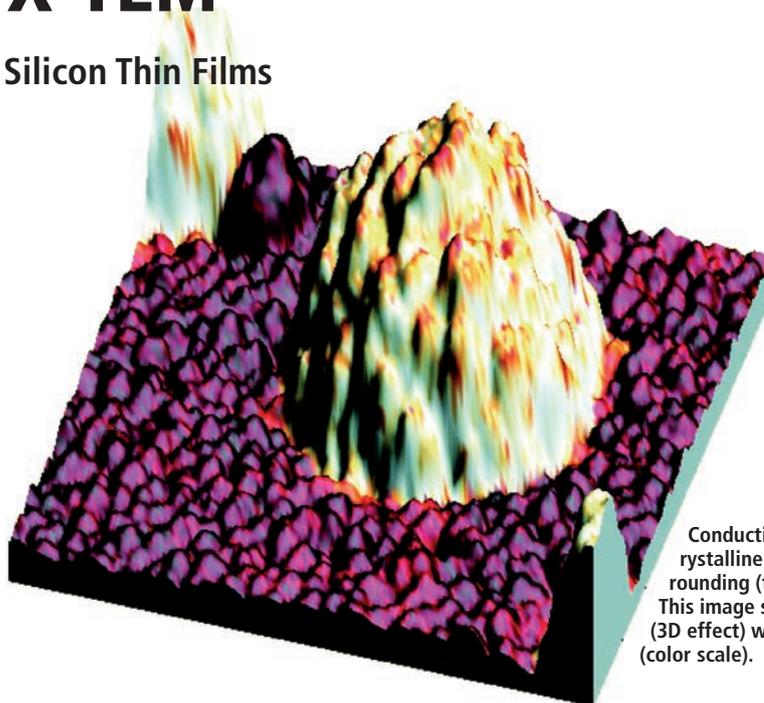


# C-AFM and X-TEM

## Studies of Mixed-Phase Silicon Thin Films



Dr. Tomáš Mates



Conductive AFM scan of the microcrystalline grain and amorphous surrounding (the same data as in Fig. 2). This image shows merged topography (3D effect) with local current values (color scale).

Thin intrinsic silicon films containing microcrystalline grains embedded in amorphous tissue were studied by two complementary microscopy techniques. The conductive atomic force microscopy was performed in standard ambient conditions with very sensitive (pA) current detection. The cross-sectional transmission electron microscopy images of the amorphous phase revealed the columnar structure, which was attributed to the bumpy structures on the surface.

### Keywords:

Conductive Atomic Force Microscopy, Cross-sectional Transmission Electron Microscopy, Silicon Thin Films

### Introduction

Thin Si films with mixed-phase structure (or close to the microcrystalline/amorphous transition) seem to be the optimal material for thin film solar cells [1]. However their complicated inner structure, containing the mixture of microcrystalline ( $\mu\text{c-Si:H}$ ) grains embedded in amorphous (a-Si:H) tissue, is strongly dependent on deposition conditions. There are still many open questions concerning the electronic properties of these films. An intensive effort to fabricate cheaper thin Si films e.g. by lowering the deposition temperature leads to new research challenges.

### Experimental

The samples (intrinsic Si films) were prepared by plasma enhanced chemical vapor deposition (PECVD) at low deposition temperature of  $T_s=39^\circ\text{C}$ , hydrogen dilution ratio  $r_{\text{H}}=[\text{H}_2]/[\text{SiH}_4]=40$  and  $f_{\text{exc}}=50$  MHz [2]. The thickness of the deposited Si films was around 900 nm. The sample "A" was deposited on Corning 1737F glass, while "B" on Cr coated glass. A part of the sample A was prepared [2] for the cross-sectional transmission electron microscopy (X-TEM) measurements.

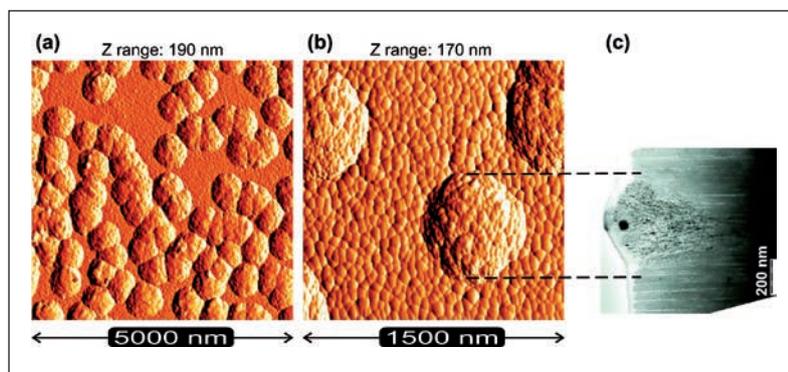


Fig. 1: Structure of the sample A: (a), (b) surface measured by the tapping mode AFM and (c) cross-section by the X-TEM. The parts (b) and (c) are shown in identical scale for the comparison.

