

# Surface Characterization

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## Abstract

This comprehensive study employs multiple surface characterization techniques, including Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray (EDX) Analysis, Optical Profilometry (OP), and Atomic Force Microscopy (AFM), to analyze the surface properties of various materials. SEM and EDX were used to investigate the structural and chemical characteristics of Zirconium Carbide coated with Zirconium dioxide ( $ZrC-ZrO_2$ ) and laser-engraved Copper (Cu). OP was applied to Copper (Cu), Aluminum (Al), glass, and laser-etched samples, providing detailed 3D topographical maps and roughness measurements. AFM was utilized to examine a Fluoride Tin Oxide-coated glass substrate (FTO), highlighting its nanoscale surface features. These methods collectively provided a deep insight into the materials' surface roughness, texture, and morphology, essential for optimizing material performance in various applications.

**Keywords:** *Surface Characterization, Material Analysis, Roughness, Elements*

# 1 Introduction

This comprehensive experiment incorporates Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray (EDX) Analysis, Atomic Force Microscopy (AFM), and Optical Profilometry to provide an in-depth characterization of material surfaces. These advanced techniques allow for the precise analysis of surface topography, structure, and composition, each contributing unique insights into the material properties at micro to nanometer scales.

SEM and EDX represent cornerstone technologies in the field of material science, providing essential insights into the structural and compositional aspects of materials at the micro to nano scale. SEM operates on principles akin to optical microscopes but uses focused beams of electrons instead of light to achieve magnifications up to the sub-nanometer level, significantly surpassing the limitations of optical instruments.

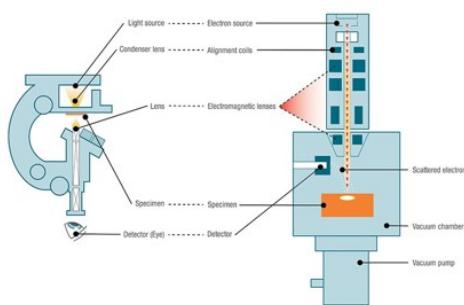


Figure 1: SEM Concept

The operation of SEM is based on the interactions between electron beams and the sample, producing various signals like secondary electrons (SE) and backscattered electrons (BSE), which help in imaging the surface topography and composition. The introduction of EDX into SEM extends its functionality by enabling elemental analysis. EDX works by detecting X-rays emitted from the sample when it is bombarded with electrons; these X-rays have energies characteristic of specific elements, thus allowing for precise compositional analysis. Together, they enable a detailed examination of surface morphology and chemical makeup, crucial for materials science, metallurgy, and semiconductor research.

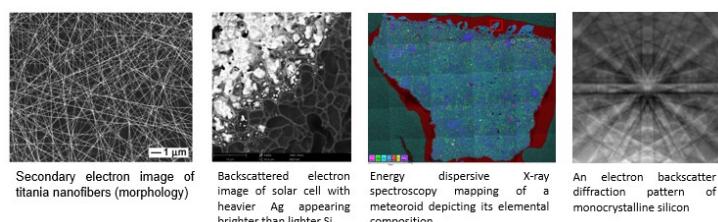


Figure 2: Various Images

OP is a non-contact method that uses light to measure the topography of a surface, providing detailed 3D imaging and roughness measurements. This technique leverages the principles of optical interference, focus variation, confocal microscopy, and other light-based methods to generate accurate topographical data of the sample surface. It is distinguished by its ability to rapidly and accurately measure surface features across a wide range of scales, from nanometers to millimeters, without touching the sample. This makes it particularly valuable for sensitive or soft materials that could be damaged by contact methods. Common implementations include White Light Interferometry (WLI) and Confocal Microscopy, which are used to achieve high-resolution measurements of surface roughness, texture, and form.

The technology operates on the principle of detecting the interference pattern created when light reflected from the sample surface combines with light from a reference beam. This interference pattern is analyzed to extract the surface's profile. Advanced techniques such as Phase Shifting Interferometry (PSI) and Vertical Scanning Interferometry (VSI) are used to enhance the measurement's precision and to cater to different types of surface characteristics.

