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Device for *in-situ* cleaving of hard crystals

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Abstract

Cleaving crystals in a vacuum chamber is a simple method for obtaining atomically flat and clean surfaces for materials that have a preferential cleaving plane. Most *in-situ* cleavers use parallel cutting edges that are applied from two sides on the sample. We found in ambient experiments that diagonal cutting pliers, where the cleavage force is introduced in a single point instead of a line work very well also for hard materials. Here, we incorporate the diagonal cutting plier principle in a design compatible with ultra-high vacuum requirements. We show optical microscopy (mm scale) and atomic force microscopy (atomic scale) images of NiO(001) surfaces cleaved with this device.

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I. INTRODUCTION

Surface science relies on the availability of atomically clean and well-defined surfaces. Cleaving a crystal in an ultra-high vacuum environment is a simple means to provide clean and atomically flat surfaces. Existing implementations of cleavers use a spring-loaded blade that moves towards an anvil [1, 2, 3] with parallel edges, an anvil with one blade that presses against the sample that is to be cleaved [4, 5, 6] or a device that pushes the sample against an obstruction [7, 8, 9]. Other techniques use one blade that is introduced from one side of the vacuum chamber with a wobble-stick type feedthrough hitting a sample that is countered by a fixed or movable anvil [10]. Cleaving crystals *in situ* is relatively simple for soft materials, such as alkali halides. However, cleaving hard materials as NiO is more difficult. We could successfully cleave a strip cut from a NiO wafer with a cross section of roughly 2 mm by 0.6 mm with the techniques described in references [7, 8, 9]. However, in particular on hard samples, the sample areas close to the crystal boundaries show very poor surface quality and it is desirable to provide cleavage faces with greater lateral dimensions to obtain good quality surface areas. We found in ambient experiments that cleaving NiO with a razor blade that touches the sample along a line is rather difficult, while cleaving it with a wire cutter is simple and requires little force even for large cross sections. In a wire cutter, the cleavage force is introduced in a single spot. This highly localized stress field is known to facilitate cleaving [11]. We therefore aimed to incorporate this principle in a vacuum compatible form, shown in the next section.

II. EXPERIMENTAL IMPLEMENTATION

Our cleaver (see Fig. 1 (a)) sits on a single CF 35 flange that holds a rotary feedthrough capable of transmitting a torque of 10 Nm mounted on a CF 16 flange. The rotary feedthrough connects to an axle that has the cleavage knife mounted to it at its end. The CF 35 flange has a pipe with venting holes welded to it, holding an anvil at its end and housing a ball bearing that supports the axle. Because the knife edge is not parallel to the surface when the crystal is not cleaved yet, the cleavage force is applied at a well-defined point (see Fig. 1 (b)), facilitating the cleavage process and usually resulting in fairly flat cleavage planes. When the knife edge is touching the crystal along its whole length, as done in many previous cleavage designs, the cleavage can start at multiple points that are on different parallel crystal planes (see Fig. 1 (c)). The rotating blade and the steady anvil resemble the two cutting blades of a wire cutter. In the present cleaver, the blade is made of stainless steel, but it could also be manufactured from hardened steel or other hard materials such as tungsten carbide. The blade angles are chosen such that once the cleave is initiated, the blade pushes the sample backwards and the blade does not scratch over the freshly cleaved surface (Fig. 1(c)). The maximum force that can be applied to the initial point of contact between knife and sample is given by the length of the arm and the maximal torque rating of the rotary feedthrough. In our setup, it is approximately 1 kN.

III. RESULTS

Figure 2 shows an optical microscopy image of a typical cleavage plane. The steplines clearly merge into the point where the initial cleaving force was applied. The cleavage process is shown in real time for soft (KBr) and hard (NiO) materials by two movies, online available at www.xxx.yyy. The advantage of a large cleavage area is that high-quality surface areas are more likely to be found. In most experiments, it is not possible to obtain atomic resolution over the whole surface area (see caption Fig. 2). Debris on surfaces is especially harmful for scanning probe microscopy studies, because the radii of the probe tips are often large and even small pieces of debris on otherwise perfectly flat and clean surfaces can prevent the tip apex from reaching the flat surface, because other tip sections may make contact with the debris before the apex reaches the sample.

With the cleaver shown in Fig. 1, we could easily cleave cross sections on the order of 2 by 4 mm^2 and we could find surfaces that are clean enough to allow atomic resolution by AFM in approximately 9 out of 10 cleavage trials. Figure 3 shows typical atomic force microscopy images of an *in situ* cleaved NiO(001) surface that was produced with this device.

Because the cleaver can be mounted on a single flange, it is very compact. As it uses only a single rotary feedthrough and few other parts, it is easy to build and quite cost effective.

