AFM Based Investigations of ZnO Varistor Ceramics

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Introduction

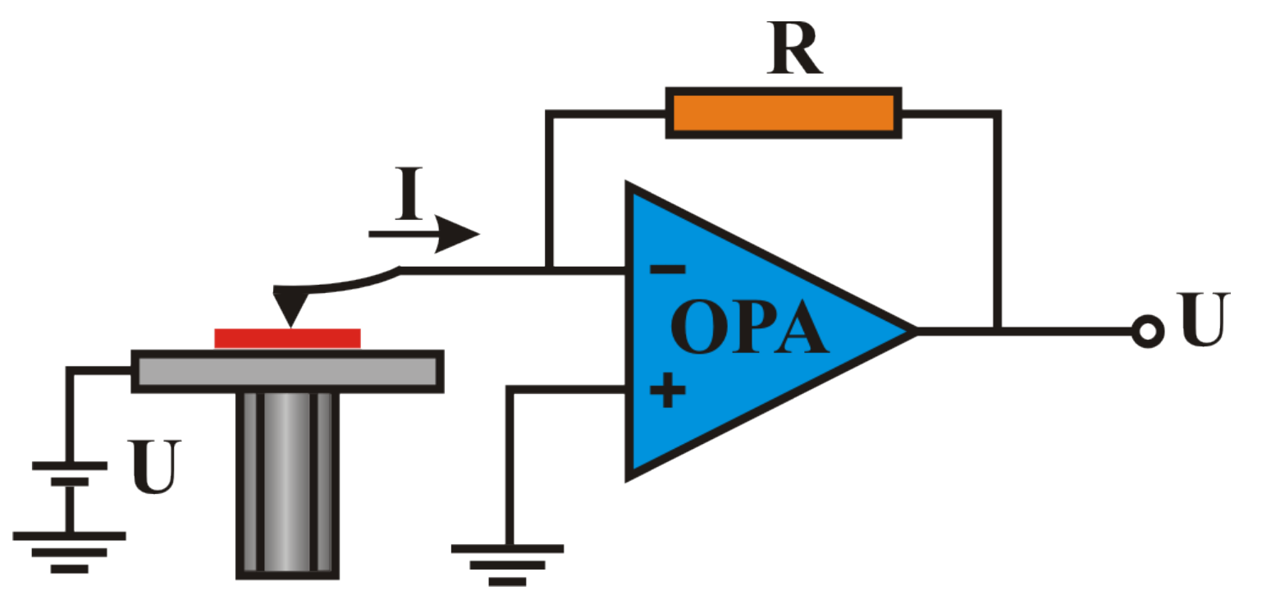
Varistors are electroceramic components used as **high voltage protection** in electronic devices. The standard varistor material is **polycrystalline ZnO** which shows **highly non-linear current-voltage characteristics** with rapidly decreasing resistance above a specific voltage. This effect is caused by double Schottky barriers at grain boundaries [1]. Therefore, the macroscopic behavior of varistors strongly depends on the microstructure of the ceramic material [2].

In this study, multilayer ZnO varistors were investigated on the microscopic scale by Conductive Atomic Force Microscopy (**C-AFM**) and Kelvin Probe Force Microscopy (**KPFM**). The crystallographic orientation was obtained with Electron Backscatter Diffraction (**EBSD**). Here we demonstrated that the conductivity, the work function, and the crystallographic orientation can be measured on the same ensemble of ZnO grains with **micrometer resolution**. Aim of this project is to find a correlation between conductivity, work function, and crystallographic orientation of ZnO grains and grain boundaries in order to get insight into the behavior of ZnO varistors. Preliminary results are shown.

Samples

- Commercial Pr-ZnO multilayer varistors from EPCOS OHG.
- Cross-sections perpendicular to electrode orientation.
- Ground and mechanically polished with 0.25 μm diamond suspension.

