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Focussed ion beam machined cantilever aperture probes for near-field optical imaging

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Key words. Aperture, cantilever probe, FIB, micro-machining, NSOM.

Summary

Near-field optical probe is the key element of a near-field scanning optical microscopy (NSOM) system. The key innovation in the first two NSOM experiments (Pohl *et al.*, 1984; Lewis *et al.*, 1984) is the fabrications of a sub-wavelength optical aperture at the apex of a sharply pointed transparent probe tip with a thin metal coating. This paper discusses the routine use of focussed ion beam (FIB) to micro-machine NSOM aperture probes from the commercial silicon nitride cantilevered atomic force microscopy probes. Two FIB micro-machining approaches are used to form a nanoaperture of controllable size and shape at the apex of the tip. The FIB side slicing produces a silicon nitride aperture on the flat-end tips with controllable sizes varying from 120 nm to 30 nm. The FIB head-on drilling creates holes on the aluminium-coated tips with sizes down to 50 nm. Nanoapertures in C and bow tie shapes can also be patterned using the FIB head-on milling method to possibly enhance the optical transmission. A transmission-collection NSOM system is constructed from a commercial atomic force microscopy to characterize the optical resolution of FIB-micro-machined aperture tips. The optical resolution of 78 nm is demonstrated by an aperture probe fabricated by FIB head-on drilling. Simultaneous topography imaging can also be realized using the same probe. By mapping the optical near-field from a bow-tie aperture, optical resolution as small as 59 nm is achieved by an aperture probe fabricated by the FIB side slicing method. Overall, high resolution and reliable optical imaging of routinely FIB-micro-machined aperture probes are demonstrated.

Introduction

As one of scanning probe microscopy (SPM) techniques, near-field scanning optical microscopy (NSOM) uses an optical probe to couple the evanescent components of the electromagnetic field that decays exponentially from the sample surface during the tip–sample interaction. Near-field optical imaging with sub-wavelength resolution down to a few tens of nanometers has been demonstrated, far beyond the diffraction-limited resolution that can be achieved by a conventional optical microscopy, and therefore has been widely used in many studies, such as single molecule detection (Betzig & Chichester, 1993), surface enhanced Raman spectroscopy (Ayars & Hallen, 2000), nanofabrication (Smolyaninov *et al.*, 1995), high-density data storage (Betzig *et al.*, 1992) and many other subjects involving optical near-field (Dunn, 1999; Hecht *et al.*, 2000).

The near-field optical probe is the key element in an NSOM system. For the aperture-type NSOM, the size of the aperture at the apex of the probe determines the ultimate optical resolution. In fact, the key innovation in the first two independent NSOM experiments is the fabrications of a sub-wavelength aperture at the apex of a transparent probe tip (quartz rod, Pohl *et al.*, 1984 and taped micro-pipette, Lewis *et al.*, 1984) with a thin metal coating. Nowadays, tapered optical fibres and micro-fabricated cantilever aperture probes are commercially available benefiting from the rapid development of various fabrication techniques for these two kinds of aperture probes. However, the commercial NSOM probes of high resolution (<50 nm) are normally marked at a high price tag and are not reproducible. The probe fabrication, particularly the fabrication of high-resolution apertures of high quality in a reproducible, simple and low-cost manner is of great interest for the development of NSOM instrumentation.

There has been a variety of fabrication approaches proposed and investigated in the literature to form the sub-wavelength aperture at the apex of a sharply pointed tip. Squeezing and pounding a metal-coated tip against a hard surface (Pohl *et al.*, 1984; Saiki & Matsuda, 1999; Naber *et al.*, 2002) is a simple and straightforward method. Since it is a mechanical wear process, the size and shape of the formed aperture

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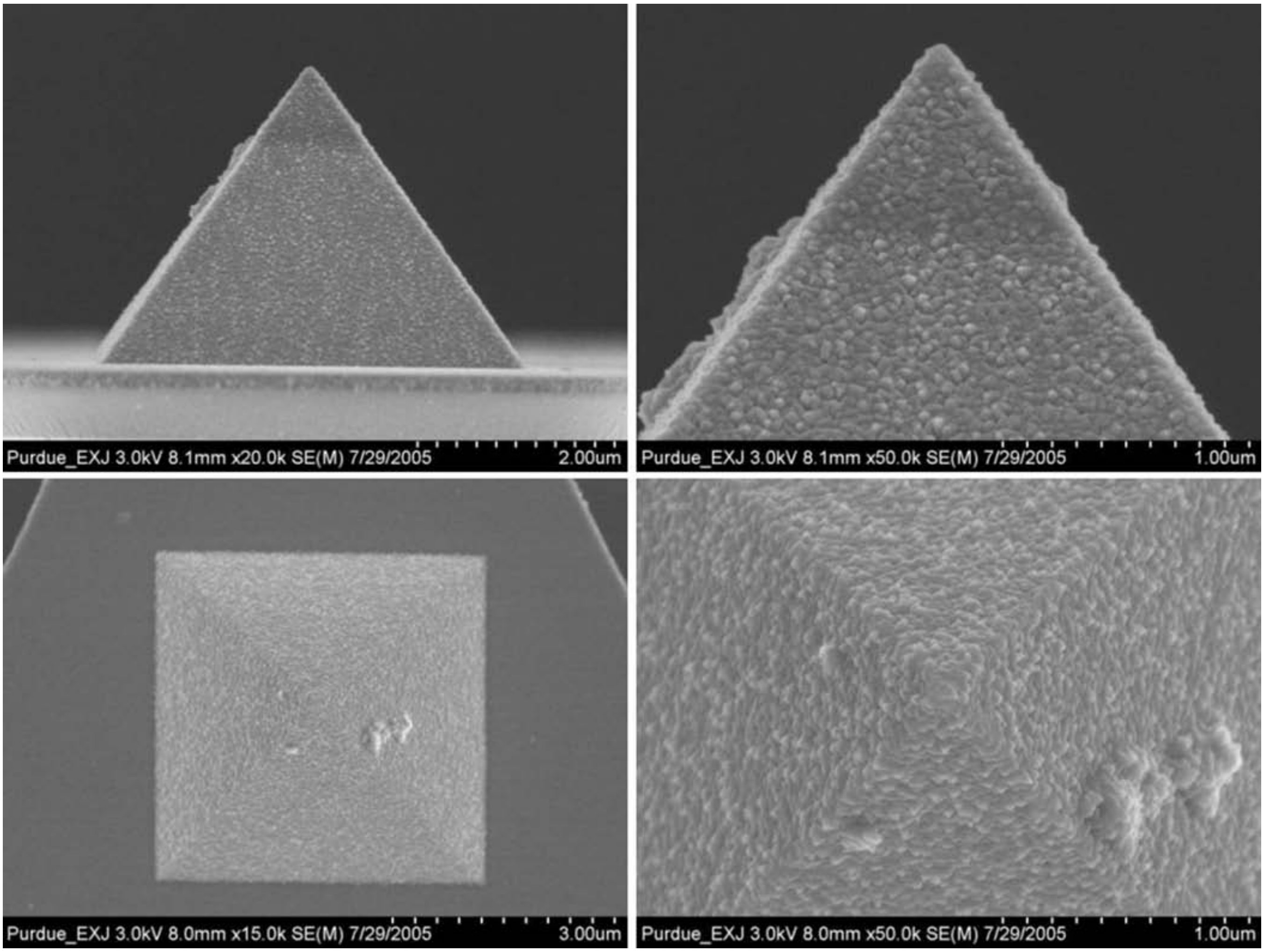

