Spin-dependent electronic structure of self-organized Co nanomagnets

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Abstract. The magnetism of small (only a few nanometers in diameter) Co nanoparticles (NPs) grown on Au(111) was investigated by means of spin-dependent scanning tunneling microscopy and spectroscopy in a broad energy range. Direct evidence is provided for the existence of localized d-states of minority and majority character that govern the spin polarization of the NPs below the Fermi level. On the other hand, the discrete electronic states resulting from the spatially confined sp-like Co surface state electrons above the Fermi level are found to be of majority character. This confirms the theoretically predicted spin-polarized character of the delocalized surface state electrons of Co NPs on Au(111).

Research on nanomagnetism is of direct relevance to nanoelectronics, e.g. magnetic data storage [1] and spintronics [2, 3], as well as to biomedical applications, e.g. hyperthermia [4] and immunoassay [5]. Whereas bulk materials can be adequately investigated by standard magnetometry and magnetic imaging techniques, the study of the local spin-dependent properties of their nanosized counterpart requires the use of local probes.

With the invention and application of spin-polarized scanning tunneling microscopy (STM) and spectroscopy (STS) in the early 1990s [6]–[8], a novel dimension was added to the range of research possibilities that were already available with spin-averaged STM and STS. The

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spin-polarized technique provides a unique means of exploring nanomagnetism with atomic resolution [9]: the local spin-resolved electronic structure of crystal surfaces [7, 10], thin films [11]–[13] and, more recently, individual nanoparticles (NPs) [14]–[17] has already been revealed.

Both the spin-averaged [18] and the spin-resolved electronic structures [14, 15, 17] of Co NPs on Cu(111) have been investigated in great detail. The particles are grown by atom deposition, thus forming bilayer triangularly shaped NPs that are randomly distributed across the Cu(111) surface and that have a rather large size distribution (the diameter of the NPs is typically in the range of 10–30 nm). Co NPs on Cu(111) are found to exhibit a net out-of-plane magnetization. Below the Fermi level, the NP spin polarization $P(E) = (n_\uparrow(E) - n_\downarrow(E))/(n_\uparrow(E) + n_\downarrow(E))$, where $n_\uparrow(E)$ and $n_\downarrow(E)$ denote the spin-up (majority) and spin-down (minority) density of states (DOS) of the NP, is governed by localized d-states of minority character [14, 18]. Delocalized sp-states of majority character dominate the spin polarization of the Co NP above the Fermi level [15]. The latter free-electron-like surface states give rise to the occurrence of standing wave patterns at the surface of the Co NPs [15, 18]. Very recently, it was found that apart from the DOS, the degree of spin polarization is spatially modulated as well [17].

We relied before on the well-known herringbone reconstruction [19] of the Au(111) surface as a template to grow bilayer Co NPs that have a narrower size distribution (the NP diameter can be tuned up to about 6 nm) and that are equidistantly arranged on the surface [20, 21]. Earlier, Rastei and Bucher [22] reported on the appearance of two types of Co NPs with different heights on Au(111) in constant current STM images recorded with magnetic Ni bulk STM tips, indicative of a net out-of-plane magnetization of the Co NPs at low temperatures. Recently, we investigated the spin-dependent local density of states (LDOS) of Co NPs on Au(111) in the occupied state regime in a limited energy range below the Fermi level by means of spin-polarized STM and STS using STM tips coated with a thin antiferromagnetic Cr layer, thereby confirming that Co NPs on Au(111) exhibit a net out-of-plane magnetization at low temperatures [23].

Here, we extend our previous work to a much broader energy range (about 5 eV) and observe multiple reversals of the spin polarization of the Co NPs below the Fermi level, providing direct evidence of the existence of localized d-states of minority and majority character. Moreover, we find that the electronic surface state residing on the nanosized Co NPs exhibits a spin-polarized character as well, confirming the recent theoretical prediction of Rastei et al [24]. The large energy separation of the quantized states of the nanosized Co particles on Au(111) [21] causes their spin-polarized nature to be only clearly discerned at energies well above the Fermi level.

