Kelvin probe force microscopy investigations of the contact charging of single crystalline insulators

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Introduction

Detailed knowledge about the **contact charging** behavior of **dielectric materials** is of great interest for technological applications like **tribocharging separation**[1,2] of mineral particles. The underlying **mechanisms** are still **not** well **understood**.

Here, an attempt is made to study the electric charging of well-defined surfaces (quartz and calcite monocrystals) upon contact with a conductive AFM tip.

Kelvin probe force microscopy (KPFM)[3] was applied to verify the electrostatic characteristic of the surfaces before and after contact charging. Both, tribocharging due to **rubbing** and **static contact** charging with applied tip **bias** have been investigated.

Experimental

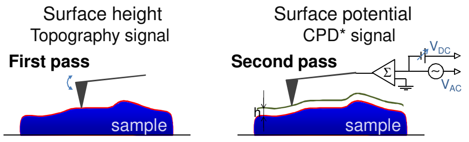
Equipment:

Asylum Research
MFP-3D AFM

Probes:

TiN coated tips for noncontact AFM, spring constant 22 – 100 N/m, tip curvature radius ~35 nm

KPFM measurements



Surface height
Topography signal

Surface potential
CPD* signal

First pass

Second pass

Samples:

monocrystalline **quartz**, SiO_2, Y-cut, $(10\bar{1}0)$ and X-cut $(11\bar{2}0)$, MTI Corporation, USA
monocrystalline **calcite**, $CaCO_3$, (100), MTI Corporation, USA

*) CPD – contact potential difference

Measurement procedure:

Topography / **KPFM** measurements → **Charging** by AFM tip → Topography / **KPFM** measurements

Charging:

Rubbing:

The AFM tip with applied bias (±10 V) is dragged on chosen surface area with defined force and speed.

Static contact:

The AFM tip with applied bias (±10 V) brought into contact with defined force and for defined time.

Results

Charging by rubbing

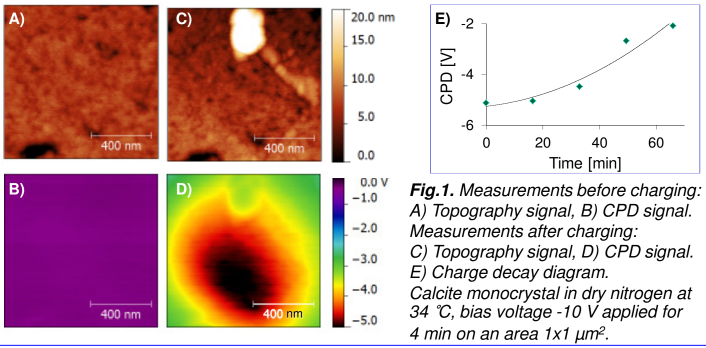


Fig.1. *Measurements before charging:*
A) Topography signal, B) CPD signal.
Measurements after charging:
C) Topography signal, D) CPD signal.
E) Charge decay diagram.
Calcite monocrystal in dry nitrogen at 34 ℃, bias voltage -10 V applied for 4 min on an area 1x1 μm².

Charging by static contact

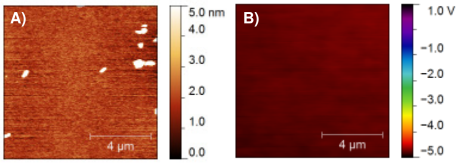


Fig. 2. *Measurements before charging (scan area 10x10 μm^2): A) Topography signal, B) CPD signal.*