Fig. 1. SEM images of side view and top view of an aluminum-coated pyramidal tip on an AFM cantilever.

needs good control. Angled metal deposition (Betzig *et al.*, 1991) shadows the apex of the tip and forms the aperture naturally in the deposition process. However, it is also a great challenge in forming a controllable aperture size and shape. Wet (Saiki *et al.*, 1996) and solid (Mulin *et al.*, 1997; Bouhelier *et al.*, 2001) electrolytic demetallization approaches allow reproducible formation of aperture in a more controllable fashion but often requires an elaborated experimental set-up. Laser-assisted selective corrosion (Haefliger & Stemmer, 2003) is able to produce high-quality aperture probes by utilizing aluminium corrosion in water under the evanescent field. This method only requires a simple total internal reflection optical set-up, but it is limited to the selection of metal coating due to the inherent aluminium corrosion mechanism. In the batch fabrication process of cantilevered aperture probes, selective reactive ion etching is often used to form a sub-wavelength aperture, which involves multiple micro-fabrication steps and various complicated tools (Mihalcea *et al.*, 1996; Ruiter *et al.*, 1996; Minh *et al.*, 2000; Choi *et al.*, 2003).

As a high-precision patterning technique, focussed ion beam (FIB) milling has been introduced to fabricate a sub-wavelength aperture at the apex of fibre-based (Muranishi *et al.*, 1997; Lacoste *et al.*, 1998; Veerman *et al.*, 1998) and cantilever-based (Dziomba *et al.*, 2001; Mitsuoka *et al.*, 2001) probes. In the FIB processing, an ion beam of high energy (typically 10–100 keV) is focussed into sub-50 nm or smaller size, and directed to impinge on the metal-coated tip. The metal material at the apex of the tip is consequently removed to form an aperture. The shape of the aperture fabricated by FIB processing could be well defined by irradiation pattern of the ion beam, and the size of the aperture could be precisely controlled by the ion irradiation dose. It has also been pointed out that the serial process of FIB technique could be compensated by combining the FIB technique with batch micro-fabrication process of cantilever probes to improve the throughput and reproducibility (Dziomba *et al.*, 2001). The major concern of FIB approach is the availability of the expensive tool. Otherwise, it is the most desirable and high-precision approach to fabricate reliable aperture NSOM probes with resolution better than 100 nm.

This paper discusses the routine use of FIB to micro-machine NSOM aperture probes from the commercially available silicon nitride cantilevered atomic force microscopy (AFM) probes. The complete fabrication procedure and details are explained. The aperture probes fabricated by FIB side slicing and head-on drilling methods are presented with controllable aperture size ranging from 120 nm to 30 nm. Patterning of nanoapertures with novel shapes by the FIB head-on drilling method is also discussed as a potential approach to improve the power throughput of an aperture probe. The high-resolution optical imaging capability of routinely FIB-micro-machined aperture probes is demonstrated by using the aperture probe as a near-field collector in a transmission-collection NSOM system constructed from a commercial AFM.

Aperture fabrication

To fabricate aperture NSOM probes, we start with the standard silicon nitride cantilevered AFM probe, which are commercially available (e.g. Veec, Santa Barbara, CA, USA). The reason for using cantilevered AFM probes instead of fibre-based probes includes the robustness, ease of handling and ease of implementing in a standard AFM system. The silicon nitride cantilevered probe we used contains four 0.6-μm-thick V-shaped cantilevers at two lengths of 100 or 200 μm and two widths of 10 or 20 μm. The nominal spring constants of the four cantilevers are between 0.06 N m^{-1} and 0.52 N m^{-1} depending on the dimensions. A pyramidal-shaped hollow tip (about 3 μm in height, 70° opening angle, 20–40 nm nominal tip radius and about 0.5 μm in side wall thickness) is located at the very end of the cantilever. Both the large opening angle and high refractive index of silicon nitride ($n = 2.35$) can contribute to the high power throughput of NSOM probes fabricated from this type of AFM probes.