Atomically flat Au(111) films are prepared as described in [21]. Co NPs are obtained by self-organization after in situ deposition of 0.3 monolayers (MLs) of Co atoms on clean Au(111) at a rate of 0.2 ML min$^{-1}$ at room temperature [20, 21]. STM and STS measurements are carried out with a low-temperature STM setup (Omicron Nanotechnology), operating at a base pressure in the range of 10$^{-11}$ mbar. All data are acquired at $T_{\text{sample}} \approx 4.5$ K. Electrochemically etched W tips are cleaned in situ by repeated flashing above 2000 K to remove the surface oxide layer and additional contamination. Spin-polarized STM tips with an out-of-plane sensitivity [8, 25] are obtained by coating the flushed W tips with about 40 MLs of antiferromagnetic Cr at a rate of 1.7 MLs min$^{-1}$, as described in [23]. Such tips with Cr coatings in the 25–45 ML range are known to have an effective negative spin polarization $P_t(E)$ around the Fermi level.
(the spin-polarized DOS of the tip is dominated by minority states near the Fermi level) [14, 26, 27].

Although the same procedure is followed for all Cr-coated STM tips, it is not known a priori whether the STM tip exhibits a spin polarization, nor whether its magnetization is oriented out of plane. Since it was not possible to apply an external magnetic field in our STM setup, we relied on the known out-of-plane magnetization of the Co on the Au(111) system for submonolayer Co coverages [28, 29] to check the spin sensitivity and magnetization direction of our Cr-coated STM tips [23]. In this way, we found that the tip preparation procedure followed yields spin-sensitive STM tips with a success rate of about 50%. STM tips with Cr coatings thicker than 45 MLs did not yield any additional LDOS contrast when compared with uncoated STM tips, which is to be expected since such tips are known to have in-plane magnetization [8]. During scanning of the sample surface, the spin sensitivity of the STM tip can be lost accidentally due to minor tip changes that occur during the scanning process. Occasionally, it appeared to be possible to restore the spin sensitivity of the STM tip by controllably inducing a minor tip change following the procedure described in [30]. This procedure relies on bringing the STM tip into contact with the sample surface for a very short time, after which often an atomic size protrusion (probably a Cr atom) is retrieved.

STM topographic imaging is performed in constant current mode. \((dI/dV)(V)\) curves are acquired with an open feedback loop by means of harmonic detection with a lock-in amplifier at a modulation frequency of 840 Hz and with a tunneling voltage modulation amplitude of 80 mV. Maps of the LDOS are acquired with a closed feedback loop using tunneling voltage modulation and lock-in amplification-based detection with similar parameter settings. In addition, local \(I(V)\) curves are recorded with an open feedback loop, from which \((dI/dV)(V)\) curves are obtained numerically. The tunneling voltages indicated in the text and figure captions are with respect to the sample, while the STM tip is virtually grounded. Image processing was performed by using Nanotec WSxM [31].

In figure 1(a), we present a typical large-scale topography image of the Au(111) surface after deposition of Co atoms: atomically flat NPs of bilayer height (1 ML of Co(0001) = 0.205 nm) and of only a few nanometers in diameter are formed by self-organization at step edges as well as on atomically flat terraces, at the so-called elbows of the herringbone reconstruction [20]. In figure 1(b), we present a LDOS map of the corresponding area acquired at \(V = -1000\) mV recorded with a spin-sensitive STM tip. Clearly, two types of Co NP can be discerned: one type has a higher LDOS when compared with the surrounding Au(111) surface, while the other type has a lower LDOS. Here, it must be noted that LDOS contrast of non-magnetic origin has previously been observed between Co NPs on Cu(111). It was shown by Pietzsch et al that two types of Co NPs exist on Cu(111), which are rotated by 180° about the surface normal and are referred to as faulted (exhibiting a stacking fault) and unfaulted (following the fcc stacking of the Cu(111) surface) NPs [14]. The two types of NPs reveal either a high or a low LDOS in specific energy ranges, which is found to be reversed in other energy ranges. These differences are related to the different stacking of the Co NPs on the Cu(111) surface and hence not to a different magnetization orientation. It was shown by Rastei et al that for the case of Co NPs on Au(111), the precise stacking of the NP on the Au(111) surface has an influence on the LDOS of the NP as well [24]. When using non-magnetic STM tips [21], we did not observe any difference in the LDOS of differently stacked (oriented) triangular Co NPs on Au(111) at the energies where pronounced contrast (for which LDOS maps are presented and discussed below) is revealed using Cr-coated STM tips. We can thus relate the LDOS contrast
observed here to the net magnetization of the NP, which is oriented perpendicular to the Au(111) surface and points either upward or downward \[23\]. There appears to be no correlation between particle size and magnetization direction for both Co NPs located at step edges and Co NPs located at elbows of the herringbone reconstruction, as illustrated in figure 1(b). The differential conductance \(\frac{dI}{dV}_{\text{SP}}(V)\), as measured with an STM tip having a spin polarization \(P_t(E)\), is sensitive to the spin polarization \(P_s(E)\) of the sample and can be written as

