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Alexander Liebig, and Franz J. Giessibl

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Alexander Liebig[a)] and Franz J. Giessibl[b)]

AFFILIATIONS

Institute of Experimental and Applied Physics, University of Regensburg, D-93040 Regensburg, Germany

[a)]alexander.liebig@ur.de
[b)]franz.giessibl@ur.de

ABSTRACT

Functionalizing a metal tip with a single CO molecule (CO tip) leads to an unprecedented spatial resolution of small organic molecules by frequency-modulation atomic force microscopy (FM-AFM) at low temperatures. O-terminated Cu tips (CuOx tips) show comparable imaging capabilities as CO tips but exhibit a much stiffer apex. So far, to verify tip functionalization with oxygen (i.e., CuOx tips), scanning tunneling microscopy and AFM images, together with force spectroscopy curves of copper oxide domains, have been compared with calculated data for different tip models. Here, we apply the carbon-monoxide front atom identification (COFI) method and additional force spectroscopy to characterize CuOx tips *in-situ* on a Cu(110) surface. In COFI, a single CO molecule adsorbed on a Cu surface is imaged to atomically resolve the tip apex. Based on our findings, we suggest accompanying tip fingerprinting with COFI and force spectroscopy to identify the atomic and chemical compositions of the apex of CuOx tips for high-resolution AFM experiments.

The spatial contrast in scanning tunneling microscopy (STM)[1] and atomic force microscopy (AFM)[2] depends crucially on the exact geometrical structure and chemical species of the tip apex.[3] The spatial resolution can be increased by controlled modification of the tip apex. In STM, it was found that by trapping a single hydrogen molecule in the tip-sample junction, the internal structure of a molecule adsorbed on a surface can be resolved.[4] Using vertical manipulation,[5] individual atoms and molecules can be picked up with an STM tip to functionalize the tip apex.[6,7] CO-terminated metal tips (CO tips), which can be prepared by picking up a single carbon monoxide molecule from a Cu surface,[6] enable submolecular resolution imaging of organic molecules and bond-order discrimination by AFM.[8,9] Terminating the tip apex with noble gas atoms achieves a similar spatial resolution.[10] Although functionalizing the tip with a CO molecule yields an increase in the spatial resolution, interpretation of atomic-scale contrast with these tips is highly nontrivial: When the tip interacts with the sample, lateral forces cause lateral deflection of the CO molecule that can create artifacts in the images and hence inhibit a direct interpretation of data.[9,11–14] Recently, Mönig and co-workers proposed using oxygen-terminated Cu tips (CuOx tips) that show a comparable spatial resolution and much higher lateral stiffness of the tip apex.[15,16] With such CuOx tips, it is for example possible to quantitatively measure intramolecular bond lengths,[16] which appear elongated when using CO tips,[9] and to scan with video rate imaging velocities theoretically.[17]

While the functionalization of a metal tip with a CO molecule is a well-controlled process,[6] CuOx tips are prepared by repeated collisions of the tip with an oxidized Cu surface.[15] This is in general a random process that involves many atoms of both the tip and the surface, which means that the exact atomic structure and the chemical species of the atoms composing the tip apex after the collision are not known. Knowledge of the chemical and structural identities of the tip is of crucial importance, especially when experiments are accompanied by theoretical calculations involving the tip structure and its chemical composition, and hence, these properties should be well-characterized in the experiment. So far, the verification of tip functionalization with oxygen relies on comparing experimental results with calculations: The experimental STM and AFM images of copper oxide domains are compared with density functional theory (DFT) and nonequilibrium Greens function (NEGF) calculations to determine the chemical identity of the tip apex.[18–21] A description of the atomic tip apex structure requires a large tip model database obtained after extensive and time-consuming simulations, and precise determination of the structural apex composition is not always possible.[21]

Conversely, a carbon-monoxide molecule adsorbed on a Cu(111) surface can be used to image the tip apex with atomic resolution, a technique known as the carbon-monoxide front atom identification (COFI) method.[22–24] In COFI, the tip is scanned at a constant height above the CO molecule which acts as a probe, and the resulting images reveal the atomic configuration of the tip apex [compare Fig. 1(a)]. Additionally, the chemical species of atoms can be distinguished by force spectroscopy. Sugimoto and co-workers[25] showed that the chemical species of different surface atoms can be determined by analyzing the minimum of the short-range interaction between the tip and the individual surface atoms. By knowing the chemical species on the surface, this concept can be transferred to determine the chemical species of the tip apex atom: Hofmann and co-workers[26] distinguished between the chemical species of the frontmost atom of different single-atom metal tips using force spectroscopy above the same chemical species on the surface, i.e., a single CO molecule adsorbed on Cu(111).

