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Letter

Cleaved thin-film probes for scanning tunneling microscopy

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Abstract

We introduce an alternative type of probe for scanning tunneling microscopy (STM). Instead of using a needle-like tip made from a piece of metallic wire, a sharp-edged cleaved insulating substrate, which is initially covered by a thin conductive film, is used. The sharp tip is formed at the intersection of the two cleaved sides. Using this approach a variety of materials for STM probes can be used, and functionalization of STM probes is possible. The working principle of different probes made of metallic (Pt, Co, and CoB), indium-tin oxide, as well as Cu/Pt and Co/Pt multilayer films are demonstrated by STM imaging of clean Cu(001) and Cu(111) surfaces as well as the epitaxial Co clusters on Cu(111).

Keywords: scanning tuneling microscopy, stm probe, probes fabrication, probes functionalization

(Some figures may appear in colour only in the online journal)

1. Introduction

Scanning tunneling microscopy (STM) has become the standard technique to study surfaces. The main element of an STM setup is a sharp conductive probe, which is generally a needle-like tip made of electrochemically etched W wire or a mechanically cut PtIr wire [1–5]. For specific studies, e.g., STM experiments that require the use of superconducting tips, or spin-polarized STM studies, different materials are used for probe fabrication [6–10]. Those probes can be obtained by similar etching of wire from appropriate materials [6, 9–19]. Alternatively, the deposition on the conventional needle-like W tip is used to create a sharp tip coated by the desired material [7, 19, 20]. Although simple, these approaches have some drawbacks. Many materials that would be useful for STM probes cannot be obtained as wire or cannot be etched to form the desirable tip shape. When a material is deposited on the needle-like tip, the properties of the deposited layer at the very end of the tip are, in many cases, uncertain. Therefore, probes with a needle-like shape are not always optimal.

In this letter we introduce an alternative type of STM probe. Instead of using a bulk conductive material, we use a thin conductive film that covers the surface of a cleavable flat substrate as the probe. To obtain the STM tip, the coated substrate is cleaved with a simple procedure, which is illustrated in figure 1. The implementation of flat thin film as the probe provides several advantages. The film in principle can be made from any conductive material or even a multilayer structure. Thus, a vast variety of materials for the STM probes can be used and the structure of a multilayer can be engineered to benefit from its specific properties. Moreover, micro-/nanostructures on the coating film can be created, which enables functionalization of the probes. In addition to these advantages, this type of probe is simple to create. The preparation of the film can be done *in* or *ex situ* of a conventional vacuum setup.

This letter is organized as follows. The next section describes preparation of the probes and samples. In section 3, the working principle of the cleaved thin-film probes is presented. Some possible applications of this kind of probe in experiments are discussed in section 4. Section 5 summarizes this letter.

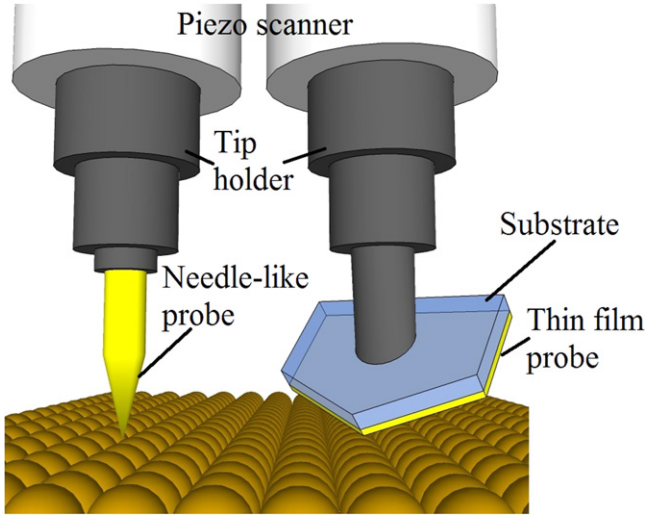

Figure 1. Comparison of the STM operation using a conventional needle-like probe (left) and a probe from a thin film (right). The yellow color indicates the conductive part of the probes. The semi transparent blue color on the right corresponds to the substrate of the cleaved tip.

