe materials. Selenide fiber typically etches to a shorter and sharply tapered tip, to a smoother surface, and with better reproducibility than sulfide fiber. It has been demonstrated in this paper that localized changes in semiconductor fabrication may be observed through reflectivity and/or absorption changes using infrared light and warrants more investigation. These insights and the fact that the wavelength transmission capability of the selenide glass extends farther into the infrared make selenide fiber tips a good candidate for near field microscopy probes. Future fabrication improvements will likely enhance optical contrast and resolution for even better probing of material surfaces.

Acknowledgements

The authors gratefully acknowledge the help of Fred Kung (NRL) with fiber assembly consultation and Guillermo Villalobos (NRL) with SEM assistance. Efforts of Vinh Nguyen (NRL) and Reza Mossadegh (NRL) are greatly appreciated. We also want to thank the control operators at Vanderbilt University for their assistance running the Free Electron Laser.

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