In this Letter, we present experimental characterization of CuOx tips by a combination of COFI and force spectroscopy on a Cu(110) surface. In this way, both the structural and the chemical compositions of the tip apices can be determined *in-situ*.

The experiments were carried out on a commercial low-temperature scanning tunneling/atomic force microscope operating at a temperature of 4.4 K (LT STM/AFM, Scienta Omicron GmbH, Taunusstein). We used a qPlus sensor[27] equipped with an electrochemically etched tungsten tip, with a resonance frequency of $f_0 = 46\,597\,\text{Hz}$, a stiffness of $k = 1800\,\text{N m}^{-1}$, and a quality factor of $Q = 482\,321$. The sensor was operated at a constant amplitude of $A = 50\,\text{pm}$ in the frequency-modulation mode (FM-AFM).[28] Except when explicitly mentioned, all data presented here are raw data.

The CuOx tips were characterized and prepared following the procedure described by Mönig and co-workers.[15] Upon low oxygen coverages, Cu(110) undergoes a (2×1)-O added-row (AR) reconstruction [shown in Figs. 1(b) and 1(c)], forming striped copper oxide domains along the [001] direction.[15,29–32] To functionalize the tip apex with oxygen, the tip is repeatedly dipped into the oxide domains until a notable enhancement in corrugation within the oxide is observed: Fig. 1(c) shows an STM image recorded in the constant-current mode with a bare metal tip, resolving the copper oxide domains as darker regions than the clean Cu surface. For comparison, in Fig. 2(d), an STM image of the AR reconstruction recorded with a CuOx tip is shown: Here, the copper oxide domain shows a larger corrugation (see line profiles in Fig. S1 in the supplementary material), which indicates a change in the chemical species of the tip terminating atom.[21] In order to characterize the tips using the COFI method, we additionally dosed approximately 0.005 ML of carbon-monoxide on the surface [see Fig. 1(c)]. It should be noted that the COFI method exploits the vertical on-top adsorption configuration of individual CO molecules on Cu(111) with the O atom pointing away from the surface. Here, we use COFI on a Cu(110) surface, where CO also adsorbs in this vertical on-top configuration as determined by photoelectron diffraction.[33]

The interaction of a CuOx tip with a CO molecule adsorbed on Cu(110) as a function of tip-sample distance is comparable to the findings of Sun *et al.* for a CO tip on CO/Cu(111).[11] Figure 2(b) shows frequency shift vs distance $\Delta f(z)$ spectra recorded with a CuOx tip above the CO molecule and above the bare Cu(110) surface (red and black curves, respectively). The zero point of the experimental z axis defines the closest tip-sample approach in the measurements with this tip. Approaching the surface from $z = 1\,\text{nm}$, the Δf values are initially more negative above the CO molecule as compared to the bare Cu surface, which we attribute to van der Waals attraction between the tip and the CO molecule. Upon further approach, the spectrum above the CO reaches a minimum and becomes less negative, which can be assigned to stronger contribution of short-range Pauli repulsion between the tip and the CO molecule. At this point of maximum contrast z_{COFI}, we record a constant-height AFM image [a COFI image, Fig. 2(a)] of the CuOx tip, resolving a single bright, circularly symmetric feature with a dark ring around it. We show COFI images of our CuOx tip as a function of tip-sample distance in the supplementary material to further illustrate this contrast evolution (see Fig. S2).