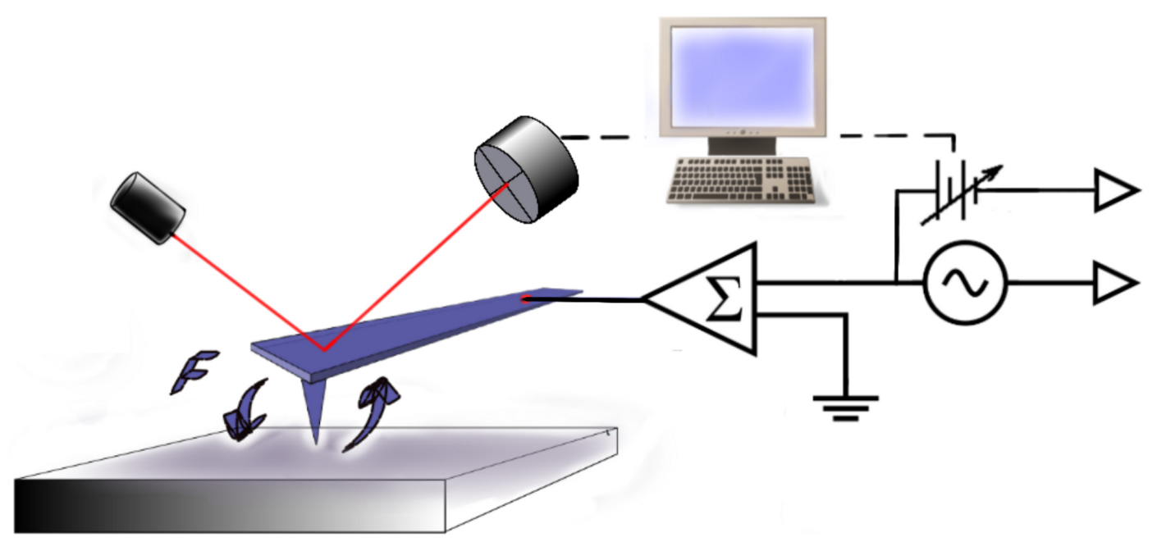
Experimental Setup



Asylum Research MFP 3D AFM

Conductive AFM (C- AFM):

- Contact mode measurement
- Voltage between tip and sample
- Current is measured
- Local I – V curves



Kelvin Probe Force Microscopy (KPFM):

- Two pass technique
- First pass: topography in tapping mode
- Second pass:
  The tip follows the surface in constant height
  cantilever is excited by AC voltage,
  DC voltage to obtain the contact potential difference (CPD)

Probes:

- Highly doped diamond coated tips for C-AFM, k = 2.5 –10 N/m, f = 47–150 kHz, r = 35 nm
- TiN coated tips for KPFM, k = 0.35 – 6.1 N/m, f = 115–190 kHz, r = 35 nm

Zeiss Leo 1525 with an EDAX EBSD detektor

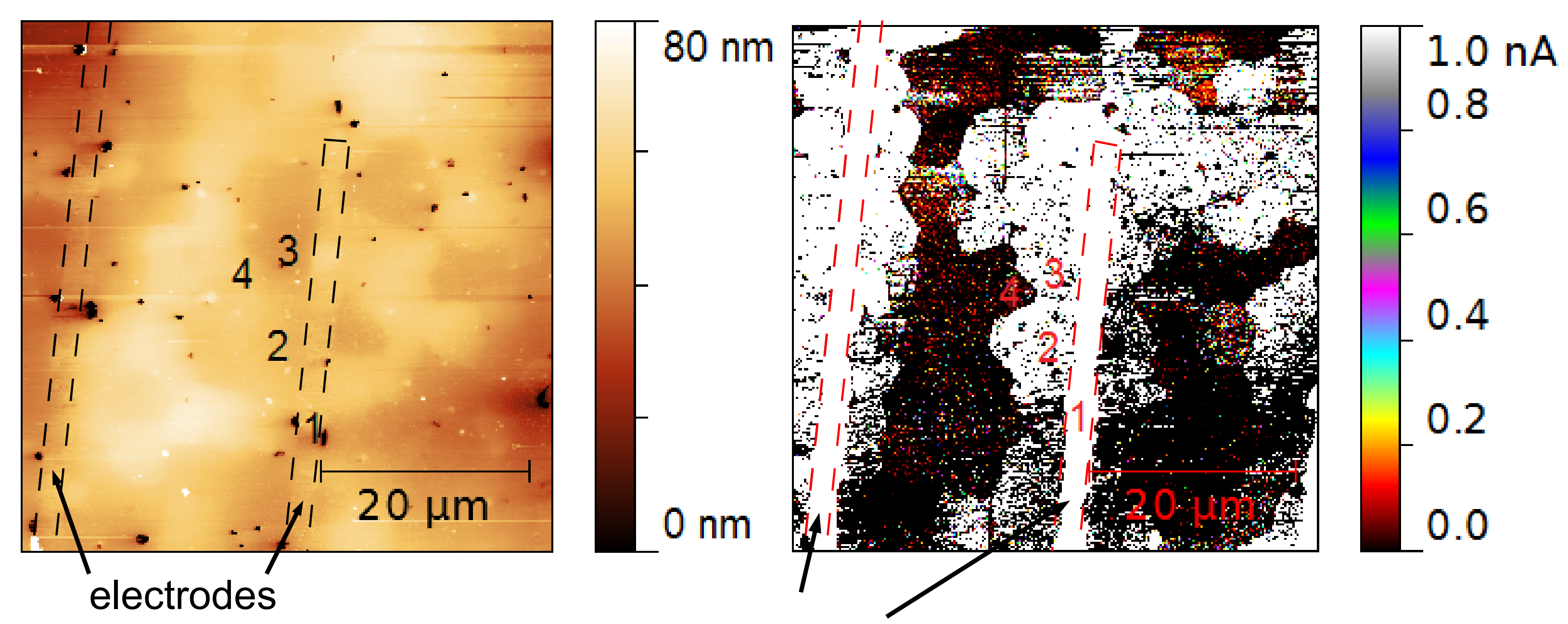
Electron Back Scatter Diffraction (EBSD):