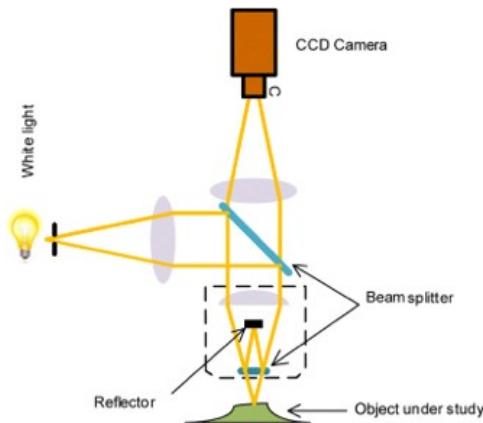


Figure 3: OP Concept

AFM, also known as Scanning Force Microscopy (SFM), is a type of scanning probe microscopy that provides detailed topographical mapping of surfaces at the nanometer scale. This powerful technique utilizes a sharp probe mounted on a flexible cantilever to interact with the surface of a sample. As the probe raster scans across the surface, it records minute force interactions between the tip and the surface, which are translated into electrical signals that can be used to construct a three-dimensional image of the surface.

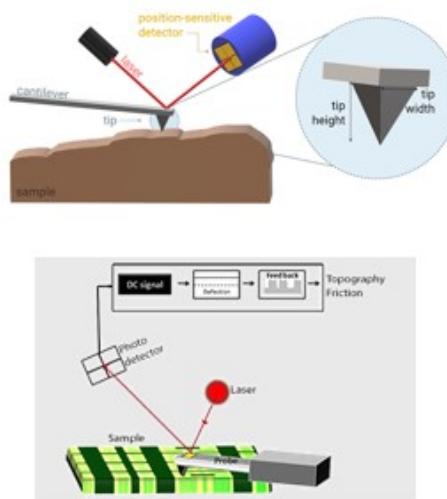


Figure 4: AFM Concept

AFM is distinguished by its remarkable resolution, capable of detecting features at the fraction of a nanometer scale, far surpassing the optical diffraction limit. This high resolution enables the observation of individual atoms and molecules on a variety of surfaces, including polymers, ceramics, composites, and biological samples. Moreover, AFM is versatile in its environmental adaptability, allowing measurements to be conducted in various settings including air and liquid, making it indispensable for studying biological processes in their native environments.

The technique is fundamentally driven by the interaction of forces including Van der Waals forces, electrostatic forces, and capillary forces, among others. These interactions help in not only determining the surface topography but also in measuring the mechanical, magnetic, and electrical properties of the sample surface.

Through its multiple operational modes, such as contact mode, non-contact mode, and tapping mode, AFM provides a comprehensive suite of analysis options, catering to the specific needs of the research or industrial application at hand, from studying the surface roughness to functional properties like phase and modulus.

## **2 Experimental Details**

This section details the procedure for each method and the expected results according to the lab manuals and the relevant equations.

### **2.1 Scanning Electron Microscope & Energy Dispersive X-Ray Spectroscopy**

#### **Equipment and Materials:**

- Tescan Vega 3 Scanning Electron Microscope (SEM) equipped with an Oxford Instruments EDX spectrometer.
- Samples: Copper (Cu) sheets and Zirconium Carbide (ZrC) coated with Zirconium Dioxide (ZrO<sub>2</sub>).
- Standard SEM accessories including secondary electron (SE) and backscattered electron (BSE) detectors.

#### **Procedure:**

##### **1. Sample Preparation:**

- Clean the surfaces of the copper sheets and ZrC-ZrO<sub>2</sub> coated samples to ensure they are free from contaminants.
- Mount the samples on SEM stubs using conductive carbon tape to ensure electrical conductivity and stability during imaging.

##### **2. SEM Imaging:**

- Turn on the Tescan Vega 3 SEM, check for proper calibration, and ensure the vacuum level is suitable for high-resolution operation.
- Place the sample in the SEM chamber, adjust the working distance and focus using the stage controls for optimal image quality.
- Set the SEM to operate at an appropriate accelerating voltage, typically around 20 kV for metals and coated materials.
- Acquire images using:
  - Secondary electron (SE) detector for topography and surface details.
  - Backscattered electron (BSE) detector for compositional contrast.