The short-range component of the interaction between the tip and the CO molecule can be obtained by performing the "on-off" subtraction method.[11,26,36] We subtracted the $\Delta f(z)$ spectrum recorded above the bare Cu(110) surface from the spectrum recorded above the CO molecule and calculated the short-range force $F_{\text{SR}}(z)$ via the Sader-Jarvis force deconvolution method [compare Fig. 2(c)].[34] The force deconvolution has been explicitly checked for well-posed behavior.[37] We obtain a short-range force minimum of -15 pN for the CuOx tip. In the supplementary material, we present $F_{\text{SR}}(z)$ spectra for twelve different CuOx tips, which all exhibit short-range force minima between -15 pN and -30 pN (see Fig. S3). For comparison, we furthermore show a COFI image and $F_{\text{SR}}(z)$ spectra of a CO-terminated tip recorded on CO/Cu(110) in the supplementary material (see Fig. S4), also exhibiting a short-range force minimum of -15 pN. The values we obtained for both the CuOx and CO tips are close to the value obtained with a CO tip on Cu(111)[11] and show that oxidizing the tip apex effectively reduces the chemical reactivity:[15] short-

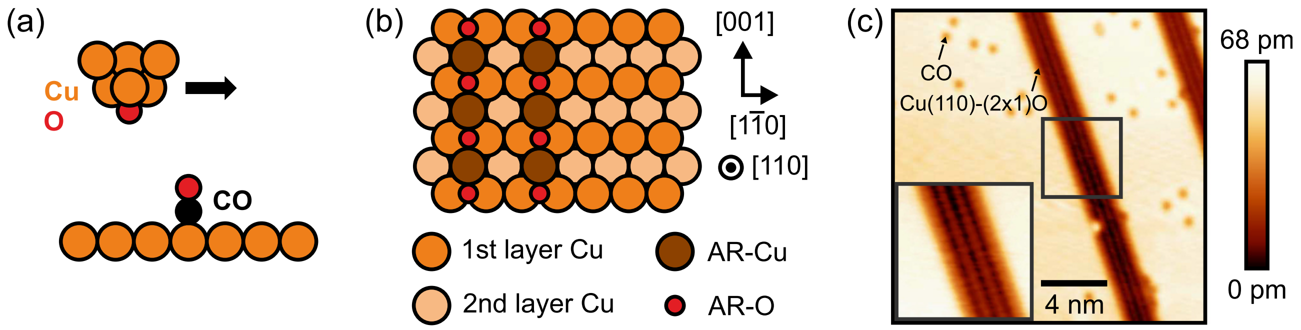

FIG. 1. (a) Schematic of the COFI method: An AFM tip, in this case, a CuOx tip, is scanned at a relatively close distance at a constant height above a CO molecule adsorbed on the Cu(110) surface. (b) Top view of the Cu(110)-(2×1)O added-row (AR) reconstruction.[15] (c) STM overview image of the Cu(110) surface recorded with a metal tip. Dark, striped regions represent the copper oxide domains on Cu(110). Single, dark points are individual CO molecules adsorbed on the bare Cu(110) surface. Imaging parameters: sample bias $V_b = -100$ mV tunneling current setpoint $\langle I_t \rangle = -100$ pA. Inset: Zoom-in of the area marked by the square.

range force minima of metal tips on CO/Cu(111) are typically lower than −130 pN.[26]

We then imaged the Cu(110)–(2 × 1)O reconstruction with the CuOx tip, as has been done in previous studies in which STM and AFM images alongside DFT-based calculations were used to determine the tip apex.[15,16] In Fig. 2(d), an STM topography image of the copper oxide domain is shown, where the maxima within the oxide coincide with AR positions in good agreement with Ref. 16. (Interestingly, the maxima were found between the added rows in Ref. 15, possibly due to the different STM imaging heights,[21] as discussed in section S1 in the supplementary material.)