The tip side of the AFM cantilever is first deposited with about an 86-nm-thick layer of aluminium film. It should be noted that other metals can also be used as the coating material. High deposition rate (10–20 Å s^{-1}) is necessary to limit the cantilever bending after aluminium coating and ensure a pinhole-free film on the tip. Figure 1 shows SEM images of side and top views of an aluminium-coated tip on the AFM cantilever. The gold coatings on the back side of the cantilever (opposite to the pyramid) are partially removed by FIB milling (FEI DB 235 dual beam machine, 30 keV Ga+ ions with 10 pA beam current) in order to let light transmitted through the tip. As shown in the SEM image of Fig. 2, a window of about 0.65×0.65 μm^2 is opened on the back side of the tip. To make an aperture opening at the apex of the tip, two FIB micro-machining approaches, FIB side slicing and head-on drilling, are employed.

The FIB side slicing method is the same as the technique used to make a flat NSOM fibre probe (Veerman *et al.*, 1998), in which ion beam is irradiating from the side of the tip at a 90° angle from the normal. The aluminium at the very end of the tip is sliced away until the silicon nitride core is exposed to form a small aperture. 30 keV Ga^+ ions with 10 pA beam current is used in the slicing process and the typical milling duration to make a sub-100 nm aperture is less than 10 s. Figure 3 shows the SEM images of the same tip as shown in Fig. 1 after FIB side slicing. It can be clearly seen that the sharp apex of the tip is removed and left with a flat end of 280 nm in side length by the ion beam irradiation. A silicon nitride core in square shape and of 80 nm × 80 nm in size is visible (the dark square in the middle of the tip as shown in the lower right image in Fig. 3) and can be used as a dielectric aperture for near-field imaging in the UV to near-IR wavelength range. The aluminium islands on the side walls, possibly induced by debris on the tip before aluminium deposition, are away from the aperture and do not affect the imaging performance of the probe. The size of the silicon nitride core can be controlled by varying the slicing height from the apex. As shown in Figs 4(a)–(d), the fabricated dielectric apertures have sizes varied from 120 nm down to 30 nm. The smallest aperture size that can be fabricated by the FIB slicing method is determined by the apex size of the original AFM tip, which is in the range of 20–40 nm for this particular type probes. The shape of the aperture fabricated by FIB side slicing is close to square since the tip has the symmetric pyramidal shape.

FIB head-on drilling is irradiating the ion beam from right on to the tip (perpendicular to the cantilever surface). Particular milling patterns can be used. To irradiate ion beam exactly at the apex of the tip, an ion beam image is taken first at high

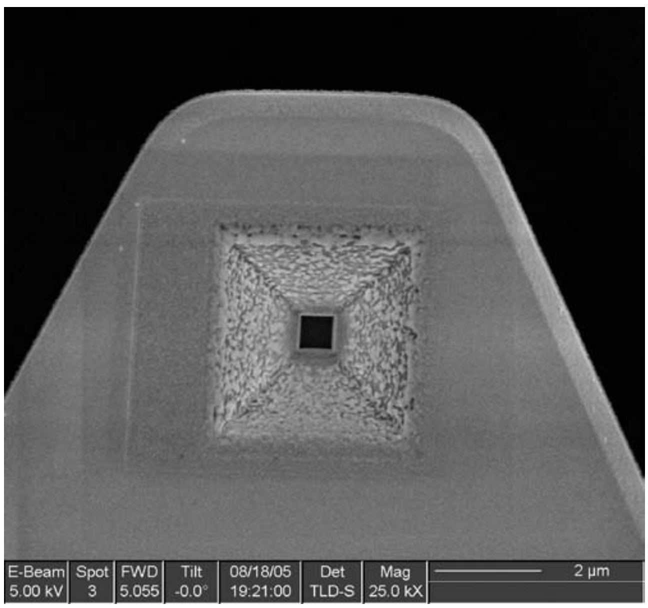

Fig. 2. SEM image of the back side of a NSOM probe. A 0.65×0.65 μm^2 window is opened by FIB milling.