##### **3. EDX Spectroscopy:**

- Use the integrated EDX spectrometer for elemental analysis of identified areas of interest

within the SEM images.

- Acquire and analyze EDX spectra to assess the elemental composition, focusing on areas such as the ZrC-ZrO<sub>2</sub> interface.
- Quantify the elemental distribution to evaluate the coating uniformity and integrity.

#### **4. Data Analysis:**

- Analyze SEM images to identify surface features like grain boundaries, defects, and coating morphology. Done through Oxford Instruments software.
- Correlate EDX data with SEM findings to understand the elemental variations and identify any anomalies or processing issues.

#### **5. Safety and Maintenance:**

- Follow all safety protocols during SEM operation, including wearing appropriate personal protective equipment.
- Keep the SEM operational environment clean and follow manufacturer guidelines for maintenance and shutdown procedures.

## 2.2 Optical Profilometry

### Equipment and Materials:

- Filmetric Profilm3D Optical Profiler.
- Samples: Aluminum (Al), Copper (Cu), Glass, a sample with laser etching, and a step height standard for calibration.

### Procedure:

#### 1. Sample Preparation:

- Clean each sample thoroughly to remove any debris or contaminants that might interfere with the optical measurements.
- Securely mount the samples on the stage of the optical profiler, ensuring that the step height standard is accurately positioned to validate measurement accuracy.

#### 2. Optical Profilometry Setup:

- Initialize the Filmetric Profilm3D Optical Profiler and perform a system calibration using the step height standard to ensure accurate height measurements.
- Configure the profiler settings appropriate for each type of sample, adjusting for specific requirements such as resolution for detailed features like laser etchings.

#### 3. Data Acquisition:

- Proceed with scanning each sample using the optical profiler. The device employs white light interferometry to create detailed 3D topographical maps from the interference patterns of light reflected off the sample surface.
- Adjust the focus and measurement parameters specifically for each material type, taking into account factors like the reflectivity and surface texture.

#### 4. Data Analysis:

- Analyze the collected data using the profiler's software to produce maps of surface topography and quantify roughness and step heights as needed.
- Pay particular attention to detailed features such as the depth and uniformity of laser etchings, utilizing the software's enhanced analysis tools.

#### 5. Calibration and Maintenance:

- Regularly recalibrate the optical profiler using the step height standard to maintain measurement precision.

- Keep the optical components clean and free from dust to prevent any interference with the accuracy of the measurements.

## **2.3 Atomic Force Microscopy**

### **Equipment and Materials:**

- hpAFM setup: Includes the cantilever assembly, probe holder, scanner, laser and photodetector, feedback system, and computer with control software.
- Sample: Fluoride Tin Oxide (FTO) coated glass substrate.

### **Procedure:**

#### **1. Sample Preparation:**

- Thoroughly clean the FTO coated glass substrate to ensure the surface is free from contaminants and residues.
- Mount the sample on the sample stage of the AFM setup, ensuring it is stable and aligned properly for scanning.

#### **2. Cantilever Calibration:**

- Select an appropriate cantilever for the expected sample properties (considering factors like spring constant and tip radius).
- Calibrate the cantilever by determining its spring constant and sensitivity using the thermal tuning method provided by the AFM software.

#### **3. AFM Scanning:**

- Configure the AFM to operate in the desired mode. For surface topography and roughness measurements, non-contact or tapping mode is generally preferred to minimize tip-sample interaction.
- Set scanning parameters such as scan size, scan rate, and setpoint.
- Begin the scanning process, ensuring that the laser is correctly aligned on the cantilever and the photodetector is capturing the cantilever's motion accurately.

#### **4. Data Acquisition and Analysis:**

- Monitor the AFM images in real-time to ensure quality data collection.
- Use the AFM software to analyze the data post-acquisition. Key analyses might include measuring surface roughness parameters, feature dimensions, and assessing the uniformity of the FTO coating.