2. Experiment

STM probes made of thin film of several materials were fabricated in this work. The fabrication process includes two main steps: the coating of the cleavable substrate by conductive film and the cleaving of it. The coating of the conductive film, in principle, can be done using any standard deposition technique on any suitable planar substrate. In this particular work, we prepared metallic (Pt, Co, and CoB) and inidium-tin oxide (ITO) films on glass. ITO was chosen to explore the use of transparent tips in STM. This material is widely used as transparent electrode in display technology, light-emitting diodes, and many optoelectronic devices. The metallic film was created by sputter-depositing 50–100 nm thick Pt, Co, or CoB on thin (120 microns) glass plates. The thickness of the metallic film was optimized for reliable STM operation. Before the deposition, a 2 nm thick seed layer of Ta was deposited to provide smooth growth and good adhesion. For the ITO film, the commercially available ITO on glass was used.

After the coating, the coated substrates were cleaved using a simple procedure which adapts the technique of preparing STM probes from semiconductor slabs [21, 22]. First, a cleaved side was formed, which was done by making a small initial scratch on the substrate (figure 2(a)), followed by applying a force on it (figure 2(b)). Second, another cleaved side was formed in a similar way by another small initial scratch at the opposite edge of the substrate (figure 2(c)). A sharp tip forms at the intersection of the two cleaved sides outside the initial scratches. Lastly, the remaining part of the plate was cut to shorten the STM tip to make it fit the tip holder (figure 2(d)). The holder provides an approximately 45° angle between the probe and the sample surface (figure 2(e)). This angle was optimized for STM operation. The resulting probes were inspected using

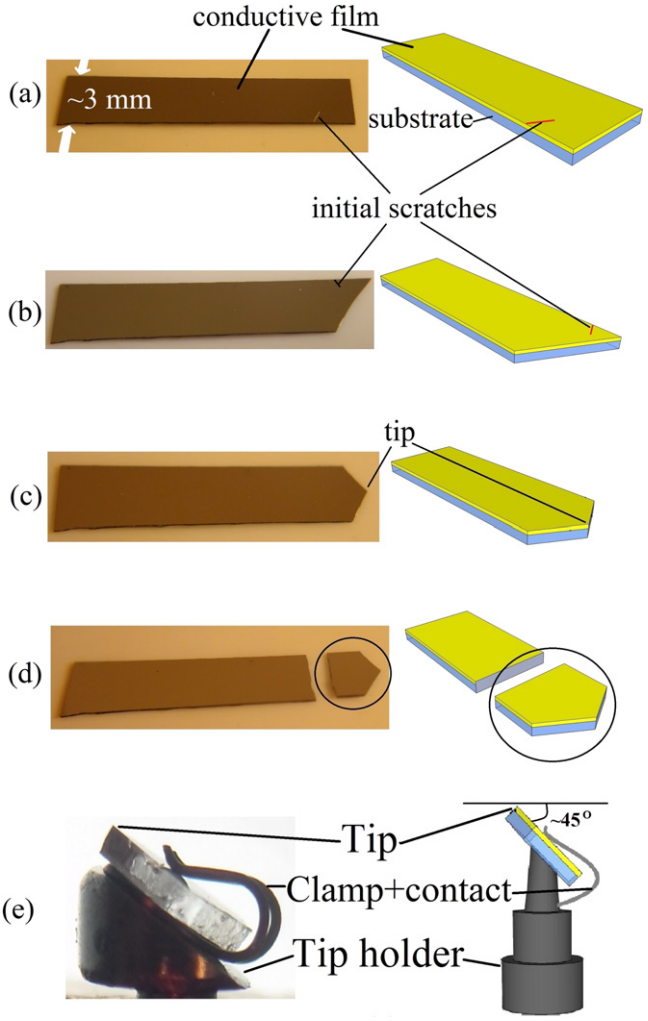

Figure 2. Images (left) and schematics (right) for each fabrication step. (a) A substrate (glass) coated with a conductive (Pt) film. The first initial scratch is made on it. (b) The first cleaved side is formed. The second initial scratch is introduced. (c) The second cleaved side is formed. The tip is formed at the intersection of the two cleaved sides. (d) The remaining part of the substrate is removed. The part in the circle is the resulting STM tip. (e) The tip is mounted on the holder with an inclination angle of approximately 45° relative to the sample surface.

scanning electron microscopy (SEM) (figure 3). Typical side and top views of the tips are shown in figure 3(a) and (b), respectively. The local curvature of the tips is typically less than 10 nm.