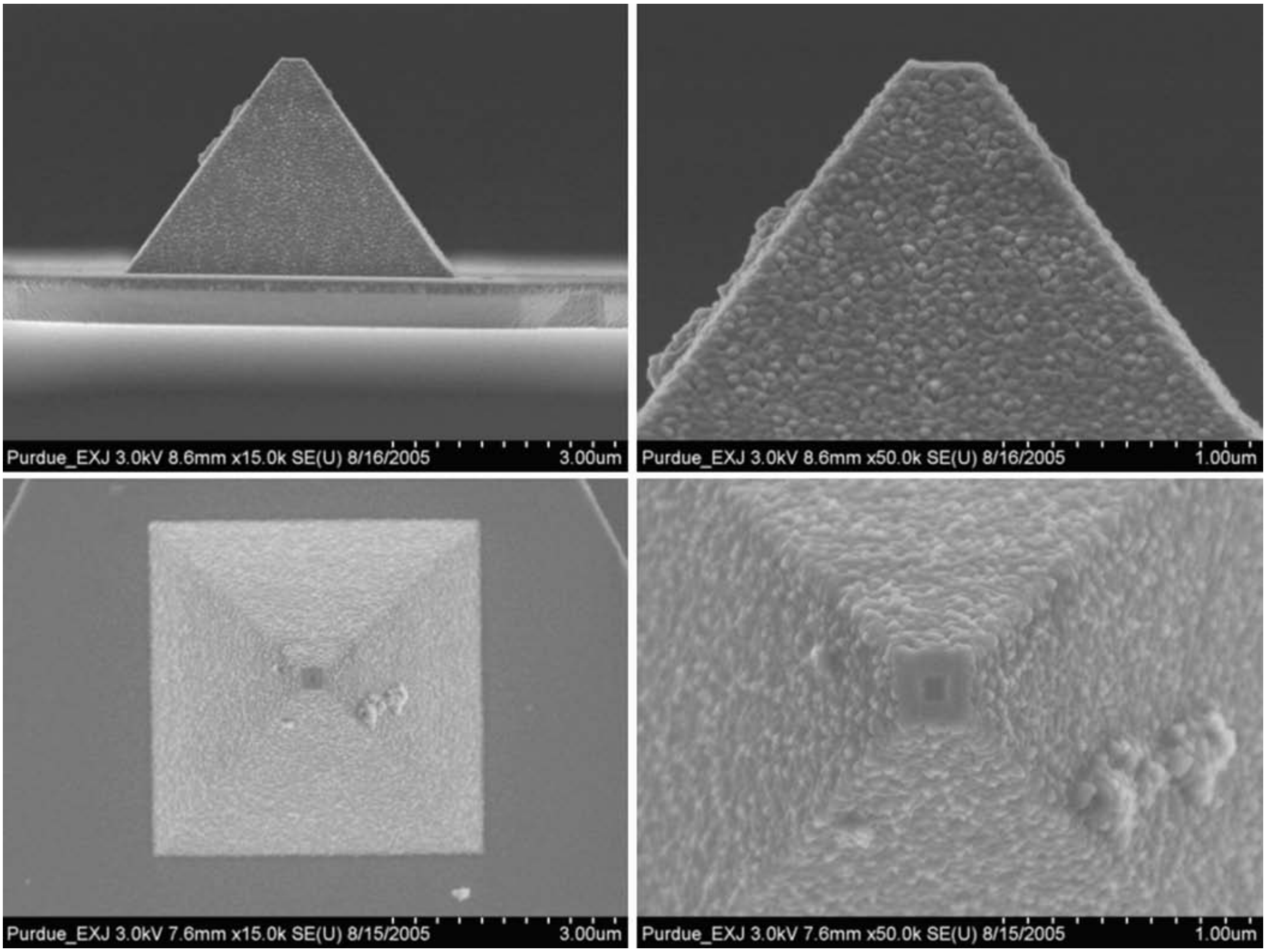

Fig. 3. SEM images of side view (first row) and top view (second row) of the same tip shown in Fig. 1 after FIB side slicing. A silicon nitride core 80 nm by 80 nm in size is exposed after aluminum removal.

magnification (50–100 kX) followed by the exposure pattern positioning. The exposure is then immediately executed to limit the image drift. Coarse beam scan needs to be employed to minimize the ion exposure damage to the aluminium film during the ion beam imaging. During the FIB head-on drilling, both the aluminium and silicon nitride core can be removed. As shown in Fig. 5, a through hole in various sizes can be formed at the tip apex. The smallest size of the aperture made by this method is limited by the finite ion beam size and beam tail effect. The spot size of ion beam normally can be controlled to be as small as 10 nm at low current of 1 pA. However, it is difficult to drill a sub-50 nm through hole directly at the exact apex of the tip due to the image drift at extremely high magnification. However, the advantage of FIB head-on drilling is the ability to pattern nanoapertures in various shapes. In addition to commonly used shape, for example, circular shape (Muranishi *et al.*, 1997; Lacoste *et al.*, 1998) or rectangular shape (Danzebrink *et al.*, 1999; Dziomba *et al.*, 2001), special apertures in C and bow-tie shapes can be fabricated by defining the desired exposure pattern of the ion beam as shown in Fig. 6. In making this type of apertures, additional fabrication steps need to be used, for example, an aluminium thin film is coated after a small platform is created on the AFM tip by FIB side slicing. These special shapes can possibly provide a high transmission throughput (Shi *et al.*, 2001; Sendur & Challener, 2003; Jin & Xu, 2004). (Characterizations of the throughput of these apertures are currently underway.)

Resolution of FIB micro-machined NSOM probes

To characterize the optical resolution of fabricated NSOM probes, an NSOM system is constructed. Figure 7 shows the schematic diagram of this NSOM system operated in the transmission-collection mode. The linearly polarized laser source at $\lambda = 633$ nm (helium neon laser) or $\lambda = 458$ nm (argon ion laser) is used to illuminate a test sample from the bottom