#### **5. Post-Experiment Procedures:**

- Carefully remove the sample from the AFM setup.

- Conduct routine maintenance on the AFM to ensure it is clean and functional for future experiments.

### 3 Results and Discussion

The following sections contain our results for each method that includes tables and graphs. This is accompanied by a discussion that includes interpretations of the results.

#### 3.1 Scanning Electron Microscope (SEM) & Energy Dispersive X-Ray Spectroscopy (EDX)

We used the SEM to image the surfaces of two samples: Zirconium Carbide coated with Zirconium Oxide ( $\text{ZrC-ZrO}_2$ ), and laser engraved Copper (Cu). With the images we performed EDX analysis on them to determine the surface composition.

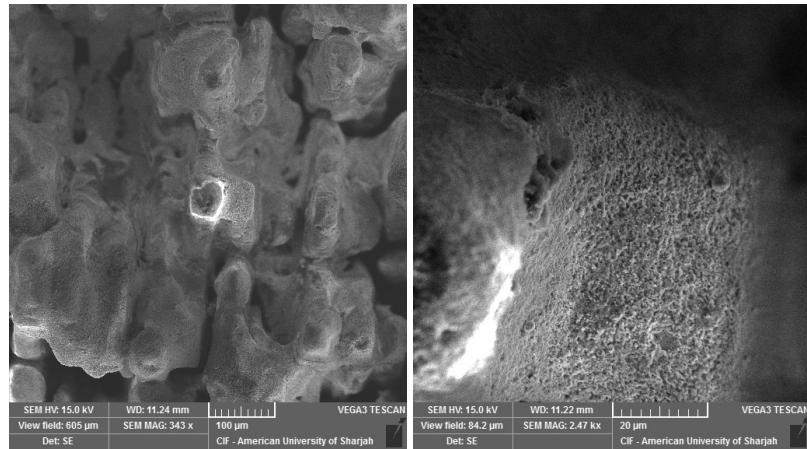


Figure 5: SEM ZrC 1

Figure 6: SEM ZrC 2

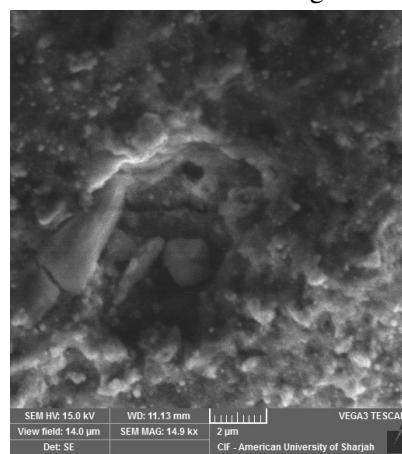


Figure 7: SEM ZrC 3

The first image shows the fine structure of the surface, seen by the contrast revealing variations in depth. The second image shows the coating of  $\text{ZrO}_2$ .