IV. ACKNOWLEDGMENTS

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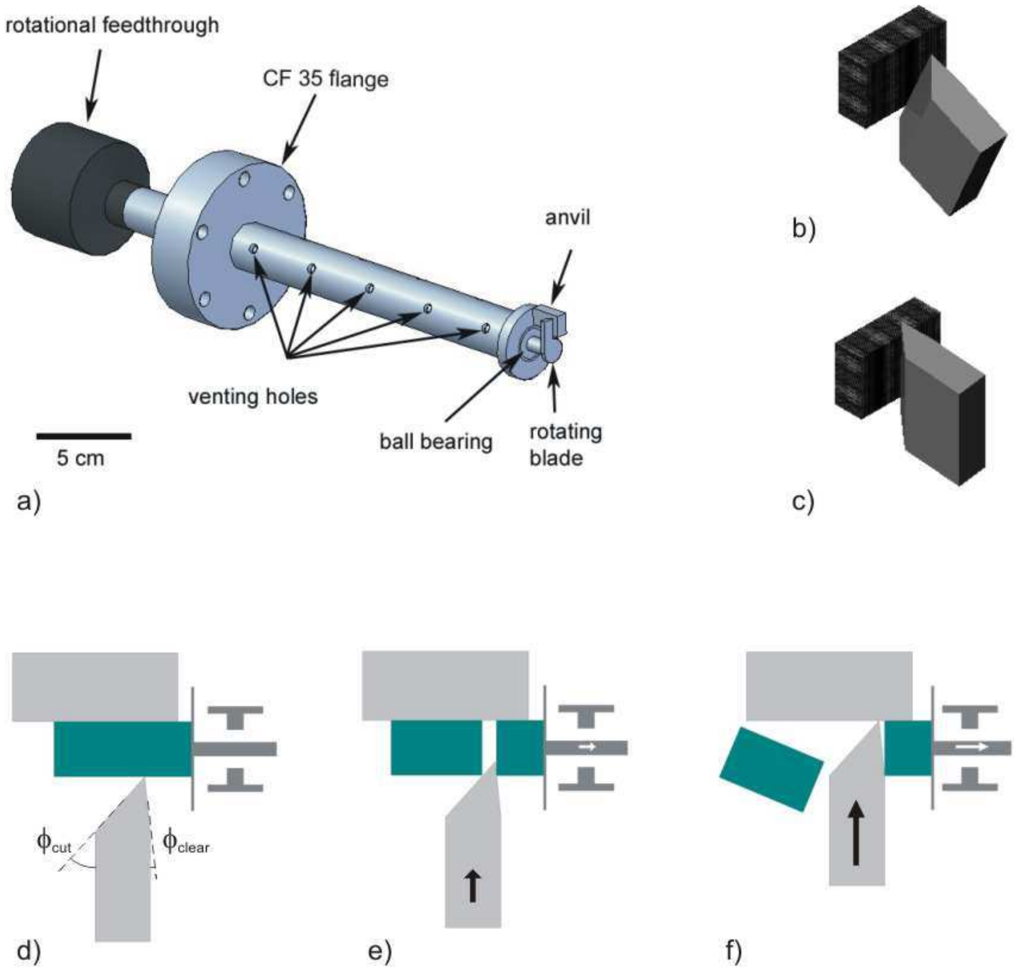

FIG. 1: (a) Perspective view of the single-flange cleaver. It consists essentially of three parts: a rotary feedthrough, an axle holding the cleavage knife that is connected to the rotary feedthrough and a tube with venting holes that has the anvil welded to it. (b) Cleaving a crystal using the wire cutter principle results in a well-defined point where the cleave is initiated. In contrast to a cleaver where the blade is applied parallel to the side faces of the crystal sample to be cleaved (c), the wire cutter principle does not require perfect alignment between the cutting edge and the intended cleavage direction. (d) Schematic view of the blade with cutting angle ϕ_{cut} and clearance angle ϕ_{clear} in the initial phase of cleavage. In our setup, $\phi_{cut} \approx 42°$ and $\phi_{clear} \approx -6°$. (e,f) The negative clearance angle ensures that the cleavage plane remains unscathed when the blade slides over the sample surface after the cleave (see also online movies). The sample holder is movable along a line perpendicular to the cleavage plane, enabling the sample to move backwards (white arrow in (e) and (f)).