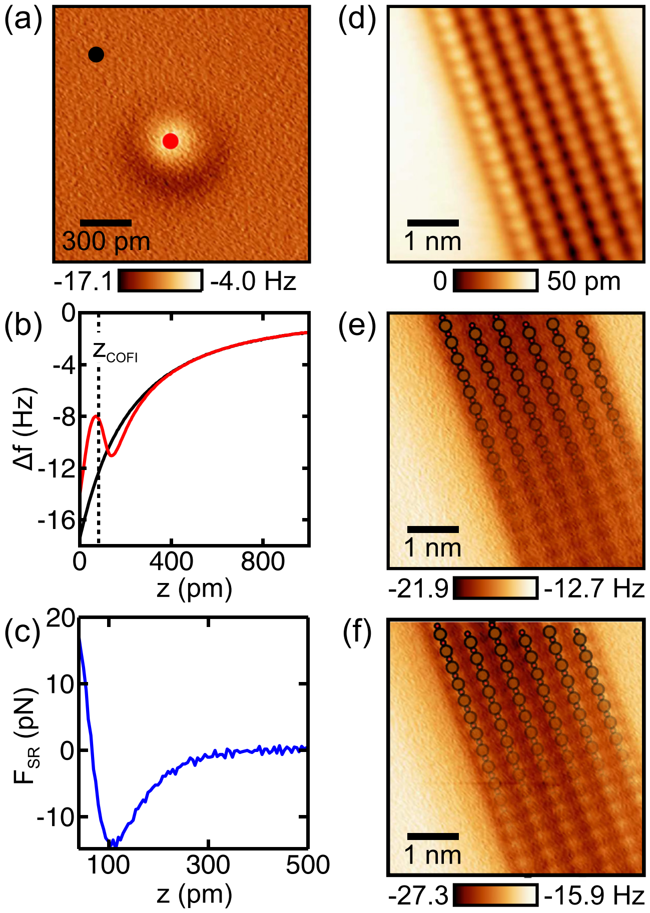

FIG. 2. (a) COFI image of a CuOx tip. (b) $\Delta f(z)$ spectra recorded above the CO molecule (red curve) and the bare Cu surface (black curve), positions marked in (a). The zero point of the z axis defines the closest approach in the measurement, $z = 100$ pm corresponds to the STM setpoint $V_b = -100$ mV and $\langle I_t \rangle = -100$ pA above the bare Cu surface. The COFI image has been recorded at z_{COFI}. (c) Short-range force vs distance $F_{SR}(z)$ spectrum obtained by subtracting the $\Delta f(z)$ spectrum acquired above the Cu(110) surface [black in (b)] from the $\Delta f(z)$ spectrum acquired in the center of the CO molecule [red in (b)] and subsequent force deconvolution.[34] (d) STM image of the copper oxide domain. Imaging parameters: $V_b = -100$ mV and $\langle I_t \rangle = -100$ pA. The image has been processed with a 2×2 Gaussian low-pass filter.[35] (e) and (f) Constant-height Δf images of the same area as in (d) recorded at $z = 50$ pm and $z = 0$ pm, respectively. Far away (e), the AR-Cu atoms are imaged dark, and at closer distance (f), AR-O atoms are imaged bright. The overlaid structure marks positions of AR-Cu and AR-O atoms.

To obtain significant contrast in constant-height AFM images, the tip was then approached to the sample from the STM imaging height. The atomic features that are resolved first are depressions [Fig. 2(e)], which can be attributed to the AR-Cu atoms.[15] The constant-height image in Fig. 2(e) is in excellent agreement with previously reported AFM images recorded of the Cu(110)–(2 × 1)O reconstruction with CuOx tips.[15,16] Further approaching the surface leads to the emergence of increasing Δf above the AR-O atomic sites [Fig. 2(f)], which is expected from short-range Pauli repulsion between the tip and the AR-O atoms (see calculated force vs distance curves in Ref. 15). The good agreement of our STM and AFM data with previous studies of Mönig *et al.*[15,16] is further confirmation that the COFI image