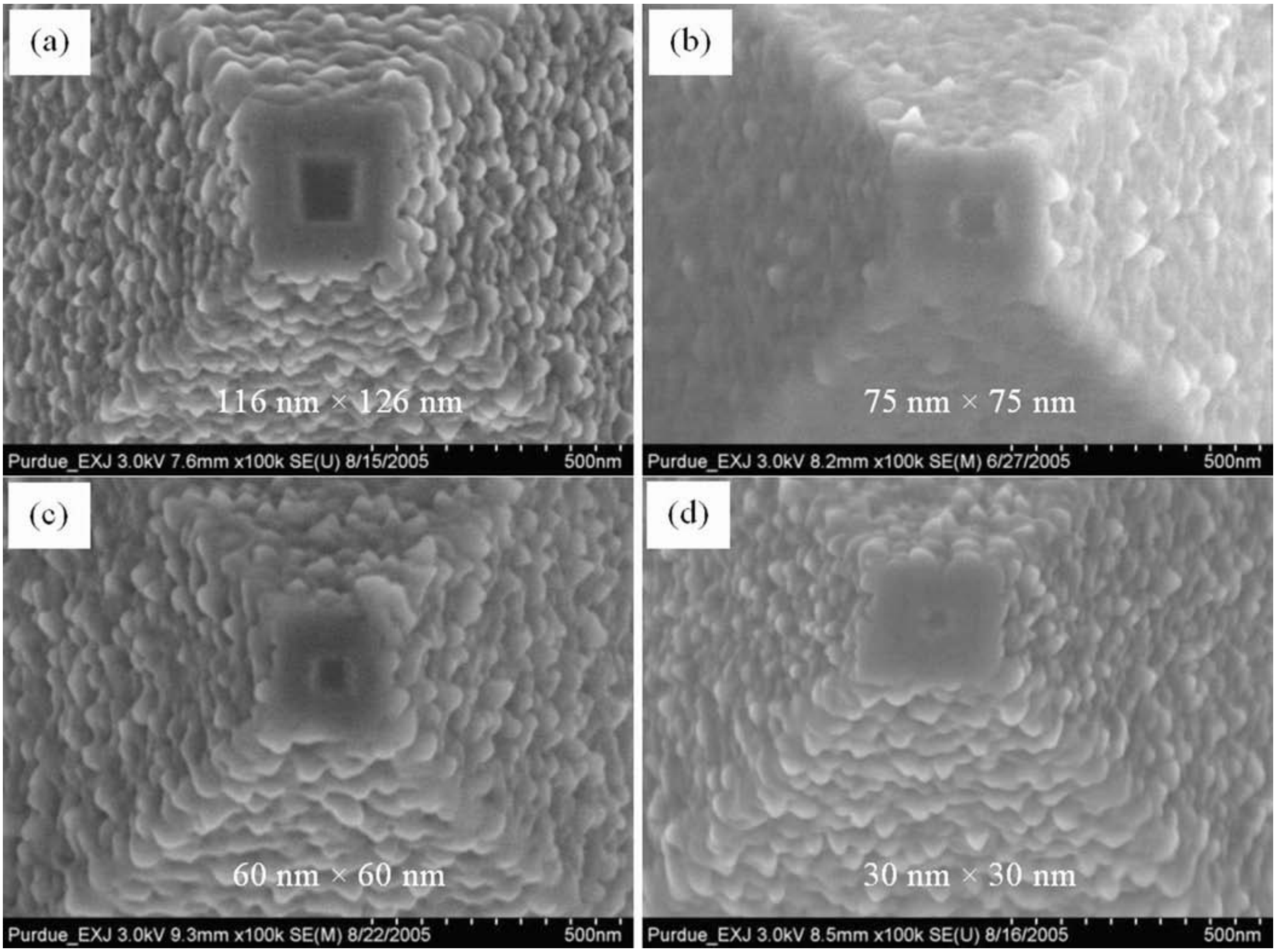

Fig. 4. SEM images of NSOM probes with a silicon nitride core in various size fabricated by FIB side slicing method.

by placing a prism underneath the sample. The test sample contains FIB-patterned nanoapertures in aluminium on the quartz substrate. The transmitted light from the aperture in the sample is collected by the NSOM probe, which contains a FIB-micro-machined nanoaperture at the apex as described earlier. The soft contact between the probe and sample surface is achieved by maintaining a small and constant normal force based on the feedback of diode laser beam deflected on the cantilever. A 20× long working distance objective (Mitutoyo MPlan Apo SL 20×, NA = 0.28, WD = 30.5 mm, Kawasaki, Kanagawa, Japan) and a set of lens, beam splitter and filters are used to direct the collected light to a photo-multiplier tube (PMT 9107B from Electron Tubes, Ruislip, UK). The photons detected by the PMT are counted by a photon counter (Stanford SR400, Sunnyvale, CA, USA), whose output (D/A output port) is connected to the AFM controller called AEM through a low-voltage module (LVM). The photon counter needs to be synchronized with the AFM scan. This is accomplished by setting the photon counting period of each data point (T_{set}) and the internal time between two data points in the photon counter (T_{dwell}), as well as the delay time (T_{delay}) after each line in the AFM scan software. A 100-μm pinhole is placed in the first image plane of the sample surface in order to block the stray light and to improve the imaging quality. A high precision piezo scanner is used for raster-scanning the aperture sample and the optical signal described earlier is recorded to form an NSOM image after scanning.

Figure 8(a) shows the SEM image of an NSOM probe fabricated by FIB head-on drilling method. The SEM image is taken a certain angle from the normal of the tip so the details of the aperture can be seen. The probe has an overall opening of 150 × 150 nm in size, but the silicon nitride core preserved in the middle of the opening (the slightly brighter area in the aperture), as a result of the different etching rate between aluminium and silicon nitride, makes the effective aperture size smaller as we will see from its optical resolution. Both the AFM topography and NSOM images can be obtained after the two-dimensional scan using this particular probe. Figure 8(b)