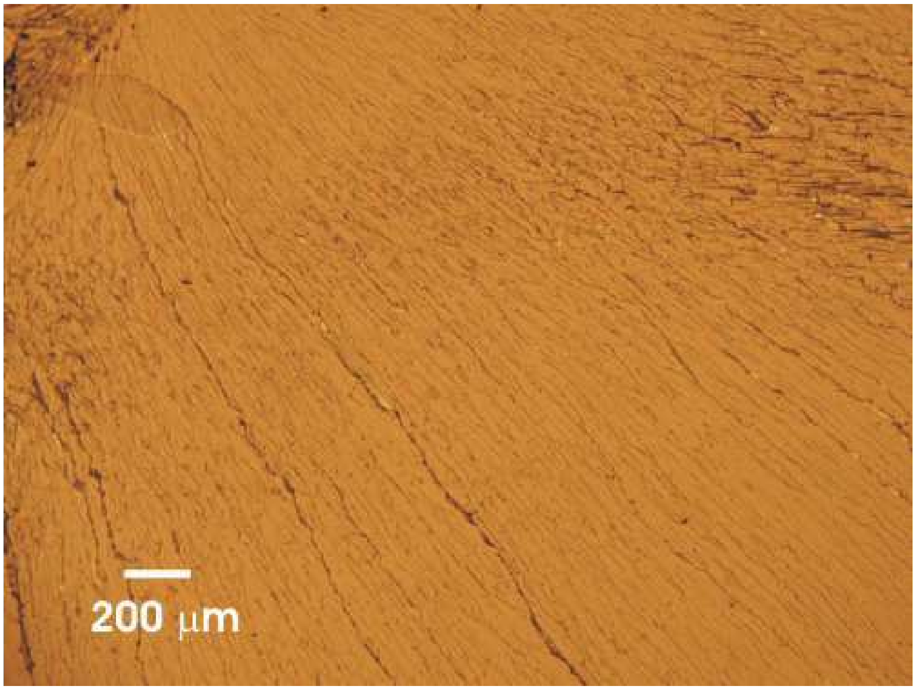

FIG. 2: Optical microscopic view of the cleaved NiO(001) surface. Cleavage was initiated on the top left corner of the sample. The central region of the cleavage plane has a rather good surface quality, while the sections close to the boundaries are more rugged and are sometimes covered with debris.

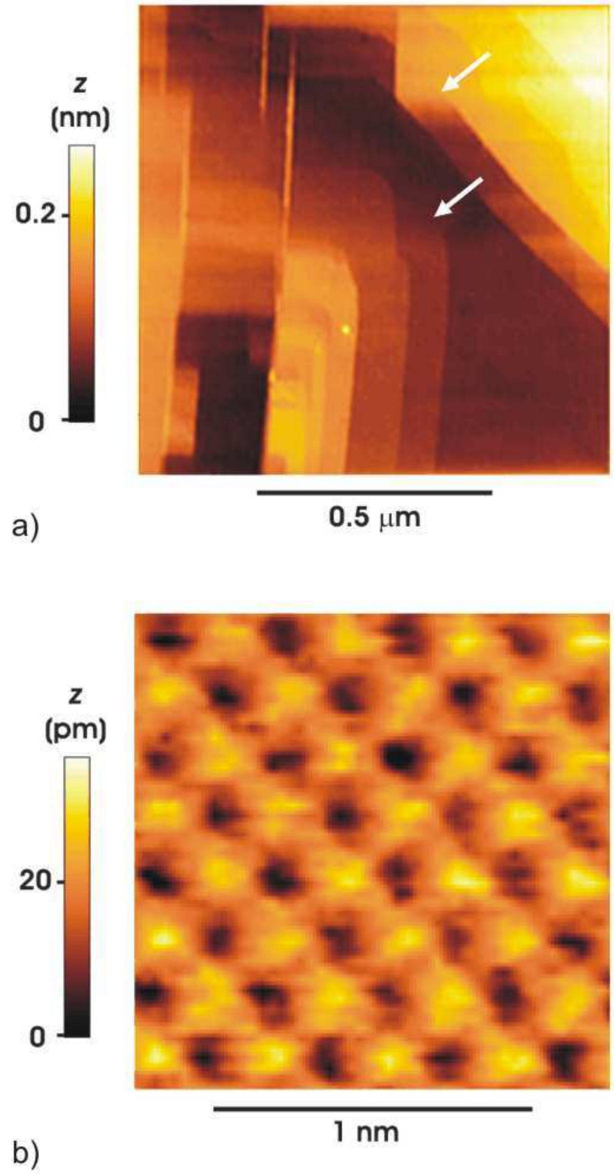

FIG. 3: Atomic force microscopy images of the cleaved NiO(001) surface. NiO(001) has a rocksalt structure with a cubic lattice constant of $a_0 = 417\,\mathrm{pm}$ and natural step heights of integer multiples of $a_0/2$. (a) Image size $1\,\mu\mathrm{m}\times 1\,\mu\mathrm{m}$, showing atomic steps with heights of $a_0/2$ and a_0. At the top, the NiO crystal is 3 unit cells higher at the right edge than at the left, and at the bottom, the left edge is 3 unit cells higher than the right edge. The misorientation angle of the actual cleavage plane with respect to the ideal (001) plane is thus on the order of $3\,a_0/1\,\mu\mathrm{m}\,\mathrm{rad} = 0.07°$. The image shows that the (001) surfaces are not ideal – a few screw dislocations are present (arrows). (b) Atomic resolution image (size $1.5\,\mathrm{nm}\times 1.5\,\mathrm{nm}$). Images of this quality could only be obtained on surface areas that are flat and free of debris for fairly large areas. The larger the area of a cleaved surface, the more likely it is possible to find such good surface areas.