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## Magnetic Response and Spin Polarization of Bulk Cr Tips for In-Field Spin-Polarized Scanning Tunneling Microscopy

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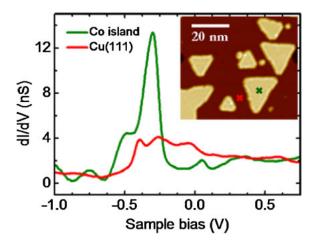
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The magnetic properties of bulk Cr tips have been investigated by spin-polarized scanning tunneling spectroscopy (SP-STS). To extract the properties of the Cr tips, we performed low-temperature SP-STS measurements on a well-known model system: nanometric Co islands on Cu(111). Our experiments indicate the existence of uncompensated magnetic moments at the apex of the Cr tips, which rotate in the direction of the applied vertical magnetic field and become aligned with it at approximately 2 T. We extracted a tip spin polarization of 45% at the Fermi energy. We showed that the tip spin polarization can change with a modification of the tip apex. © 2012 The Japan Society of Applied Physics

ips made of bulk antiferromagnetic materials such as chromium<sup>1-3)</sup> have been proposed for spin-polarized scanning tunneling microscopy (SP-STM<sup>4-6)</sup>) and have recently been shown to provide magnetic contrast on Cr(001) and Fe/W(110).<sup>7,8)</sup> As compared with W tips covered with an ultrathin film of antiferromagnetic material,<sup>6,9,10)</sup> they present the advantage of a simpler preparation which does not require a dedicated tip preparation stage. Bulk Cr tips do not require high-temperature flashing or material deposition. Here we present a characterization of the Cr tip spin polarization and the response of the tip apex magnetization orientation to an external magnetic field. These properties are essential for the reliable use of such tips in SP-STM as we show in this paper.

We performed spin-polarized scanning tunneling spectroscopy (SP-STS) measurements on cobalt islands on Cu(111), a system for which the electronic and magnetic properties have been thoroughly characterized. 11-14) The Co islands have an out-of-plane easy magnetization axis. The energy-dependent spin polarization of Co islands has been measured and accounted for by ab initio calculations. 14) Using bulk Cr tips we found no magnetic contrast on asdeposited Co islands at 0T. This suggests that uncompensated magnetic moments at the Cr tip apex are oriented inplane at zero field. The magnetic moments at the Cr tip apex can be oriented along the vertical direction by an applied magnetic field of approximately 2T. Here, we quantify the spin polarization of the Cr tip and its energy dependence. We also show that the spin polarization of a specific Cr tip is not a general property, as it can change significantly with a modification of the tip apex.

The tips were produced from polycrystalline Cr rods with a nearly square cross section of  $0.7 \times 0.7 \,\mathrm{mm^2}$  obtained by cutting a 99.99% Cr foil (from Super Conductor Materials). The rod was electrochemically etched in  $1.5 \,\mathrm{M}$  NaOH solution following the procedure reported in ref. 7 and then directly inserted in the chamber used for STM measurements. The only *in-situ* preparation of the tip consisted of the application of voltage pulses between tip and sample during STM experiments. We applied voltage pulses only on the clean Cu surface to ensure the absence of any potential Co contamination on the tip apex. We also avoided deliberate indentation of the tip into the Co islands.



**Fig. 1.** (Color online) dI/dV(V) spectra measured with a Cr bulk tip at the center of the marked Co island (green) and on the Cu(111) surface (red). Stabilization parameters: 1 nA and +0.5 V. Inset: constant-current image ( $V_{\rm gap} = 0.1 \, {\rm V}, \, I_{\rm t} = 1 \, {\rm nA}$ ) of two-atomic-layer-high (0.4 nm) Co nanoislands on Cu(111). The green and red marks indicate the positions where the two differential conductance dI/dV(V) spectra were taken.