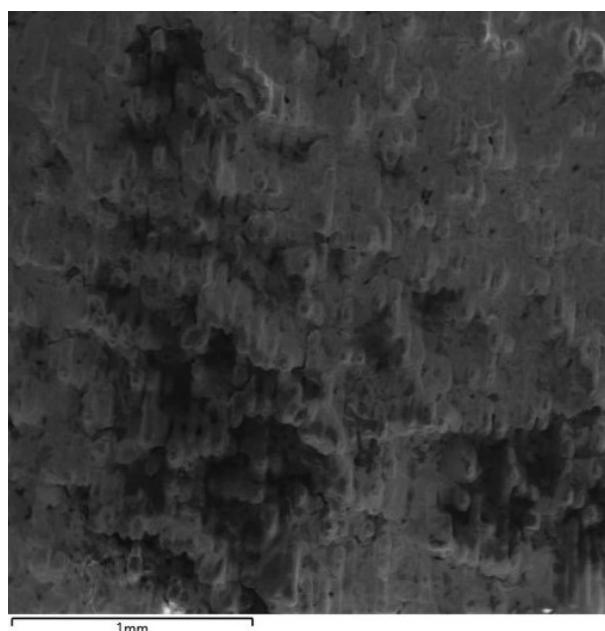


Figure 8: ZrC-ZrO<sub>2</sub> EDX Map Image

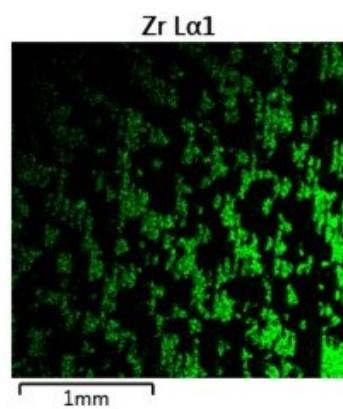


Figure 9: Zr presence in ZrC-ZrO<sub>2</sub> sample

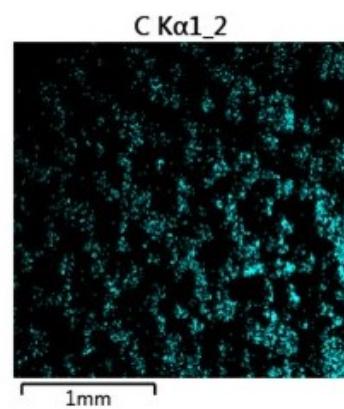


Figure 10: C presence in ZrC-ZrO<sub>2</sub> sample

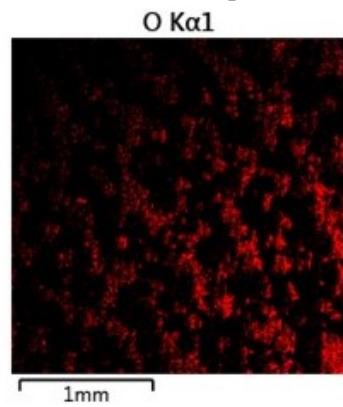


Figure 11: O presence in ZrC-ZrO<sub>2</sub> sample

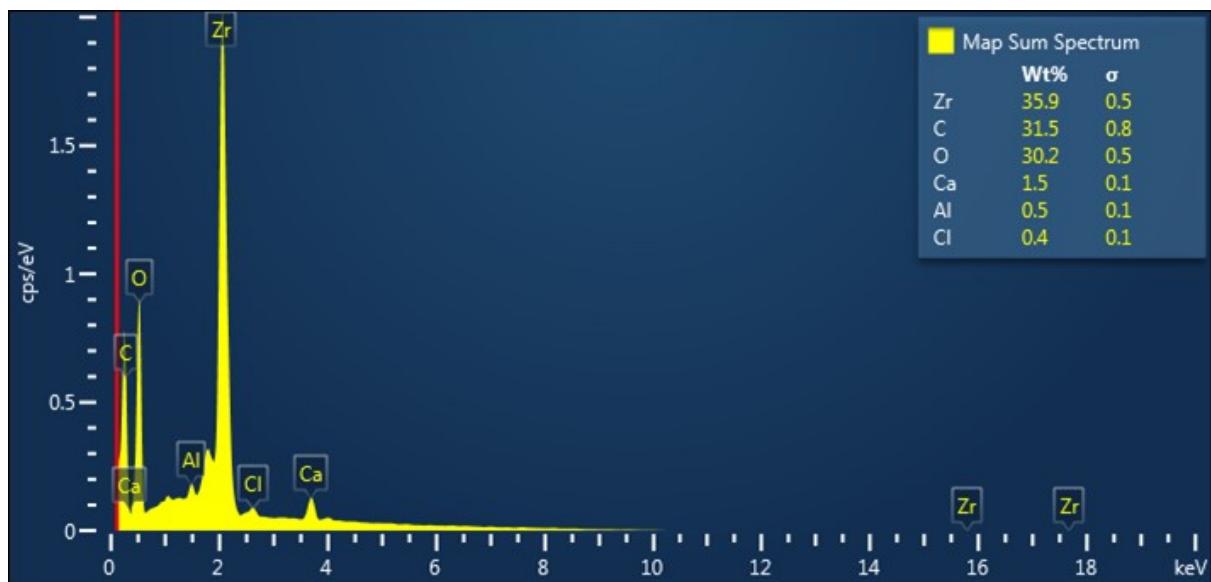


Figure 12: ZrC-ZrO<sub>2</sub> EDX Map Sum Spectrum

Table 1: ZrC-ZrO<sub>2</sub> EDX Map Sum Spectrum Atomic Weight

Map Sum Spectrum	Atomic %
C	52.73
O	37.98
Al	0.36
Cl	0.24
Ca	0.78
Zr	7.91
Total	100