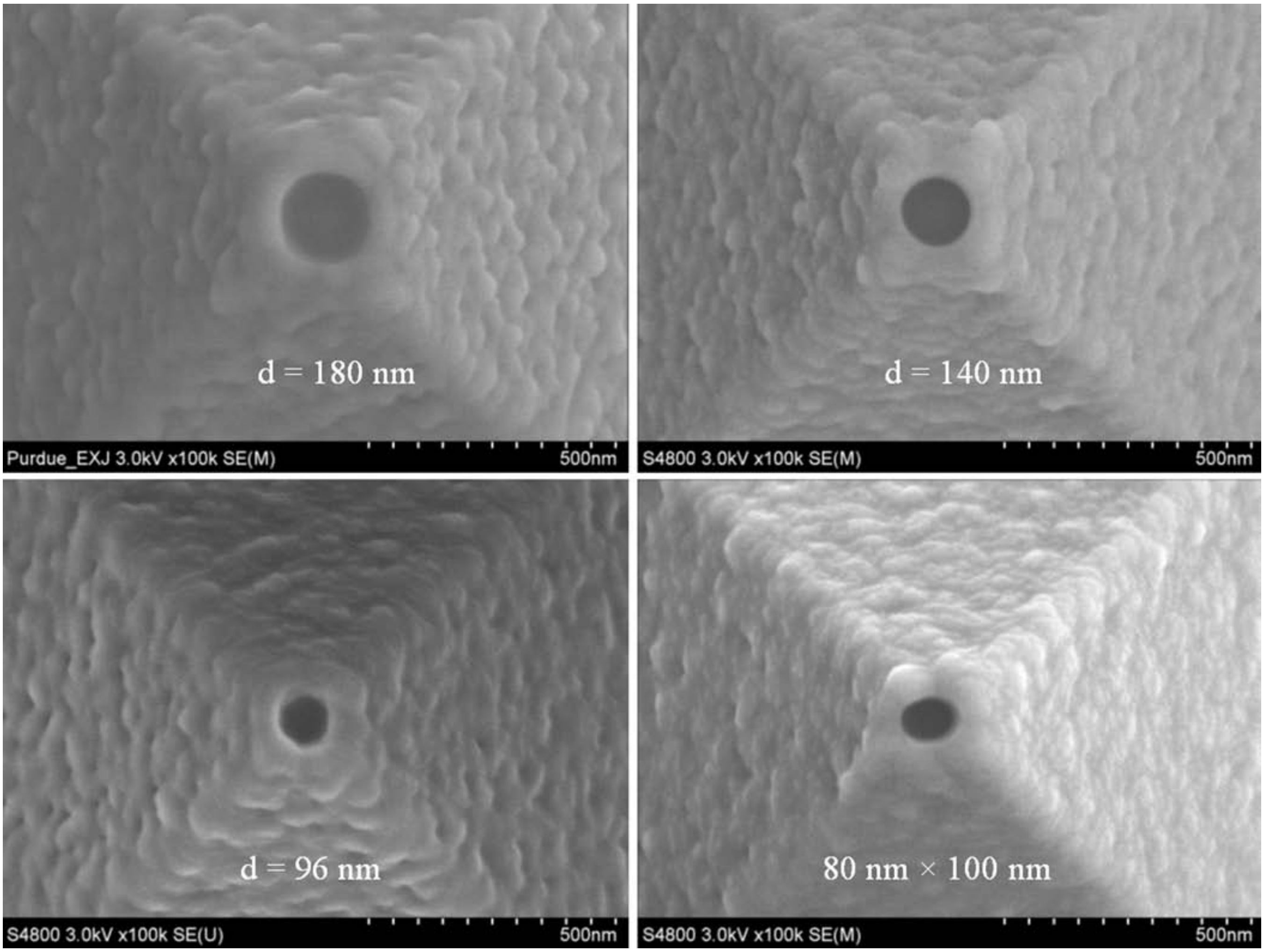

Fig. 5. SEM images of NSOM probes with an aperture in various size fabricated by FIB head-on drilling method.

shows the AFM topography of a pair of 160-nm holes separated by 80 nm. The inset shows the SEM image of the hole pair. These two holes are clearly separated in the simultaneously recorded NSOM image as shown in Fig. 8(c). The topography imaging is obtained because of any small aluminium protrusion near the aperture rim or the silicon nitride core in the middle since the tip made by the FIB head-on drilling process is not even (the head-on milling does not produce an even surface). In fact, there is an offset between the AFM and NSOM images as seen in Figs 8(b) and (c), which further confirms this assertion. To determine the optical resolution, a line scan is performed as shown in Fig. 8(c), and the NSOM intensity profile along this line scan is shown in Fig. 8(d). The measured 10–90% edge resolution is 78 nm for this aperture probe, which is about 1/6 of the 458 nm illumination wavelength. This optical resolution is also smaller than the overall size of the aperture, indicating that the silicon nitride core determines the near-field optical resolution for this type of NSOM probes.

To characterize the NSOM probes of higher optical resolutions, a point-like light source is needed. For this purpose, a bow-tie–shaped nanoaperture is fabricated in the aluminium sample by FIB milling as shown in Fig. 9(a), which is able to provide a nanoscale near-field spot with enhanced optical transmission under proper illumination (Sendur & Challener, 2003; Jin & Xu, 2005, 2006). The bow-tie aperture has an outline of about 216 nm × 248 nm and a 33-nm gap between the two tips, and the size of the near-field spot produced by the bow tie is about the same as the gap between the two tips, 33 nm. The NSOM probe prepared by the FIB side slicing method as shown in Fig. 9(d) is used to scan the optical near field from this bow-tie aperture. This aperture tip has a silicon nitride core of 45 nm × 45 nm surrounded by aluminium. The overall size of the tip end is 257 nm × 257 nm as measured from the side of the tip (see the inset of Fig. 9(d)). A 458 nm argon ion laser polarized across the bow-tie tips is used as the illumination source in this measurement. The obtained NSOM