\[
\frac{dI}{dV}_{\text{SP}}(V) = \frac{dI}{dV}_{\text{SA}}(V)[1 + \text{SP}_{ij}(V)],
\]

where \(\frac{dI}{dV}_{\text{SA}}(V)\) denotes the spin-averaged differential conductance, \(\text{SP}_{ij}(V)\) is the spin polarization of the tunnel junction and \(\theta\) is the angle between the magnetization of the tip and the sample \[32\]. Note that equation (1) relies on the assumption of a constant LDOS of the STM tip in the investigated energy range and hence of an energy-independent spin polarization \(P_t(E) = P_t(E_F)\). It must, however, be kept in mind that at negative voltages \(V\), (possibly energy-dependent) empty tip states are involved in the tunneling process, of which the polarization and its sign are not known \textit{a priori} and may vary as a function of energy (also see below). On the other hand, at positive voltages, tunneling is dominated by electrons that tunnel from the tip’s Fermi level \(E_F\) (having a polarization \(P_t(E_F)\)) to empty sample states at \(E = E_F + eV\) (having a polarization \(P_s(E_F + eV)\)).

It can be seen from figure 1(b) that the Co NPs with the lower LDOS exhibit an increased LDOS at their edges, while the Co NPs with the higher LDOS exhibit a similar LDOS at their edges. For the case of Co NPs on Cu(111), it was shown previously by Pietzsch \textit{et al} that a spin-polarized state near the Fermi level exists at the edge of the NPs, having an opposite polarization.
Figure 2. (a) A $50 \times 50 \text{nm}^2$ combined topography image and LDOS map (see the text for more details) of Co nanomagnets on Au(111), illustrating either upward or downward oriented magnetization of the NPs. (b) 2D visualization of the $(dI/dV)_{SP}(V)$ spectra along the white dotted arrow in panel (a). Regions with the most pronounced spin-polarized contrast between the NPs in the empty and filled states regimes are marked by dotted and solid green rectangles, respectively. (c) Height profile taken along the white dotted arrow in panel (a). (d) Typical $(dI/dV)_{SP}(V)$ spectra of a type $\downarrow \downarrow$ NP and of a type $\uparrow \downarrow$ Co NP. The inset shows the corresponding derived SP$_{ij}$$(V)$ curve. The zero line is added as a guide to the eye.

when compared with the interior of the NPs [15]. Considering the similarities between Co NPs on Cu(111) and Au(111) surfaces, a magnetic rim state may be expected for Co NPs on Au(111) as well. However, topography-related height variations play an important role in STM and STS measurements at step edges, such as those of Co NPs [21, 33]. Within the resolution of our measurements, disentangling topography-induced features from magnetic features is not straightforward and requires detailed theoretical modeling, which is beyond the scope of the present work. In the following, we will therefore focus on the atomically flat interior of the Co NPs only.