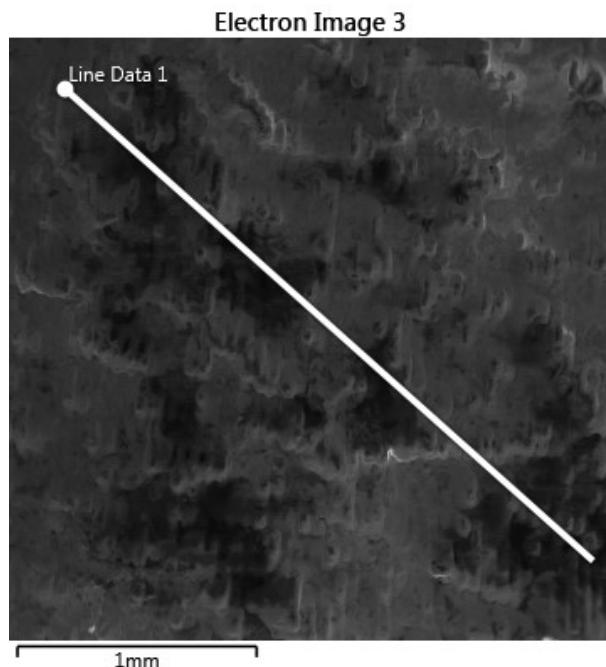


Figure 13: ZrC-ZrO<sub>2</sub> EDX Line Image 1



Figure 14: ZrC-ZrO<sub>2</sub> EDX Line Image 2

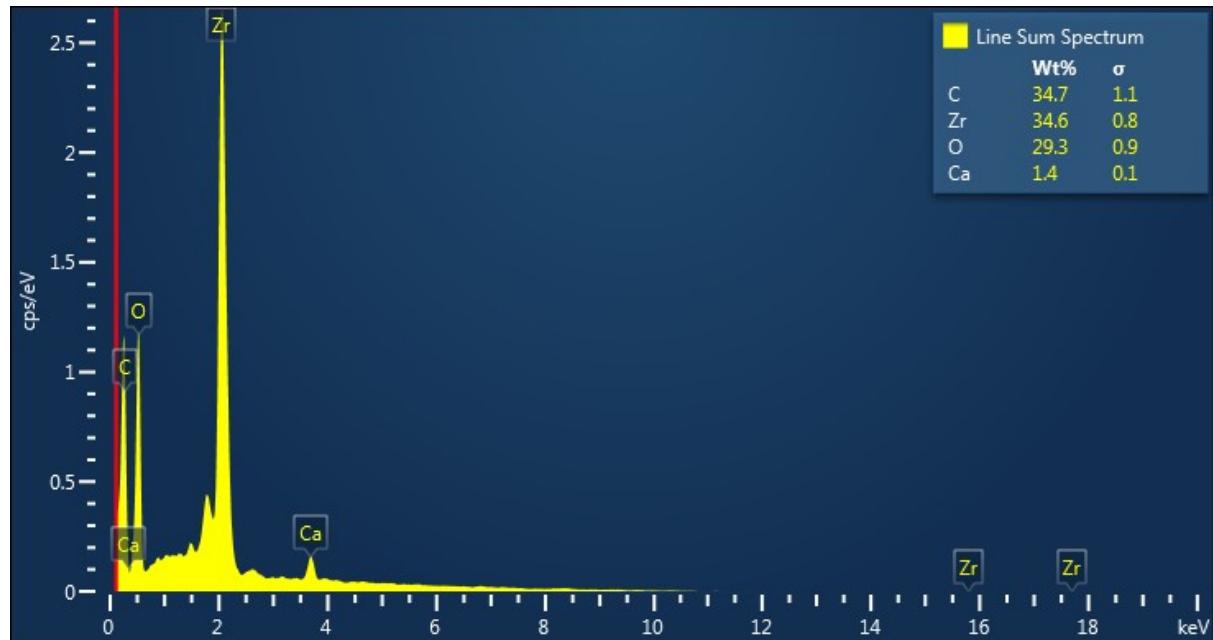


Figure 15: ZrC-ZrO<sub>2</sub> EDX Line Sum Spectrum

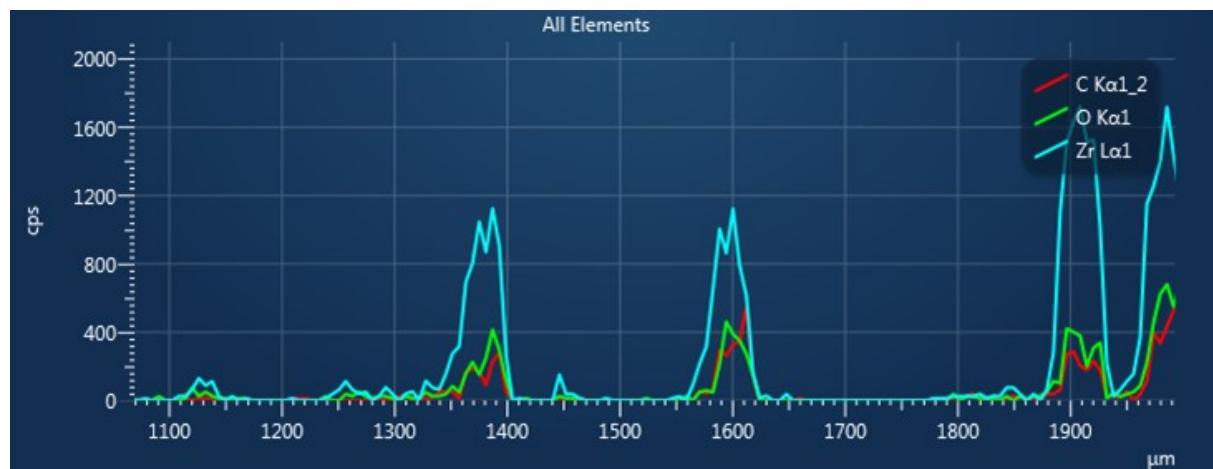


Figure 16: ZrC-ZrO<sub>2</sub> EDX Line Counts Spectrum

Table 2: ZrC-ZrO<sub>2</sub> EDX Line Sum Spectrum Atomic Weight

