Exercise

Atomic Force Microscopy observation of \( \text{V}_2\text{O}_5 \) nano-rods prepared by sol-gel method

The aim

This exercise consists in the investigation of the structure of vanadium pentoxide thin films with the help of Atomic Force Microscopy method.

Introduction

\( \text{V}_2\text{O}_5 \) thin films have attracted substantial interest among the transition metal oxide semiconductors due to useful properties such as high chemical stability, electrochemical safety, low cost, easy preparation and relatively low toxicity [1, 2]. Compared to bulk, vanadium oxides with nano and micro-structures appreciably improve the performance in devices for energy storage and sensing due to their distinct physical and chemical properties because of their large surface area and unique morphology. Growth of 1D nano and micro-structures in thin film form is more suitable for device applications than that of other forms, and high surface area metal oxides are usually preferred. \( \text{V}_2\text{O}_5 \) with different morphologies in fine particles and thin film have been prepared by a variety of methods such as temple-assisted growth based on electrodeposition, surfacant/inorganic self-assembly, e-beam sputtering, chemical-vapor deposition and pulsed laser deposition. But above methods usually require template materials, and the growth conditions are hard to control [1]. The sol-gel process offers a suitable synthetic path for creating materials with very unusual properties as compared to traditional solid-state chemical techniques. Thin films can be deposited via dip-coating or spin coating techniques by using the sol-gel method [1, 3]. This method is based on the hydrolysis and condensation of molecular precursor, for instance metal alkoxides or hydroxylated metal ions in aqueous solutions [4].

Preparation of samples

Vanadium pentoxide thin films which will be measured in this exercise, were prepared by the sol-gel method. The starting solution was prepared by mixing vanadium (V) oxytripropoxide in an anhydrous ethyl alcohol as solvent and acetic acid. The coatings were deposited by spin-coating technique at a rate of 100 rps on pre-cleaned quartz glass and silica substrates and dried at 50 °C for 48 h in air atmosphere. Above procedure was repeated three times to obtain films with 600 nm thickness. Next samples were controlled crystallised at 600 °C to obtain nano-rods. The widths of rods range from 1 µm to 4 µm, the lengths range from 5 µm to 15 µm and their thickness range from 100 nm to 500 nm.
**Measurement method**

The rod-like structure of V_{2}O_{5} thin films will be observed with the help of the Nanosurf easyScan 2 AFM system and Nanoeducator system. These systems are an atomic force microscopes that can measure the topography and several other properties of a sample with nanometer resolution. Atomic Force Microscopy allowed electrically conducting and insulating materials to be analyzed.

This microscopy technique work without optical focusing elements. Instead, a small sharp probing tip and the sample surface is so small that atomic-range forces act between them. In an AFM, a tip is attached to the end of a cantilever in order to measure these forces. The force acting on the tip can then be determined by detecting the deflection of this cantilever. The measurement of the cantilever deflection can be used to control the tip-surface distance on an atomic scale. Thus, enormous resolution can be achieved, so that even the atomic arrangement of surfaces can be probed. This measurement is a so-called static operating mode, in which the static deflection of the cantilever is used.

Generally, the forces acting on the tip will cause it to snap onto the sample, which result in an effective, nanometer-range flattening of the tip, and friction and stiction between the tip and the sample. To circumvent the aforementioned problems, the so-called Dynamic force microscopy modes can be used. In these modes, the cantilever vibrates during the operation. In the dynamic modes, changes in the free resonance frequency and the damping of the cantilever vibration caused by the forces between the tip and the cantilever can be measured and used to regulate the tip-sample distance. The main parts of the basic system are the easyScan 2 AFM Scan Head, the AFM Sample Stage and the easyScan 2 Controller with AFM Basic module. Measurements are performed, displayed and evaluated using the SPM Control Software.

**Exercise**

1. Turn on the computer and the power of the Nanosurf easyScan 2 controller.
2. Start the Nanosurf easyScan 2 program.
3. Put the sample on the round Sample Holder lying on AFM Sample Stage with the help of forceps and slip all under the easyScan 2 AFM Scan Head. The sample should be located under the cantilever (red laser light should fall on it).
4. Select the Static Force mode and SICON-A cantilever.
5. **Approaching the sample**: To start measuring, the cantilever tip must come within a fraction of a nanometer of the sample without touching it with too much force. To achieve this, a very careful and sensitive approach of the cantilever is required. Select Side view in the Video panel located in the Info pane to see the cantilever and sample. Select the Acquisition tab and all controls for positioning the cantilever with respect to the sample are located in the Approach group. Click and hold the Advance button until the tip is close enough to the sample (all the time observe the distance between tip and sample in the side view). You should see the shadow of the cantilever if the sample is not reflective, if it is reflective you should stop when you see the sample surface. Now click Approach button to automatic...
final approach. A message "Approach done" should appear. Click **OK** button.

6. The first measurement should start automatically after approach. Click **Imaging** button to see **Topography** and **Deflection Scan forward**. To obtain first quick overall topography image, select the following **Parameters**:
   - **Image size**: 50 µm;
   - **Time/Line**: 1 s;
   - **Points/Line**: 256;
   - **Rotation**: 0;
   - **Setpoint**: 20 nN;
   - **P-Gain**: 10000;
   - **I-Gain**: 1000;
   - **D-Gain**: 0.

7. After first measurement choose optional smaller measurement area on your image by click the **Zoom** button in the **Chart bar**. The mouse pointer becomes pen-shaped when moving over the color map. Click on one corner of the region to be selected using the left mouse button, and keep the button pressed. Drag the mouse to the other corner of the region. The size and the position of the square are shown in the **Tool** results panel of the **Info pane**. Release the mouse button when the size of the square covers approximately one period of the grid. Confirm the selection by double clicking the color map graph using left mouse button. Now the selection is enlarged to the whole display size. You can abort the zoom function by clicking the **Zoom** button again. You can also change the **Parameters** to obtain more precise image. For example increase **Points/Line** to 512 and decrease **Time/Line** to 0.5.

8. To see your image click **Gallery** panel of the **Info pane** and double click on the selected image.

9. Finishing scanning: In the Imaging group of the **Acquisition** tab, click the **Stop** button to stop the measuring. Open the positioning window, **side view** in the **Video** panel. Retract the cantilever to a safe distance from the sample by clicking and holding **Retract** button in the **Approach group** of the **Acquisition** tab, next click the **Home** button and wait for the automatic full withdraw process to finish. Remove the sample holder from the sample stage and sample from the sample holder.

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**References**