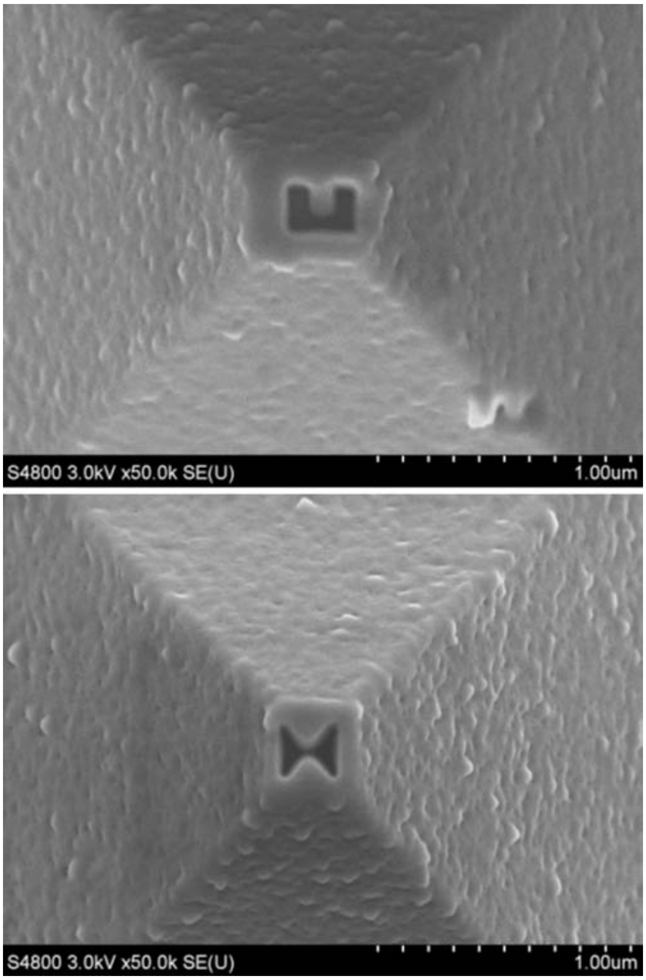

Fig. 6. SEM images of NSOM probes with a C and bowtie aperture at the apex.

image is displayed in Fig. 9(b). Since the flat end of the probe is larger than the size of the bow-tie aperture, no topography information can be obtained. The size of the NSOM spot is 88 nm × 68 nm in FWHM. However, this light spot is essentially representing the convoluted coupling between the optical near field from the bow-tie aperture and the aperture probe (Jin & Xu, 2006), meaning the actual light spot is smaller. The edge resolution of this probe from the line scan profile is measured to be 59 nm, approximately the sum of the aperture size 45 nm and twice the skin depth of aluminium 6.5 nm at the 458 nm wavelength.

Conclusions

In summary, two FIB micro-machining approaches, side slicing and head-on drilling, are employed to fabricate aperture NSOM probes. The detailed fabrication procedure has been presented. Both FIB approaches allow the precise control of the aperture formation at the apex of the aluminium-coated tip. The FIB side slicing is able to produce a silicon nitride aperture on the flat-end tips with controllable sizes varying from 120 nm to 40 nm. The FIB head-on drilling, on the other hand, is capable to pattern nanoapertures of various shapes, including circular, square, C and bow-tie shapes. To characterize the optical resolution of FIB-micro-machined aperture tips, an NSOM system using the aperture probe as the near-field collector is constructed. By imaging a closely patterned pair of nanoholes, the optical resolution of 78 nm is demonstrated by an aperture probe fabricated by FIB head-on drilling. The same probe is also able to obtain a topography image simultaneously benefitting from the aluminium protrusion on the aperture rim. By mapping the nanoscale optical near field from a bow-tie aperture, optical resolution as high as 59 nm is achieved by an aperture probe fabricated by the FIB side slicing method. These measurements demonstrated high resolution and reliable optical imaging of the FIB-micro-machined aperture probes.

Acknowledgements

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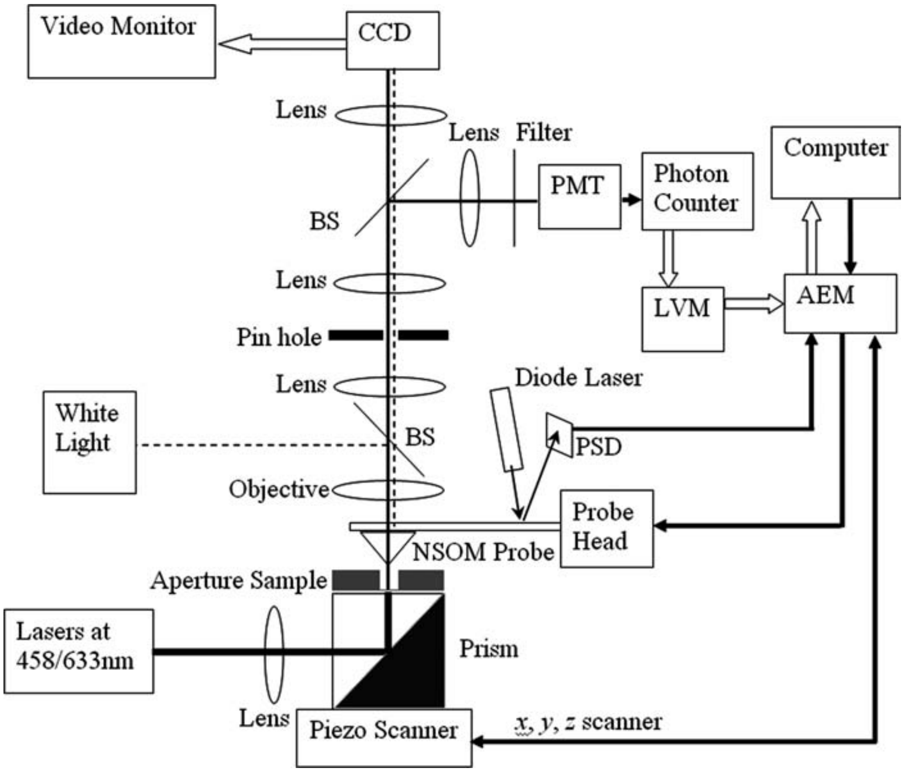

Fig. 7. Schematic view of the NSOM system in transmission-collection mode.