The diffraction pattern of backscattered electrons in a Scanning Electron Microscope (SEM) is analyzed to obtain the crystallographic orientation of the grains at the surface of the polished, cross-sectioned sample.

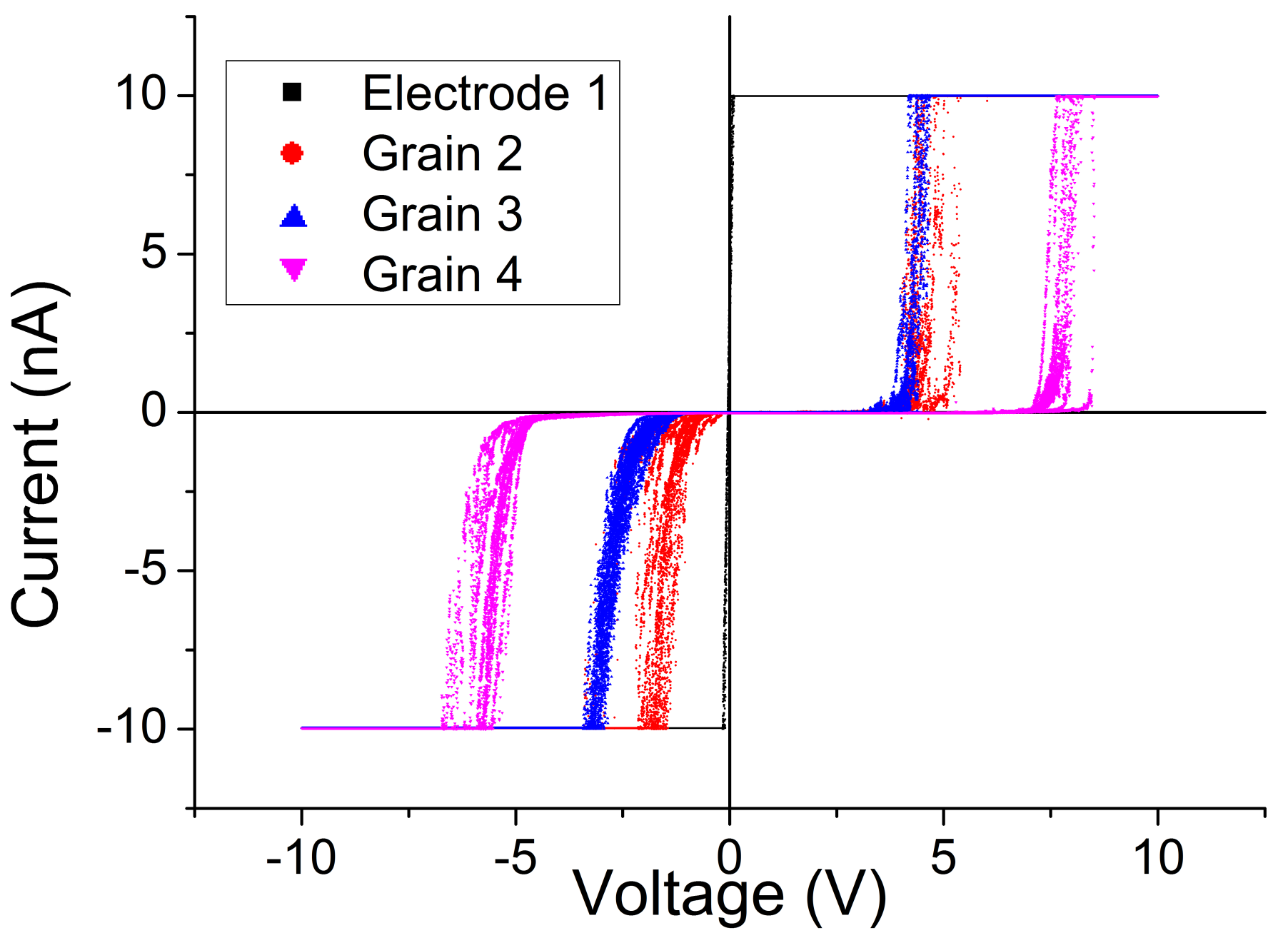
C-AFM, KPFM and EBSD on the same area

Height image

Current image @ + 8 V

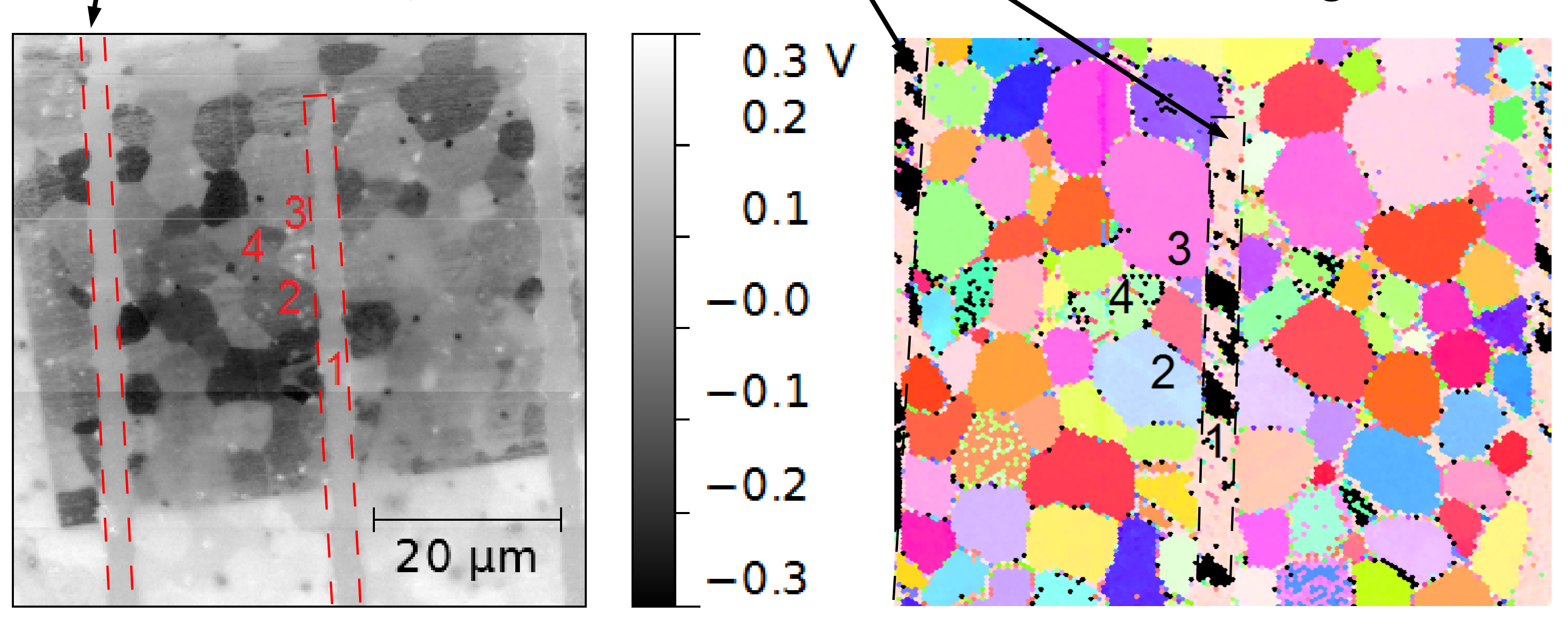


I - V curves



CPD image

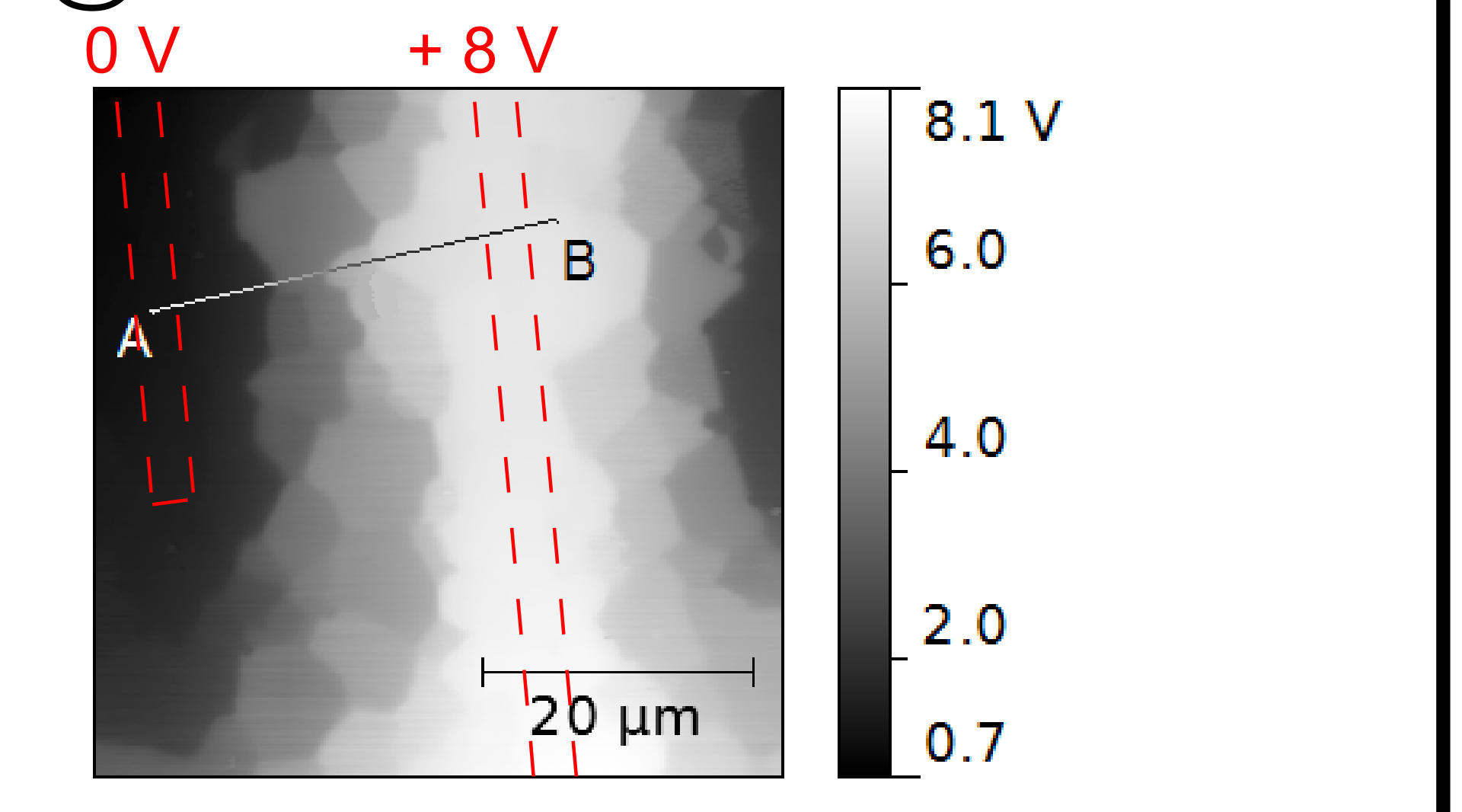