Since STM imaging was performed in an ultrahigh vacuum (UHV) system (Omicron) [23], additional tip cleaning and processing in vacuum were required. The tips were degassed by heating in vacuum. Low-dose sputtering was also done to remove several atomic layers that contain native oxides.

In this work, we also demonstrate STM scans using probes from metallic multilayer films. Probes from Cu/Pt and Co/Pt multilayer films were chosen. To form such probes, we added a few extra atomic layers of Cu or Co on the cleaved probes from Pt film. The deposition was done *in situ* using the e-beam evaporation technique from calibrated Cu (~1 ML/min) and Co (~0.2 ML/min) sources. In addition to forming

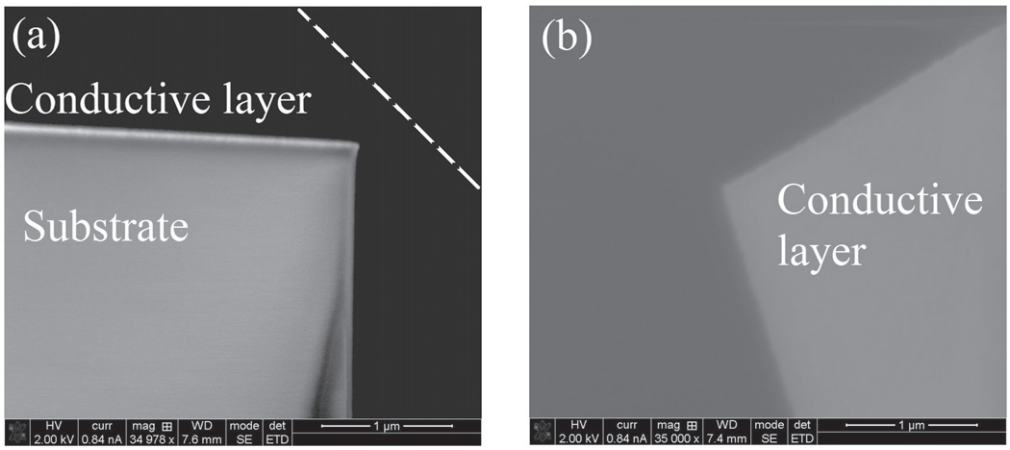

Figure 3. SEM images of the tip of a Pt film on glass. (a) The side view. The dashed line indicates the position of the sample surface relative to the probe tip. (b) Top view.

the multilayer films, this extra deposition also allows us to reuse the tips that have been damaged after accidental tip crash or over-sputtering. Generally such damaged tips cannot be used anymore because of the loss of conductive material at the very end of the tip. But adding a few extra atomic layers of conductive material as mentioned above actually repairs the tips.

To prove the working principle of cleaved thin-film probes, the fabricated probes were used to scan surfaces and structures with well-known features. We chose clean Cu(001) and Cu(111) surfaces as well as Co nanostructures on Cu (001) as samples for STM imaging. Prior to any measurements and treatments, the single crystalline Cu(001) and Cu (111) samples were cleaned using a sputter-anneal procedure. Submonolayer Co was deposited *in situ* on the Cu(111) at room temperature. The typical result of this growth is reported elsewhere [24–26]. The measurements were carried out at liquid nitrogen temperature with a base pressure $<10^{-10}$ mbar. The imaging was performed at constant-current mode.

3. Results and discussion

To prove the reliability of the probes for STM measurements, we address their stability and sharpness, which can be determined via careful analysis of the resulting STM images. Several probes from each film were used in the STM operations. Each probe was used to scan more than three areas, which were separated by several millimeters. A stable tunneling current was always achieved. STM imaging was always performed for more than 4 hours, and the quality of the resulting images did not change during the measurement time.