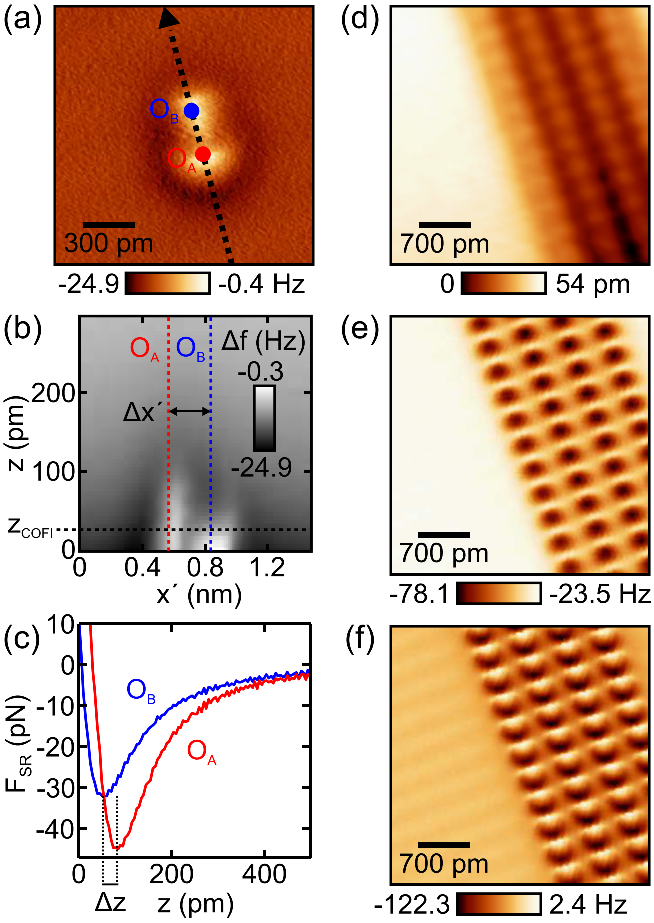

FIG. 3. (a) COFI image of a dimer CuOx tip. (b) $\Delta f(x', z)$ extracted along the dashed line in (a) from a set of z-dependent COFI images of the dimer CuOx tip. Vertical, dashed lines mark the positions of the two O atoms (O_A and O_B) at the tip apex. The COFI image in (a) has been recorded at z_{COFI}. (c) Short-range force vs distance $F_{SR}(z)$ spectra recorded above the two O atoms, positions marked in (a) and (b), respectively. (d) STM feedback image of the Cu(110)-(2 × 1)O added-row reconstruction recorded with the dimer CuOx tip. Imaging parameters: $V_b = -100$ mV and $\langle I_t \rangle = -100$ pA. The image has been processed with a 2×2 Gaussian low-pass filter and z-drift corrected.[35] (e) and (f) Constant-height Δf images of the same area as in (d), recorded 190 pm and 240 pm closer to the surface with respect to the STM setpoint on the bare Cu surface, respectively.

in Fig. 2(a) indeed corresponds to a CuOx tip with a single O atom at the apex [compare Fig. 1(a)].

In Fig. 3, we show experimental data for a tip with an apex consisting of more than one oxygen atom. The COFI image [Fig. 3(a)] shows two bright, repulsive features, which we attribute to two individual oxygen atoms at the tip apex. In the supplementary material, we show COFI images as a function of tip-sample distance for this tip to further illustrate this interpretation (Fig. S5). As the two O atoms are not at the same height above the plane of the surface, the COFI image has an associated tilt asymmetry. Figure 3(b) displays a vertical cut $\Delta f(x', z)$ extracted along the dashed line in Fig. 3(a) from a full set of z-dependent COFI images recorded with this tip. From this vertical cut, the lateral distance between the two O atoms at the tip apex was determined to be $\Delta x' = 250\,\mathrm{pm}$. However, since the lateral deflection of the CO molecule leads to an increased lateral size of atomic features in AFM images,[9] the value of $\Delta x'$ determined here defines an upper boundary for the lateral offset between the two O atoms at the tip apex. Figure 3(c) shows $F_{SR}(z)$ spectra recorded above the two O atoms, respectively. From the two spectra, short-range force minima of −45 pN (atom O_A) and −32 pN (atom O_B) are obtained, respectively, which are lower compared to the values for CuOx tips that have a single O atom at the apex. This can be attributed to the combined interaction of the two apex atoms with the CO molecule that cannot be separated using the on-off subtraction method. To estimate the vertical offset between the two O atoms at the tip apex, we compare the z-positions of the minima in $F_{SR}(z)$ spectra (z^*-method).[38] Based on this method, we determined the vertical offset from the $F_{SR}(z)$ spectra in Fig. 3(c) to be $\Delta z = 35\,\mathrm{pm}$. From the values obtained for $\Delta x'$ and Δz, the distance between the O atoms is found to be $d = 252\,\mathrm{pm}$.