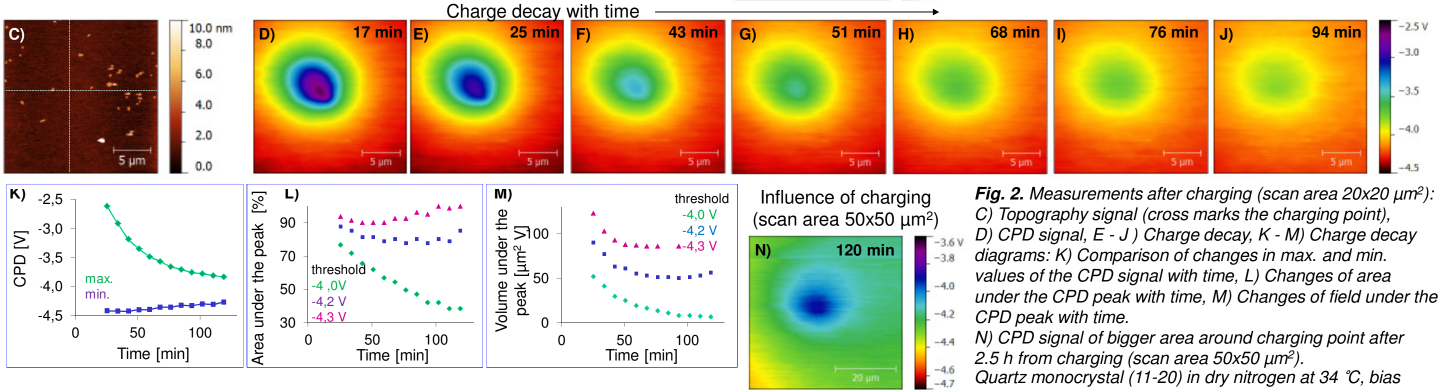


Fig. 2. *Measurements after charging (scan area 20x20 μm^2): C) Topography signal (cross marks the charging point), D) CPD signal, E - J) Charge decay, K - M) Charge decay diagrams: K) Comparison of changes in max. and min. values of the CPD signal with time, L) Changes of area under the CPD peak with time, M) Changes of field under the CPD peak with time.*
N) CPD signal of bigger area around charging point after 2.5 h from charging (scan area 50x50 μm^2).
Quartz monocrystal (11-20) in dry nitrogen at 34 ℃, bias voltage +10 V applied for 10 min.

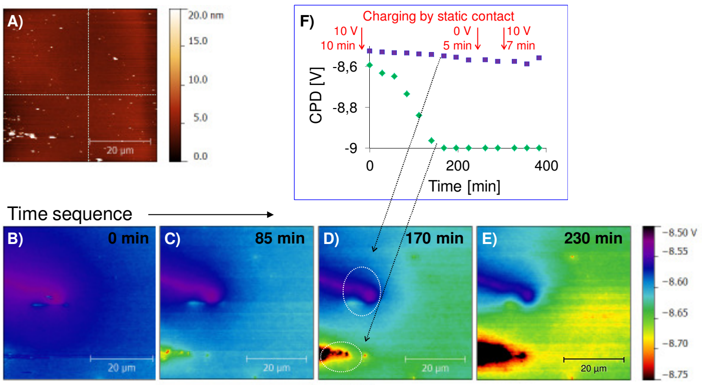


Fig. 3. *Measurements after charging:*
A) Topography signal (cross marks the charging point), B) CPD signal immediately after charging. C - E) Charge development of specific features presented on the insulator surface (the feature is the middle of the scan is unknown, the feature in the bottom left corner corresponds to particles present on the surface). F) Charge development diagram.
Quartz monocrystal (10-10) in dry nitrogen at 34 ℃, a bias voltage +10 V applied for 10 min, scan area 50x50 μm^2.

Discussion and conclusion

1. Charging by rubbing (Fig. 1) and by static contact (Fig.2) can cause significant changes in CPD signal, ΔCPD is -5V and +2V, respectively.
2. Electrical changes on the surface due to charging reach a large area around the charging place (Fig. 2H); even for measurements in dry atmosphere (5 % rH.).
3. An additional effect of charging by rubbing is a slight change in topography caused by dragging the AFM tip on the surface (Fig. 1).
4. Charging is not always effective (Fig. 3). There are areas which do not change potential upon contact with the biased AFM tip.
5. Decay of the charge was observed (Fig. 1, 2):
    a. (Fig. 2K) Maximum value (height of the CPD peak) is strongly reduced with time. Minimum value (surrounding of the CPD peak) slightly increases which can be attributed to slight spreading of the charge on the surface.
    b. (Fig. 2L) Significant differences in the changes of the area under the CPD peak for different threshold values (in particular a strong decrease of the area above -4.0V) show that most of the charge was lost by interaction with the atmosphere.
    c. (Fig. 2M) The decrease of the integral CPD signal for all threshold suggests that most of the implemented charge decays by interaction with enviroment or is spreaded in bigger area.
6. Specific features on the insulator surface (or in the subsurface layer) can develope charges during scanning (Fig. 3). Rate of CPD changes varies between features. Changes caused by these features are much stronger than charging the surface by static contact with the AFM tip (max value of the tip bias was +10 V).

Literature

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