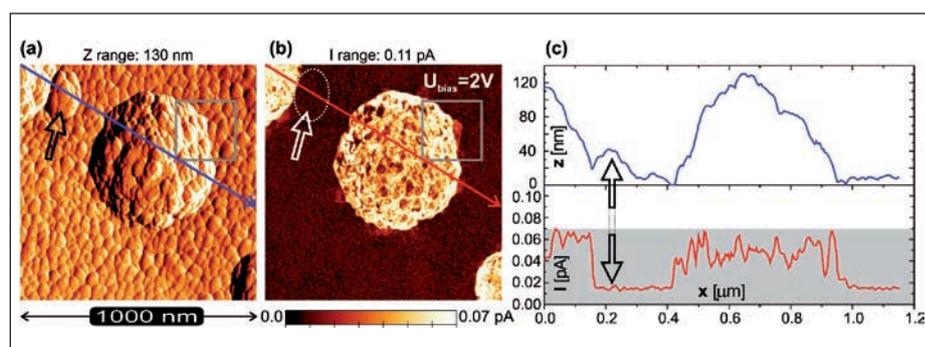


Fig. 2: C-AFM measurement of the sample B showing (a) topography and (b) local current map measured simultaneously at  $U_{\text{bias}} = 2\text{V}$ . The part (c) shows the line profiles of the local height ( $z$ ) and local current ( $I$ ) along the longer arrows indicated in (a) and (b). The grey stripe in (c) represents the narrowed data range used for the local current map in (b).

The other part of the sample A was measured by the tapping mode AFM, the sample B was measured by the contact mode conductive AFM (C-AFM) with a very sensitive current detection (pA range). The AFM topography images show the real signal obtained as the amplitude or the deflection of the cantilever. The "Z range" or "I range" above each image shows the full height or current data scale of the inspected area. The AFM data were processed by the WSxM software [3], further details of the measurements can be found in [4].

## Results

The AFM topographies in Figs. 1(a), and (b) indicate that the sample A is a typical mixed-phase Si sample containing  $\mu\text{-Si:H}$  grains in a-Si:H tissue. The larger scale view in fig. 1(a) reveals the arrangement of these grains into the interconnected network, just behind the percolation threshold. This is probably the reason for the best optoelectronic properties of this sample [2].

A smaller scale topography image (fig. 1(b)) is shown together with the X-TEM image (c), both in the same scale. This comparison of two different microscopy methods used for one sample shows a very good agreement of the size of  $\mu\text{-Si:H}$  grains. The conical shape of the  $\mu\text{-Si:H}$  grain observed in fig. 1(c) is in agreement with our simple model of growth of  $\mu\text{-Si:H}$  [5].

However, there is something new and surprising in the X-TEM image – the bright horizontal lines in the bottom part of fig. 1(c) indicate some columnar structure of a-Si:H, which has not yet been seen in similar films by the X-TEM before. The a-Si:H columns in the X-TEM image in fig. 1(c) correspond to the small topography bumps in fig. 1(b) [4].

This interesting structure of the a-Si:H motivated us for

the further study of the sample B (the same structure prepared on Cr coated glass enabling the C-AFM). The topography in fig. 2(a) fully corresponds to the topography of the sample A shown in fig. 1(b). The local current map in fig. 2(b) exhibits brighter spots for the more conductive  $\mu\text{-Si:H}$  grains, while the dark surrounding represents the less conductive a-Si:H. The same is valid for the line profiles in fig. 2(c). There is only one exception – a bump in local height indicated by the short arrows – which must be amorphous, since it has no corresponding local current signal.