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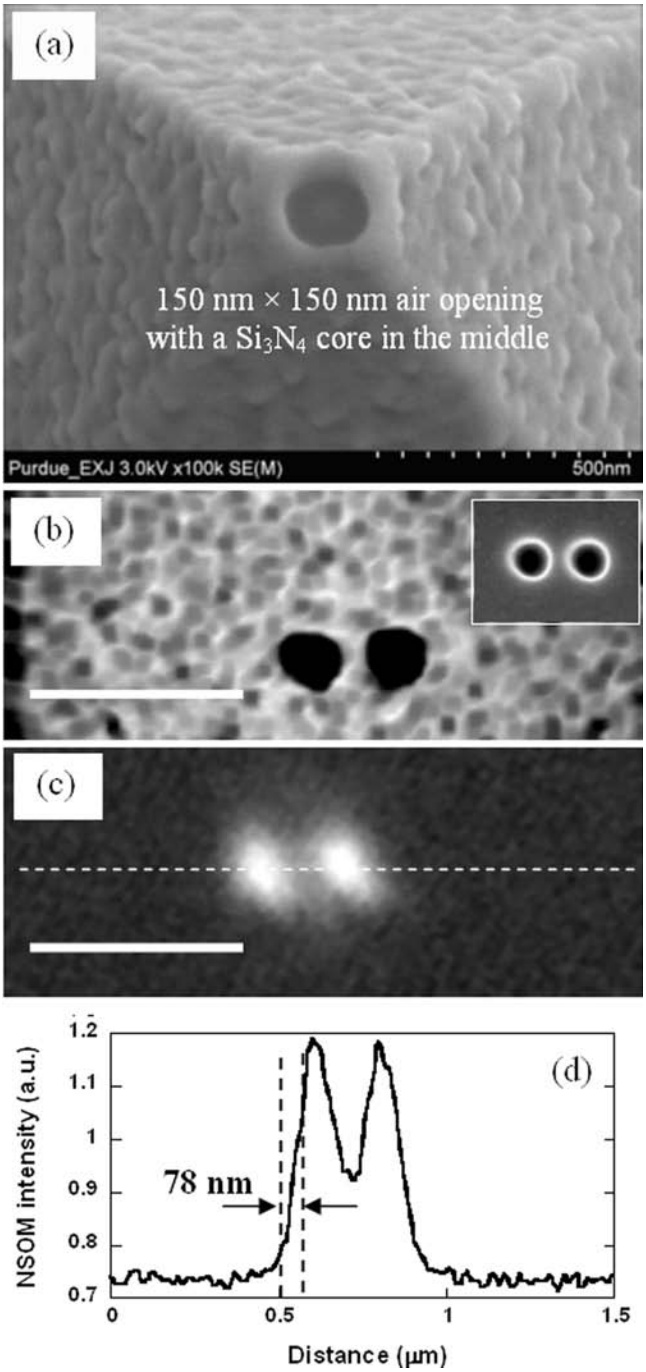

Fig. 8. Characterizing the optical resolution of a NSOM aperture probe fabricated by FIB head-on drilling method. (a) SEM image of the NSOM probe showing a 150 nm × 150 nm aperture with a silicon nitride coer in the middle, (b) AFM topography image of a pair of nanoholes in the aluminum sample obtained by the NSOM probe (The inset is the SEM image of the hole pair), (c) NSOM image of the nanoholes obtained by the NSOM probe, and (d) intensity profile along the line scan on the NSOM image showing the 10%-90% edge resolution is 78 nm. The scale bars in (b) and (c) are 500 nm.

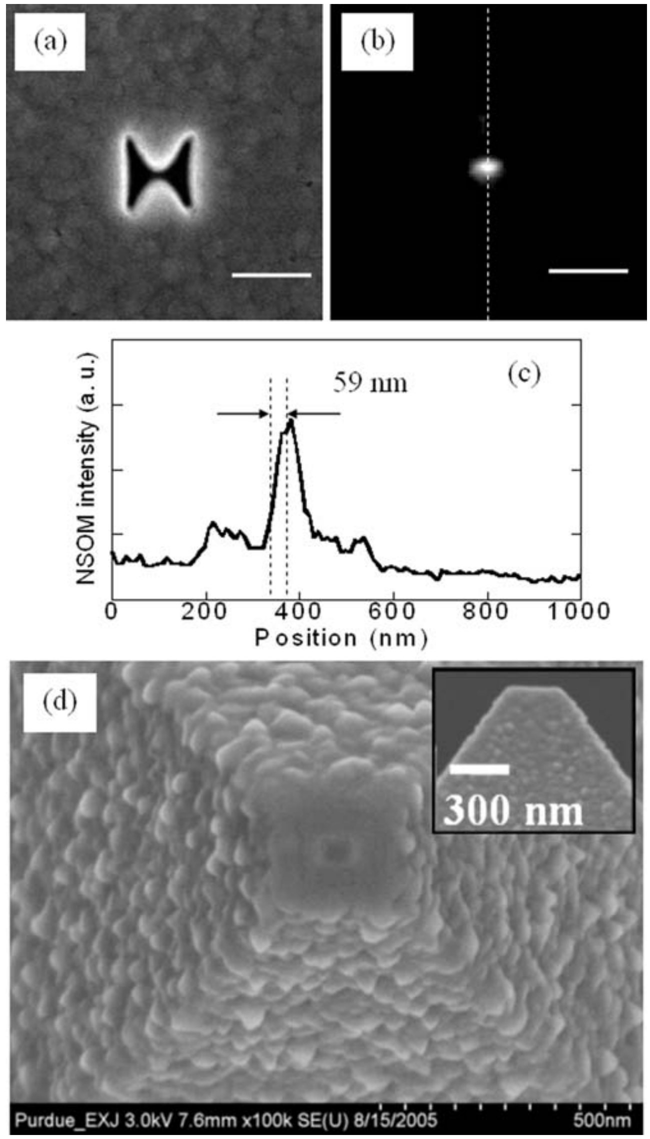

Fig. 9. The near-field ptical image (b) is collected from a bowtie nanoaperture in an aluminum test sample shown in (a) using the FIB-micromahined aperture probe as shown in (d). The inset of (d) is the side SEM image of the same tip. The 10-90% edge resolution of this probe is 59 nm as shown in the optical profile (c) along the dash line in (b). The scale bars in (a) and (b) are 250 nm.

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