The probes from the metallic film were used to scan the clean Cu(001) surface. Typical results are shown in figure 4, which shows several atomically flat terraces ~100 nm wide with sharp step edges. The height difference between the two adjacent terraces is ~0.18 nm, corresponding to the interlayer spacing in the Cu(001). The resulting STM images do not show any sudden change in height on the flat terraces. Such sudden changes in height may be attributed to the change in probe tip. Thus, the absence of sudden changes of height in the images leads to a conclusion that the probes are stable.

The sharpness of the probes at the mesoscopic and atomic scales were also inspected. At the mesoscopic scale we analyze the atomic step edges or the edges at the nanostructure on the surface. Since the STM images show very sharp step edges, we conclude that the tips are mesoscopically sharp. Measurement results obtained using a blunt tip would have shown a gradual change in the height across the steps. The sharpness at atomic scale is confirmed by the atomically resolved features. Figure 4(b) shows an STM image with the atomic resolution, which shows a square pattern with small depressions of the atoms at the corners. At the center of the square, an atom with slight apparent protrusion is seen, which could be due to the electronic effect induced by an impurity atom embedded in or below the surface [27].

It is important to note that the results presented in figure 4 are also typical for measurements using Cu/Pt and Co/Pt multilayer probes (not explicitly shown here). This confirms that the cleaved thin-film probes are suitable for artificial structures embedded in them. Further discussion of functionalizing multilayer structure, and the micro- or nano- structure in general, in STM experiments is given later in this paper.

The probes from ITO were used to scan the Cu(111) surface. Figure 5 shows the typical measurement results. Figure 5(a) shows an atomically resolved image of the clean Cu(111) surface, confirming that the probes are atomically sharp. Figure 5(b) shows the morphology of the surface after the Co deposition. Co islands of bilayer height with triangular and star-like shapes on a flat terrace are seen, which is in good agreement with previous reports of Co growth on Cu(111) [24–26]. The islands are bounded by a rim area of around 1.5 nm wide. This rim shows increased apparent height, similar to that reported in [26]. The appearance of this rim in the STM images is attributed to the electronic states close to the edge of the nanoislands, which are different from in the interior. By doing a careful analysis of the image, as done above, the stability and sharpness of the probes are confirmed.

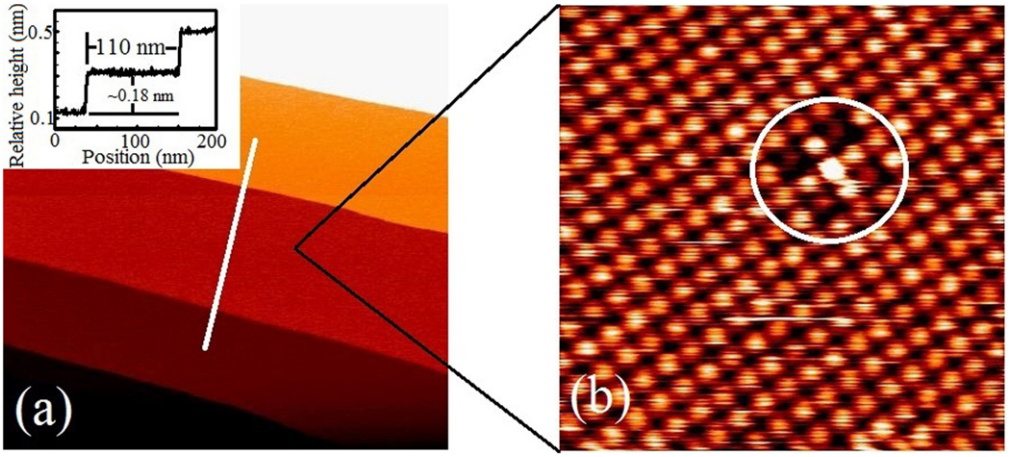

Figure 4. Morphology of a Cu(001) surface obtained by STM imaging using a Pt probe. (a) The large-scale image, 400 × 400 nm^2 ($V = 0.4$ V, $I = 1$ nA). Inset: The surface profile along the cross-section shown in (a). (b) An atomically resolved image of the surface on a flat terrace, 4 × 4 nm^2 ($V = 0.013$ V, $I = 62$ nA). The white circle shows a square pattern on the surface.