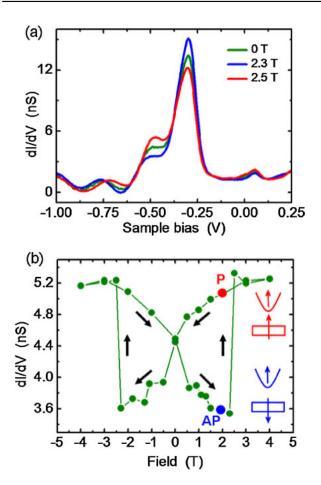
The results presented in this work were obtained at a temperature of 8 K and in external magnetic fields of up to 4T oriented along the sample normal, along the tip axis. The preparation of the sample has been described elsewhere. <sup>[10]</sup> It leads to the formation of double-layer-high Co islands with a base length from approximately 5 nm up to 30 nm. <sup>[5]</sup> STS measurements were performed by a lock-in technique (10 mV, 5 kHz) to obtain the differential conductance dI/dV(V) (I: tunnel current, V: sample voltage). The dI/dV(V) signal is related to the local electronic and magnetic properties of sample and tip. <sup>[6]</sup>

Figure 1 shows dI/dV spectra taken on the Cu surface and on a Co island. The positions where the two spectra were taken are indicated in the inset of Fig. 1. We observed a step at  $-0.44 \,\mathrm{V}$  in the dI/dV spectra taken on Cu(111) and a pronounced peak at  $-0.3 \,\mathrm{V}$  in the spectra obtained on the Co island. Both these features have been reported in previous studies and correspond to the onset of the Cu surface state<sup>17)</sup> and the occupied  $d_{z^2}$ -like Co surface state respectively.<sup>12)</sup> Also the smaller features at -0.5 and  $+0.3 \,\mathrm{V}$  in the Co

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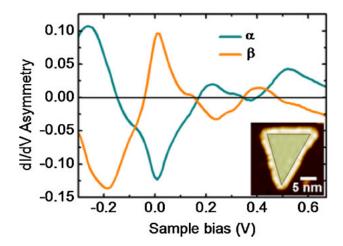
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**Fig. 2.** (Color online) (a) dI/dV(V) spectra measured at the center of the Co island marked in Fig. 1 at different magnetic fields. (b) dI/dV hysteresis loop of the Co island in Fig. 1 ( $V_{\rm gap}=-0.5\,{\rm V}$ ). Arrows indicate the sequence in which the data were measured. Red and blue points respectively indicate parallel (P) and anti-parallel (AP) states between the magnetization direction of tip and sample at  $+1.8\,{\rm T}$  where the dI/dV asymmetry shown in Fig. 3 was measured.

island spectra have been interpreted as sample-related peaks.  $^{11-13)}$  The spectroscopy measurements on Co and Cu using a bulk Cr tip revealed the intrinsic electronic properties of Co and Cu. The spectra are very similar to those obtained with W and Cr-covered W tips. Therefore, we conclude that the used Cr tip has a rather smooth local density of states (LDOS) in this energy range, as we did not observe any tip-related specific features in the dI/dV data.

Figure 2(a) shows dI/dV spectra at different applied magnetic fields measured at the center of the Co island marked in Fig. 1. Clear differences in the shape and amplitude of the spectra were found, although the main features are similar for all voltages. Increasing the applied magnetic field from 0 to 2.3 T, the dI/dV signal at -0.5 V decreases in intensity and then sharply increases at an applied field of 2.5 T. The field-induced change in the dI/dV signal of the main peak located at -0.3 V is opposite in sign to that of the shoulder at -0.5 V. In order to link the magnetic field dependence of the differential conductance to the magnetic properties of the tunneling system (tip + sample), we recorded dI/dV(V) spectra at different field values during a complete cycle of the applied magnetic field. Extracting the magnitude of the dI/dV signal at a fixed



**Fig. 3.** (Color online) Energy dependence of the dI/dV asymmetry averaged over the highlighted inner part of the Co island shown in the inset [the Co island marked in Fig. 1(a)]. The two curves refer to two different measurements performed by the same macroscopic bulk Cr tip but with different tip apexes. The procedure of the extraction of the dI/dV asymmetry is described in the supporting online material of ref. 14.