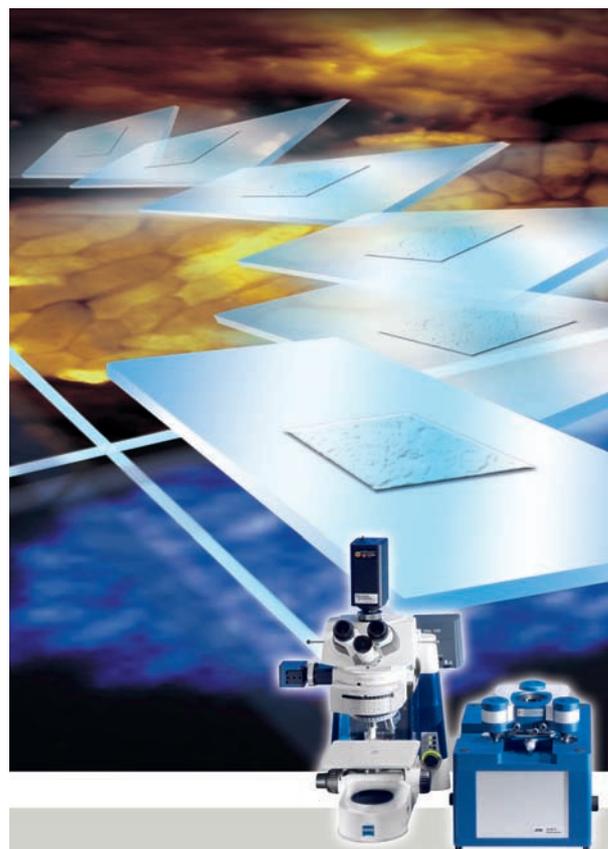
Since our first work done in UHV [6] it has been supposed that the surface oxidation of the Si films is a major obstacle for their measurement in air. Here we demonstrate that the C-AFM in air is feasible if a sensitive current detection is employed. Due to other factors influencing the C-AFM measurement (normal force, scanning speed, tip condition, etc. [4]) the local current should be only considered as a qualitative characteristics of the electronic properties.

Macroscopically, the  $\mu\text{-Si:H}$  is by 3–5 orders of magnitude more conductive than the a-Si:H structure. Therefore, one would expect the local current signal to slowly decrease as the distance from the  $\mu\text{-Si:H}$  grain edge increases. However, this is not the case of our low temperature samples! A selected area of  $250 \times 250 \text{ nm}^2$  – grey squares in fig. 2) was further inspected by the C-AFM. The part of the  $\mu\text{-Si:H}$  grain and its edge can be easily recognized by the bright spot in the left half of the local current maps in fig. 3(b). The a-Si:H phase is again significantly less conductive (darker) with only one exception indicated by the arrow. This spot precisely corresponds to the topography bump in fig. 4(a), which is nothing but the surface part of the a-Si:H column

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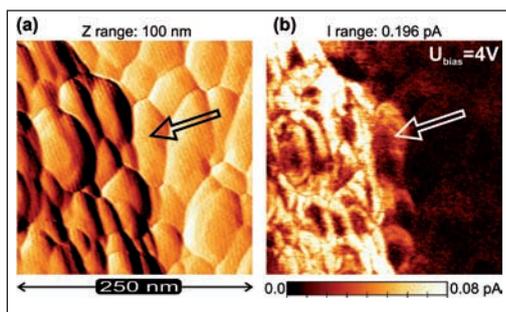


Fig. 3: Small scale C-AFM measurement of the sample B showing (a) topography and (b) local current map measured at  $U_{bias} = 4V$ . The arrows indicate more conductive amorphous column attached to the  $\mu c$ -Si:H grain.

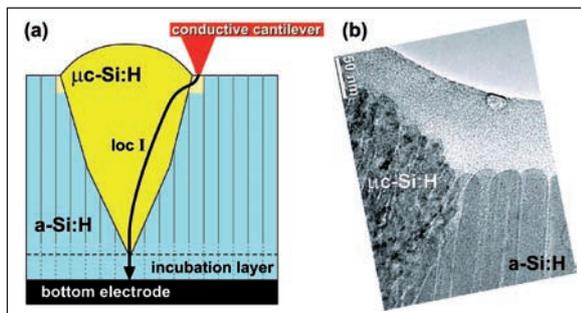


Fig. 4: Sketch of the cross-section of the sample (a) with expected path of the local current explaining the results in Fig. 3. X-TEM image (b) of a small area around the edge of the  $\mu c$ -Si:H grain with nicely visible columns in the a-Si:H phase.

observed in the TEM image in fig. 1(c). It seems that this particular a-Si:H column is in a good electrical contact with the more conductive  $\mu c$ -Si:H grain and allows the local current pass through the sample as shown in fig. 4(a). However, most the other a-Si:H columns in fig. 3 show rather low local current signal, which means, they must be electrically separated (in the lateral direction) from the  $\mu c$ -Si:H grain and also from each other. This could be due to hydrogen concentrated to the a-Si:H column boundaries [4] or simply by spaces between the columns. Both would give similar contrast in the X-TEM image (fig. 4(b)).

### Conclusions

We have observed a nanometer-size columnar structure of a-Si:H phase in the samples prepared from silane and hydrogen at low deposition temperatures. We have correlated the cross-sectional TEM and surface AFM observations of the same sample and identified both conical microcrystalline grains and amorphous columns. We have succeeded in the conductive AFM measurement of intrinsic Si films in ambient conditions utilizing very sensitive current detection. The multi-signal AFM analysis is a powerful technique for the study of local properties as demonstrated also recently [7] by the simultaneous measurement of topography, local current, surface potential and micro-Raman.

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