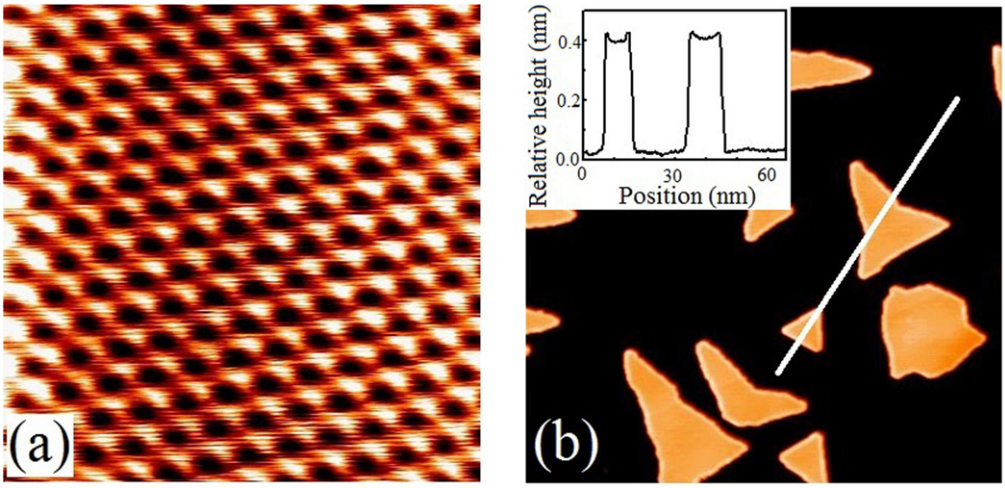

Figure 5. (a) STM image, 3 × 3 nm^2, of a clean Cu(111) surface ($V = 0.02$ V, $I = 1.5$ nA). (b) The topographic map, 100 × 100 nm^2, of the Cu(111) surface after room temperature deposition of 0.6 ML Co ($V = -1$ V, $I = 6$ pA). Inset: The surface profile along the cross-section shown in (b). The images are obtained with ITO-film probes.

To the best of our knowledge ITO has never been used as a probe material. STM probes from ITO may be attractive for some studies, since its transparency can be used to enhance the light-collection efficiency in scanning tunneling luminescence (STL) experiments [28]. The point contact experiment between ITO and molecular materials can also be realized using an STM setup. For operations in air, in particular, probes from ITO film, are easier to prepare than those made of metals since ITO resists oxidation. However, it should be taken into account that the electronic band structure of ITO has a wide energy gap around the Fermi level [29, 30], which could hamper a convenient measurement at a low bias voltage.

4. Outlook

As discussed, our approach opens up possibilities to use various materials for the STM probes as well as to functionalize micro- and nano- structures in STM experiments. Our approach also provides flexibility in choosing the substrate.

Functionalizaton of the probes by forming micro-/nano-structure on the coating film can be realized. Engineering the structure on the probe provides specific properties of the probes. For example, probes with a certain magnetic anisotropy or stable magnetization can be useful in spin-polarized STM. For this, films of multilayer structure that exhibit perpendicular magnetic anisotropy (PMA), artificial antiferromagnetic behavior [31], or exchange bias [32] become very attractive. The probes from a cleaved Co/Pt film which is used in this study, can provide PMA for the spin-resolved STM [33]. More complicated structures can be created using lithography techniques. For example, in STM experiments that require detection of emitted light from the sample or the tip, probes with integrated photonic structures can be beneficial. Figure 6(a) illustrates a probe with a photonic crystal waveguide and a photon detector incorporated in the covering