Line Sum Spectrum	Atomic %
C	56.29
O	35.65
Ca	0.67
Zr	7.38
Total	100

Electron Image 4



Figure 17: ZrC-ZrO<sub>2</sub> EDX Line Image 1

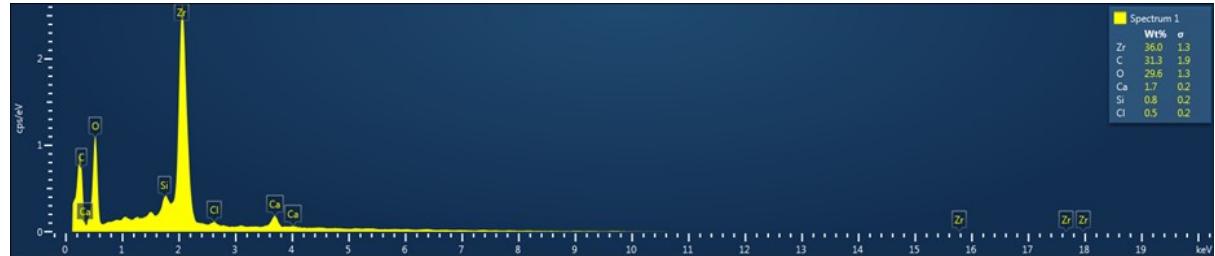


Figure 18: ZrC-ZrO<sub>2</sub> EDX Line Sum Spectrum

Table 3: ZrC-ZrO<sub>2</sub> EDX Point Sum Spectrum Atomic Weight

Point Sum Spectrum	Atomic %
C	52.79
O	37.47
Si	0.59
Cl	0.29
Ca	0.87
Zr	7.99
Total	100