With this "dimer" CuOx tip, we recorded STM and AFM images of the Cu(110)–(2 × 1)O added-row reconstruction for tip fingerprinting as done in Ref. 15. The STM feedback image [Fig. 3(d)] is similar to the image recorded with the tip that had a single O atom at the apex. To obtain significant AFM contrast, the tip was again approached towards the surface from the STM imaging height. Note that the tip had to be moved closer to the surface than the single O atom tip. This can be attributed to the additional tunneling current contribution from the second O atom at the tip apex, which leads to a greater tip-sample distance with STM feedback on.[20,21] Interestingly, at first, we see again the emergence of depressions [Fig. 3(e)], which can be attributed to attractive interaction between the tip and the AR-Cu atoms. This appearance of the copper oxide domain is very similar to images that we recorded with tips that had a single O atom at the apex [compare Fig. 2(e)]. Only when the tip further approaches the sample [Fig. 3(f)], the images of the Cu(110)–(2 × 1)O added-row reconstruction exhibit an asymmetry due to the multi-atom tip apex that is clearly distinct from images recorded with the CuOx tip that had a single O atom at the apex: As compared to Fig. 2(f), the bright features that arise from Pauli repulsion between the tip and the AR-O atoms show a clear asymmetry. However, the determination of the exact tip structure based on the AFM images of the copper oxide domain would require theoretical modeling of such a dimer CuOx tip that has so far not been pursued.

This illustrates the danger of tip characterization with images of the oxide domain which show atomic resolution: Simply because individual atoms of the oxide can be resolved does not mean that the tip apex can be accurately determined, even when compared to simulated data. One attribute of the COFI method is to acquire data at a tip-sample height at which there is significant repulsive interaction between the tip and the adsorbate. This results in a very detailed and complete picture of the apex atoms responsible for AFM imaging. Of course, the effect of multiple atoms will also be seen on the oxide domain when acquiring data at smaller tip-sample distances. However, it is more straightforward to directly determine the geometric positions of the apex atoms with an image of a point feature (a single CO molecule) vs that of a lattice. This is especially true for instances in which the spacing between apex atoms on the tip is comparable to that of the lattice, as in the example of our dimer tip.

In conclusion, we presented an efficient *in-situ* characterization of CuOx tips via the COFI technique and force spectroscopy. COFI images of CuOx tips terminating in a single O atom exhibit a circularly symmetric repulsive feature, very similar to images of CO-terminated tips on Cu(111)[11] and Cu(110). Via force spectroscopy curves recorded with these single atom CuOx tips on CO/Cu(110), we measured short-range force minima ranging from −15 pN to −30 pN, which is similar to data obtained for CO tips and illustrates the decreased reactivity of these tips as compared to bare metal tips.[11,26] While AFM images of the Cu(110)–(2 × 1)O added-row reconstruction recorded with a tip terminating in two O atoms at tip-sample distances at which atomic resolution appears first in constant-height images showed similar features as tips terminating in only one O atom, the COFI image directly resolved the two-atom tip apex. Based on our findings, we suggest to *in-situ* characterize CuOx tips using COFI and force spectroscopy to unambiguously determine the chemical and structural compositions of the tip apex. Knowledge of these properties is of crucial importance, especially if the tips are used for experiments on other sample systems that are accompanied by theory, and the approach presented here provides an efficient way of tip characterization.

See supplementary material for line profiles extracted from the STM images of the copper oxide domain, COFI images of the CuOx tip as a function of tip-sample distance, $F_{SR}(z)$-spectra for twelve CuOx tips, a COFI image and a $F_{SR}(z)$-spectrum recorded with a CO-terminated tip on CO/Cu(110), and COFI images of the dimer CuOx tip as a function of tip-sample distance.

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