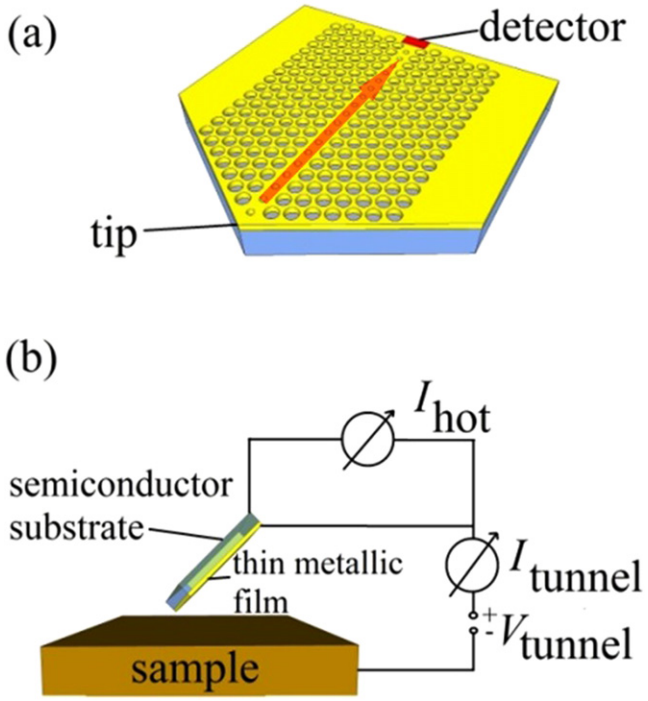

Figure 6. (a) Illustration of a cleaved thin-film probe with a photonic crystal waveguide and a photon detector incorporated in the layer. The red arrow shows the propagation direction of the light along the waveguide. (b) Schematic of the BEEM-like configuration. Part of the tunneling electrons (detected as I_{tunnel}) with high enough energy can penetrate the substrate and be detected as I_{hot}.

film [34–36]. These structures can be made on a film from conductive material with a high refractive index like Si or InP. With such a probe, the emitted light can be confined and guided to propagate toward the detector, which should result in efficient light detection.

Apart from engineering the covering film, we can also take advantage of using various substrates, which can be made from any cleavable slab. Slabs from single crystalline dielectrics (e.g., sapphire or magnesium oxide) or semiconductors (e.g., Si or GaAs) suit our approach. Easy cleaving along certain crystallographic directions of some single crystals can be used to obtain a tip with a well-defined angle. The electronic properties of the substrate can also be exploited. For example, by implementing separate contacts to the conducting film and a semiconducting substrate we can separate the currents of electrons with different energies (see figure 6(b)). This is similar to ballistic-electron emission microscopy (BEEM) [37], but with the metal-semiconductor structure on the tip instead of on the sample. This probe can be used to study systems with scattering-assisted tunneling.

5. Summary

In summary, we introduced an alternative type of STM probe, which uses a thin conductive film as the tunneling probe. This approach enables the implementation of various materials in STM probes as well as their functionalization by micro- and nano-structuring. To fabricate the probe, a simple procedure was proposed.

The proof of working principle of probes made of Pt, Co, CoB, and ITO films as well as from multilayer films of Co/Pt and Cu/Pt were presented. Careful analysis of the resulting STM images shows that such probes are reliable in STM operations. Several possible applications of this type of STM probes were also discussed.