The random direction (upward or downward) of the magnetization of the Co nanomagnets is further illustrated in figure 2(a). This three-dimensional (3D) visualization is obtained by superimposing a spin-resolved LDOS map (such as the one in figure 1(b)) acquired at $V = -1000 \text{mV}$ onto the corresponding topography image (such as the one in figure 1(a)). Red corresponds to a higher LDOS, while blue corresponds to a lower LDOS. We probed the spin-dependent electronic structure of the Co nanomagnets in detail by recording a grid of $200 \times 200$ $(dI/dV)_{SP}(V)$ spectra with a spin-sensitive STM tip across the area presented in figure 2(a), in a voltage range from $V = -1250 \text{mV}$ to $+1500 \text{mV}$. In figure 2(b), we present a color visualization of 175 thus obtained $(dI/dV)_{SP}(V)$ grid spectra taken along the white dotted arrow in figure 2(a). These spectra are obtained by means of harmonic detection with a lock-in amplifier. The electronic features in figure 2(b) can be linked to the NPs in (a) via the blue solid and red dotted height profiles in (c), which are taken as well along the white dotted arrow in (a). A typical $(dI/dV)_{SP}(V)$ spectrum of two Co NPs with opposite magnetization is presented in figure 2(d). Each of the latter spectra in (d) is obtained numerically from 16 averaged $I(V)$ taken in the center of the corresponding NPs.
It is clear from figures 2(b) and (d) that oppositely magnetized NPs exhibit pronounced differences in their LDOS around $V = -500$ mV and $V = -1000$ mV, consistent with [23]. It was predicted theoretically by Rast et al that Co NPs on Au(111) exhibit a spin-polarized state around $E = -500$ meV [24]. This state originates mainly from $d$ minority states, similar to the case of isolated Co atoms [34], Co NPs [18] and Co thin films [13] on Cu(111). The electronic structure of the STM tip is dominated by minority states around the Fermi level as well [14, 26]. It was reported, however, by Pietzsch et al that the spin polarization $P_t(E)$ of Cr-coated STM tips with an out-of-plane sensitivity [14] changes from negative to positive around $V = -380$ mV, implying that the electronic structure of the Cr-coated STM tip is dominated by majority states rather than minority states below this energy. This implies that tunneling between the STM tip and Co NPs around $V = -500$ mV will be favored for Co NPs of which the magnetization is aligned antiparallel to that of the STM tip. Co NPs that exhibit the higher LDOS around $V = -500$ mV are therefore referred to as type $\uparrow \downarrow$ NPs hereafter, while the Co NPs with the lower LDOS will be referred to as type $\downarrow \downarrow$ NPs. The Co NPs in figure 2(a) are labeled correspondingly.

From the spectra in figure 2(d), it is possible to infer, based on equation (1), a measure of the spin polarization of the tunnel junction $SP_{ij}(V)$ from the differential tunneling conductance asymmetry that is given by

$$\frac{dI/dV_{\downarrow \downarrow}(V) - dI/dV_{\uparrow \uparrow}(V)}{dI/dV_{\uparrow \uparrow}(V) + dI/dV_{\downarrow \downarrow}(V)},$$

(2)

where $dI/dV_{\downarrow \downarrow}(V)$ and $dI/dV_{\uparrow \uparrow}(V)$ denote the differential conductance recorded at a type $\downarrow \downarrow$ and $\uparrow \uparrow$ Co NP, respectively [17, 30]. The thus obtained $SP_{ij}(V)$ behavior is presented together with the two corresponding $(dI/dV)_{SP}(V)$ spectra in figure 2(d). The $SP_{ij}(V)$ curve reveals a clear dependence on the applied tunneling voltage $V$ and reaches a maximum value of about 15% near $V = -500$ mV.

It has been demonstrated by spin-resolved photoemission measurements that both Co(0001) crystals [35] and thin Co layers on Cu(111) [36] exhibit an additional spin-polarized state around $E = -1$ eV. This state was found to originate mainly from 3d minority states, with a small contribution from 3d majority states. Although the main contributions to the spin-polarized LDOS around both $V = -500$ mV and $V = -1000$ mV originate from minority states, the LDOS of the type $\uparrow \downarrow$ Co NP changes from higher (around $V = -500$ mV) to lower ($V = -1000$ mV) than that of a type $\downarrow \downarrow$ Co NP. We therefore attribute the observed reversed contrast of the LDOS of the type $\uparrow \downarrow$ and $\downarrow \downarrow$ Co NP to another change of the spin polarization $P_t(E)$ of the STM tip (from positive to negative). The spin polarization $SP_{ij}$ of the tunnel junction hence changes from negative to positive in this voltage region, as can be seen in figure 2(d).