EBSD image



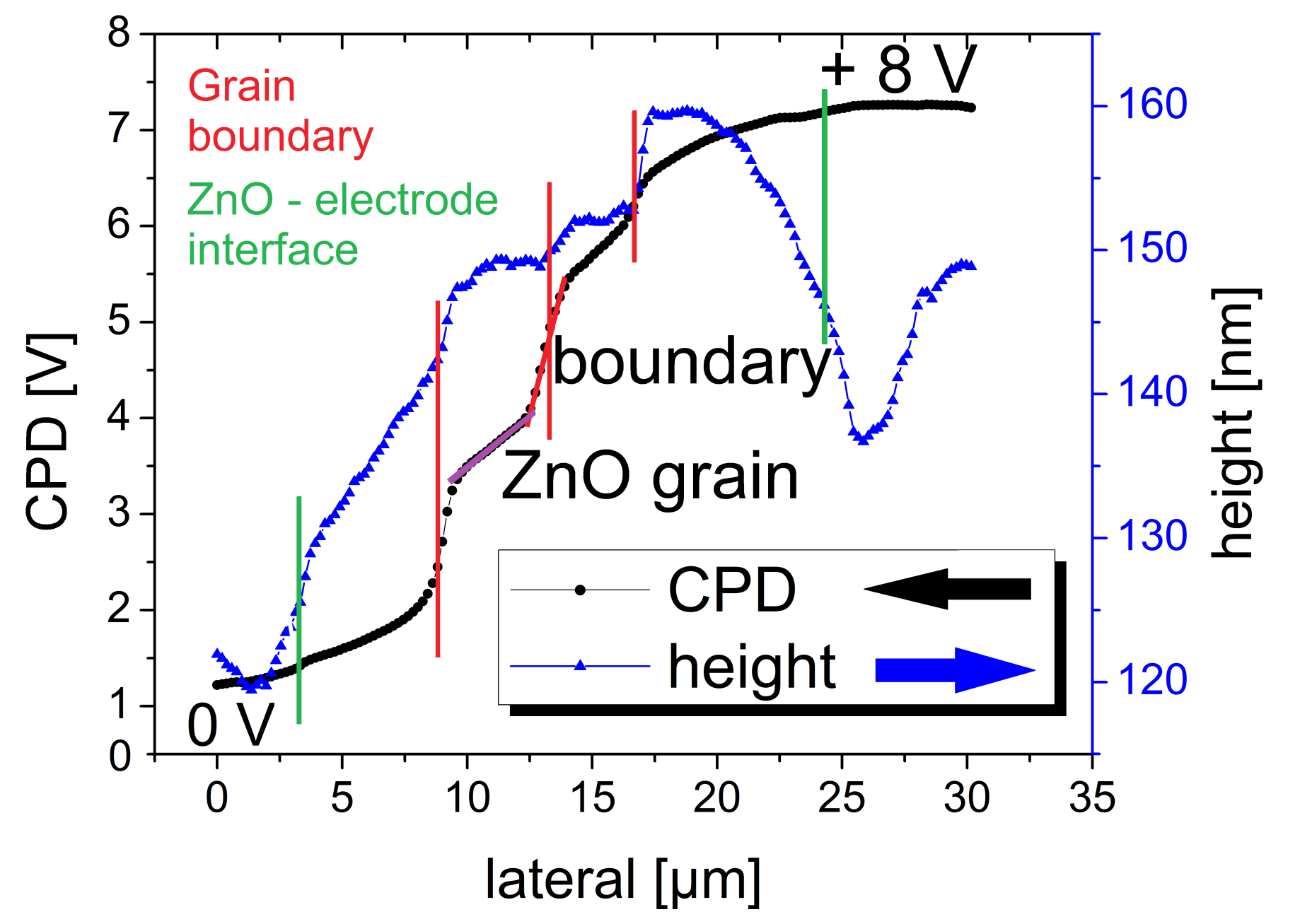
- Height-, current-, and CPD images coincide with EBSD measurements.
- Single grains in current, CPD, and EBSD images are clearly identified.
- In C-AFM, an almost constant current signal within a grain is measured.
- The difference of individual grains in CPD is clearly visible.
- I - V curves depend on the individual grain and its distance to the electrode.

Biased KPFM

CPD @ 8 V bias between the electrodes



Cross section A - B



- No substantial voltage drop at interface between ZnO grains and electrode
- Voltage drop mainly at grain boundaries

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Literature

[1] D. R. Clarke, J. Am. Ceram. Soc. 1999; **82**: 485-502
[2] Ö. Özer et al., Acta Materialia 2010; **58**: 4126-4136
[3] S. Hirose et al., J. Appl. Phys. 2008; **104**: 013701

Summary

- C-AFM and biased KPFM show a higher resistance at grain boundaries compared to the bulk material.
- A difference in conductivity as well as in work function for individual grains can be seen.
- Biased KPFM shows clear evidence of Schottky barriers between ZnO grains but no pronounced Schottky barrier between the electrode and the adjacent ZnO grains in agreement with recent literature [3].

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