bias as a function of the field yielded the hysteresis loop shown in Fig. 2(b). The dI/dV signal changes gradually with increasing field and at a critical applied magnetic field of 2.4 T, it changes abruptly. A butterfly hysteresis cycle results, which has been observed in previous studies using Cr-coated W tips.  $^{10,18)}$  The abrupt change in the dI/dVsignal is ascribed to the reversal of the magnetization direction of the Co island. The gradual change is ascribed to the rotation of the spin orientation of the tip apex in response to the applied magnetic field, as discussed in ref. 10. At first sight the reorientation of the spin moment of a bulk antiferromagnetic material in a modest field of 2T might come as a surprise. However, since clusters at the end of the tip need to be considered in the tunneling process, <sup>19)</sup> the presence of uncompensated spin moments at the tip apex is a reasonable assumption. Indeed, uncompensated magnetic moments in Cr clusters have already been observed experimentally<sup>20)</sup> and treated theoretically.<sup>21)</sup>

To determine the spin polarization of the tip  $(P_t)$ , we measured the energy dependence of the dI/dV asymmetry A on a single Co island for parallel (P) and anti-parallel (AP) orientations between tip and sample magnetic moments. The result is shown in Fig. 3 (curve  $\alpha$ ). Each point in Fig. 3 was obtained by the following procedure: (1) We sweep the magnetic field from 0 T up to +1.8 T (anti-parallel state AP, blue point in the hysteresis loop in Fig. 2). (2) We record a dI/dV spectra for each point of the image in Fig. 3. (3) We sweep the magnetic field up to +4 T in order to switch the magnetization direction of the sample. (4) We sweep the magnetic field down to  $+1.8 \,\mathrm{T}$  (parallel state P, red point in the hysteresis loop in Fig. 2) and we repeat step (2). (5) At this point, we have a dI/dV map for parallel and anti-parallel state at each bias voltage in the set range. From the measured dI/dV maps we calculate a dI/dV asymmetry map for each measured sample bias. We define the dI/dV asymmetry as  $A = (dI/dV_{AP} - dI/dV_P)/(dI/dV_{AP} + dI/dV_P)$ . This value is related to  $-P_tP_s$ , where  $P_s$  is the spin polarization of the sample. 6,14,16) (6) To extract the energy dependence of the dI/dV asymmetry A, we average each dI/dV asymmetry map over the inner part of the Co island in order to avoid the contribution of the rim state and to average out the modulation of the Co spin polarization. The procedure is also described in the supporting online material of ref. 14.

The asymmetry varies in sign and magnitude with the applied voltage and reaches a maximum value of -0.123 at the Fermi energy. In order to quantitatively extract the spin polarization of the tip, the effect of the tip–sample distance on the extraction of the spin polarization must be taken in account.<sup>22,23)</sup> We use the calculated energy dependence of the spin polarization of a 2-ML-high Co film  $(P_s)$  at the tip position from ref. 14. From curve  $\alpha$  in Fig. 3 we obtain  $P_t = 0.173$  and 0.450 at V = -0.26 and 0 eV, respectively. The calculated spin polarization at the Fermi energy for this tip has the opposite sign and is significantly larger than the spin polarization of the Cr(001) surface (P = -0.18).<sup>24)</sup> This value obtained at the Fermi energy is comparable to the spin polarization obtained for Fe-coated W tips (P = 0.42).<sup>6.25)</sup>

The characterization of the tip spin polarization is important for obtaining a correct interpretation of the experimental data since it can change from tip apex to tip apex, as we discuss in the following. Curve  $\beta$  in Fig. 3 shows the measured dI/dV asymmetry averaged over the island after the tip apex changed its configuration during an STS measurement. We observed that the overall shape and amplitude of the two curves are very similar, but the sign of the dI/dVasymmetry is different. The change in the tip apex could only be noticed from the slightly different shape of the dI/dV spectra (not shown), whereas the constant-current image did not show any notewothy difference after the tip had changed. Moreover, the response of the spin moment of the tip apex to the applied magnetic field did not change. We ascribe the change in the tip apex to structural modification of the tunneling apex. This measurement clearly shows that even a small change in the tip apex can modify the value of the tip spin polarization as well as reverse its sign. This is in line with recent theoretical calculations which reveal that the spin polarization of a Cr atom can strongly change depending on the orientation of the surface on which it lies.<sup>26)</sup>

In conclusion, we performed a magnetic characterization of a bulk Cr tip. The significant spin polarization, the possibility of tuning the spin orientation of the tip apex using a magnetic field, and the easy fabrication process make bulk Cr tips excellent candidates for future antiferromagnetic SP-STM/STS probes.

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