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References

[1] Ekvall I, Wahlstrom E, Claesson D, Olin H and Olsson E 1999 *Meas. Sci. Technol.* **10** 11–8
[2] Oliva A I, Romero G A, Peña J L, Anguiano E and Aguilar M 1996 *Rev. Sci. Instrum.* **67** 1917–21
[3] Hacker B, Hillebrand A, Hartmann T and Guckenberger R 1992 *Ultramicroscopy* **42-44** 1514–8
[4] Yu Z Q, Wang C M, Du Y, Thevuthasan S and Lyubinetsky I 2008 *Ultramicroscopy* **108** 873–7
[5] Marchenko A and Cousty J 2002 *Surf. Sci.* **513** 233–7
[6] Bassi A L *et al* 2007 *App. Phys. Lett.* **91** 173120
[7] Bode M, Getzlaff M and Wiesendanger R 1998 *Phys. Rev. Lett.* **81** 4256–9
[8] Bode M 2003 *Rep. Prog. Phys.* **66** 523–82
[9] Kohen A, Noat Y, Proslier T, Lacaze E, Aprili M, Sacks W and Roditchev D 2005 *Physica* C **149** 18–24
[10] Kohen A, Proslier T, Cren T, Noat Y, Sacks W, Berger H and Roditchev D 2006 *Phys. Rev. Lett.* **97** 027001
[11] Wang X, Liu Z, Zhuang M-D, Zhang H M, Wang X, Xie Z X, Wu D Y, Ren B and Tian Z Q 2007 *App. Phys. Lett.* **91** 101105
[12] Watanabe M O and Kinno T 1994 *App. Surf. Sci.* **76/77** 353–8
[13] Shimizu R, Hitosugi T, Hashizume T, Fukuo N and Hasegawa T 2010 *Japan. J. Appl. Phys.* **49** 028004
[14] Hofmann T, Welker J and Giessibl F J 2010 *J. Vac Sci. Technol.* B **28** C4E28–30
[15] Nam A J, Teren A, Lusby T A and Melmed A J 1995 *J. Vac Sci. Technol.* B **13** 1556–9
[16] Baykul M C 2000 *Mat. Sci. Eng.* B **74** 229–33
[17] Albonetti C, Bergenti I, Cavallini M, Dediu V, Massi M, Moulin J F and Biscarini F 2002 *Rev. Sci. Instrum.* **73** 4254–6
[18] Cavallini M and Biscarini F 2000 *Rev. Sci. Instrum.* **71** 4457–60
[19] Murata Y, Kishida M, Motoyoshi K, Kimura T, Honda S, Okamoto K, Matsui Y, Tagawa S and Katayama M 2007 *Japan. J. Appl. Phys.* **46** 8005–7
[20] Kelly K F, Sarkar D, Oldenburg S J, Hale G D and Halas N J 1997 *Synt. Met.* **86** 2407–10
[21] Sutter P, Zahl P, Sutter E and Bernard J E 2003 *Phys. Rev. Lett.* **90** 166101
[22] Sutter P, Palmer J, Zahl P and Sutter E 2003 *Surf. Sci.* **5325** 1166–70

[23] Kurnosikov O, Adam O A O, Swagten H J M, de Jonge W J M and Koopmans B 2008 *Phys. Rev.* B **77** 125429
[24] Pedersen M O, Bonicke I A, Laegsgaard E, Stensgaard I, Ruban A, Norskov J K and Besenbacher F 1997 *Surf. Sci.* **387** 86–101
[25] de la Figuera J, Prieto J E, Ocal C and Miranda R 1993 *Phys. Rev.* B **47** 13043–6
[26] Pietzsch O, Okatov S, Kubetzka A, Bode M, Lichtenstein A and Wiesendanger R 2006 *Phys. Rev. Lett.* **96** 237203
[27] Weismann A, Wenderoth M, Lounis S, Zahn P, Quaas N, Ulbrich R G, Dederichs P H and Blugel S 2009 *Science* **323** 1190–3
[28] Keizer J G, Garleff J K and Koenraad P M 2009 *Rev. Sci. Instrum.* **80** 123704
[29] Matino F, Persano L, Arima V, Pisignano D, Blyth R I R, Cingolani R and Rinaldi R 2005 *Phys. Rev.* B **72** 085437
[30] Rosen J and Warschkow O 2009 *Phys. Rev.* B **80** 115215
[31] Parkin S S P, Bhadra R and Roche K P 1991 *Phys. Rev. Lett.* **66** 2152–5
[32] Nogues J and Schuller I K 1999 *J. Magn. Magn. Mater.* **192** 203–32
[33] den Broeder F J A, Hoving W and Bloemen P J H 1991 *J. Magn. Magn. Mater.* **93** 562–70
[34] Joannopoulos J D, Johnson S G, Winn J N and Meade R D 2008 *Photonic Crystals Molding the Flow of Light* (Princeton, NJ: Princeton University Press) 135–264
[35] Pernice W H P, Schuck C, Minaeva O, Li M, Goltsman G N, Sergienko A V and Tang H X 2012 *Nat. Commun.* **3** 1325
[36] Najafi F *et al* 2015 *Nat. Commun.* **6** 5873
[37] Prietsch M 1995 *Phys. Rep.* **253** 163–233