When focusing on the positive voltage range in figure 2(b), lateral variations in the LDOS can be observed on the atomically flat area of the Co NPs for all NPs. These variations originate from the 2D free-electron-like surface state that is known to exist at the surface of Co NPs on Au(111) at energies above an onset energy about $-200$ meV [21]. Electrons occupying the Co surface state are able to move freely across the Co NP surface. Scattering of the electrons at the NP edges gives rise to the occurrence of standing wave patterns in the LDOS, which are observed as the lateral LDOS variations at the Co NP surface in figure 2(b). On closer inspection of the positive voltage region in figures 2(b) and (d), it can be seen that the type $\downarrow \downarrow$ NPs exhibit depending on the voltage either a higher or a lower LDOS when compared to the type $\uparrow \downarrow$ NPs. This implies that, in addition to the localized d-states, the delocalized surface
states of Co NPs exhibit an effective spin polarization as well. Previous theoretical calculations that were performed up to energies of 500 mV above the Fermi level predicted that the surface states at Co NPs on both Au(111) [24] and Cu(111) [18] originate from sp-states of majority character. Since in the positive voltage region the tunneling electrons originate mainly from the minority states near the Fermi level of the STM tip, the spin polarization $SP_{ij}$ of the tunnel junction is expected to be negative in this energy region. Type $\uparrow\downarrow$ Co NPs are consequently expected to yield a higher LDOS than type $\downarrow\downarrow$ NPs Co NPs, which is in agreement with the spectra in figure 2(d). At energies above about 750 mV, however, it can be seen in figure 2(d) that the polarization $SP_{ij}$ reverses for reasons that remain unknown to us. To understand the origin of this reversal as well as to understand the voltage dependence of the polarization $SP_{ij}$ in full detail, theoretical calculations similar to that of [17] are obviously required.

Next, we focus in more detail on the spin-dependent LDOS of the Co NPs for which the topography is presented in figure 3(a) within a broad energy range from $-2400 \text{ meV}$ to $+2400 \text{ meV}$. A schematic overview of the different contributions of the majority and minority states to the overall LDOS of the Co NPs, based on our experimental findings and previous results [24, 35, 36], is provided in figure 3(g). For negative voltages, the LDOS of the type $\uparrow\downarrow$ Co NPs is observed to change several times from lower to higher (and vice versa) than that
of the type $↓↓$ Co NPs, in agreement with the spin-resolved $(dI/dV)_{SP}(V)$ spectra discussed above. This implies that the effective spin polarization $SP_{ij}$ of the NP–vacuum–tip tunnel junction changes sign several times within the energy range under investigation. The LDOS maps that revealed the highest contrast between type $↑↓$ and type $↓↓$ NPs for negative voltages are presented in figures 3(b)–(d). Apart from the two minority states-dominated LDOS maps recorded at $-400$ mV (figure 3(d)) and $-1000$ mV (figure 3(c)), high contrast is observed at about $-2000$ mV (figure 3(b)). This spin-polarized state can be linked to another Co 3d band that is known to exist around $E = -2000$ meV. This band is of majority character [35, 36] and hence gives rise to a reversal of the LDOS contrast when comparing the NPs in figure 3(c) to the NPs in figure 3(b).

For positive voltages, where standing waves dominate the LDOS of Co NPs, we are able to resolve LDOS contrast between type $↑↓$ and $↓↓$ Co NPs as well for voltages above 1000 mV, up to the maximum applied voltage of 2400 mV (see figures 3(e) and (f)). This is consistent with the results presented in figure 2(b). Previously, clear spatial contrast in spin-resolved LDOS maps was observed at moderate positive voltages for the case of Co NPs on Cu(111) [15, 17]. As already discussed above, these NPs were significantly larger than the here investigated Co NPs on Au(111). The discrete energy levels of Co NPs on Au(111), resulting from the strongly confined delocalized sp-like Co surface state electrons (represented by a continuous dispersion in figure 3(g)), are shifted to considerably higher energies when compared to the larger Co NPs on Cu(111) due to a much stronger quantum confinement experienced by the spin polarized Co surface state electrons (the energy levels roughly scale with the inverse of the square of the Co NP diameter) [21]. Consequently, the standing wave patterns that occur for these energy levels can only be observed at more elevated energies.

In summary, we have unraveled the spin-dependent electronic structure of Co nanometer size particles on Au(111) by means of spin-polarized STM and STS in a broad energy range. Our measurements provide direct evidence of the existence of localized d-states of minority and majority character below the Fermi level. The out-of-plane spin polarization of the Co NPs is found to reverse several times within the investigated energy range as a result of energy-dependent majority and minority d-state contributions to the LDOS. Moreover, the discrete electronic states resulting from the strongly confined delocalized sp-like Co surface state electrons above the Fermi level are found to be of majority character. Our findings verify the theoretical prediction of Rastei et al [24] that the surface states of Co NPs on Au(111) have an effective spin polarization.

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