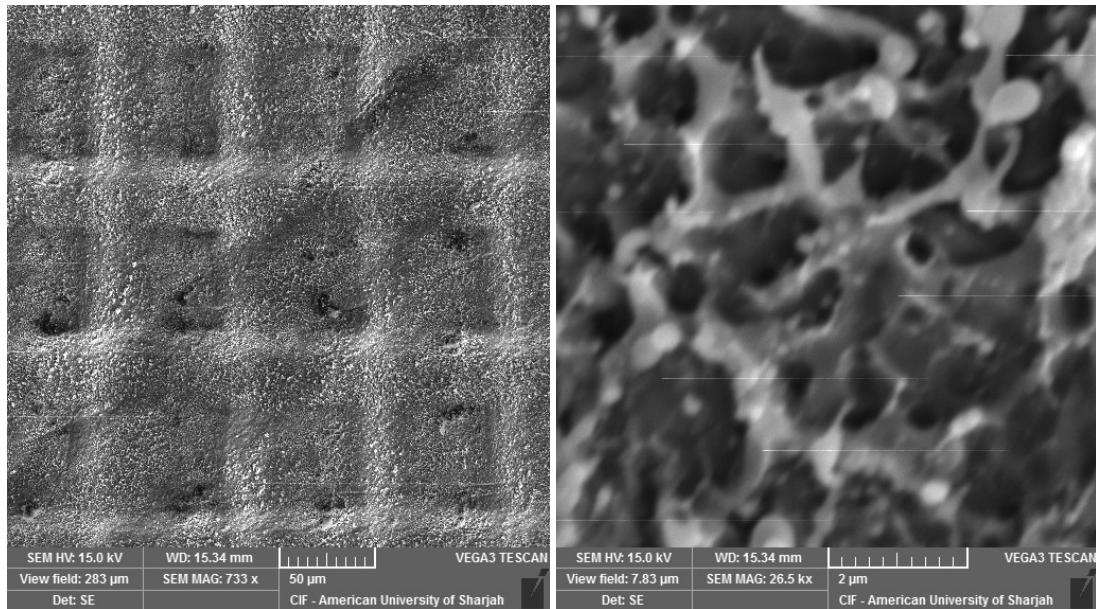


Figure 19: Laser Etched Cu SEM 1

Figure 20: Laser Etched Cu SEM 2

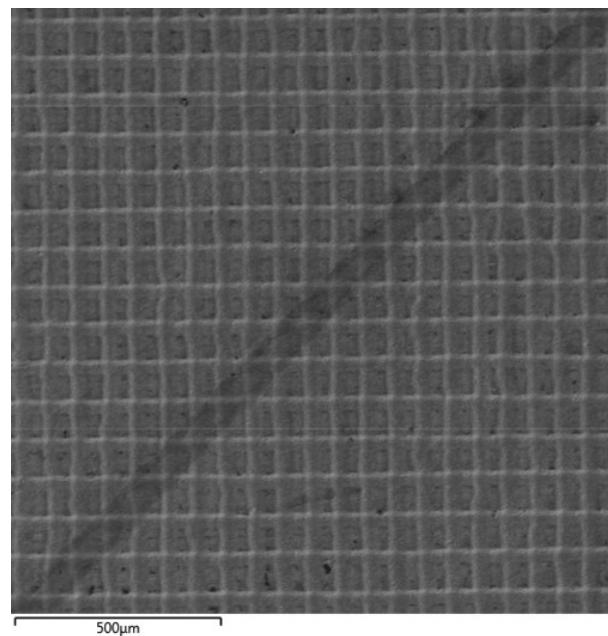


Figure 21: Cu EDX Map Image

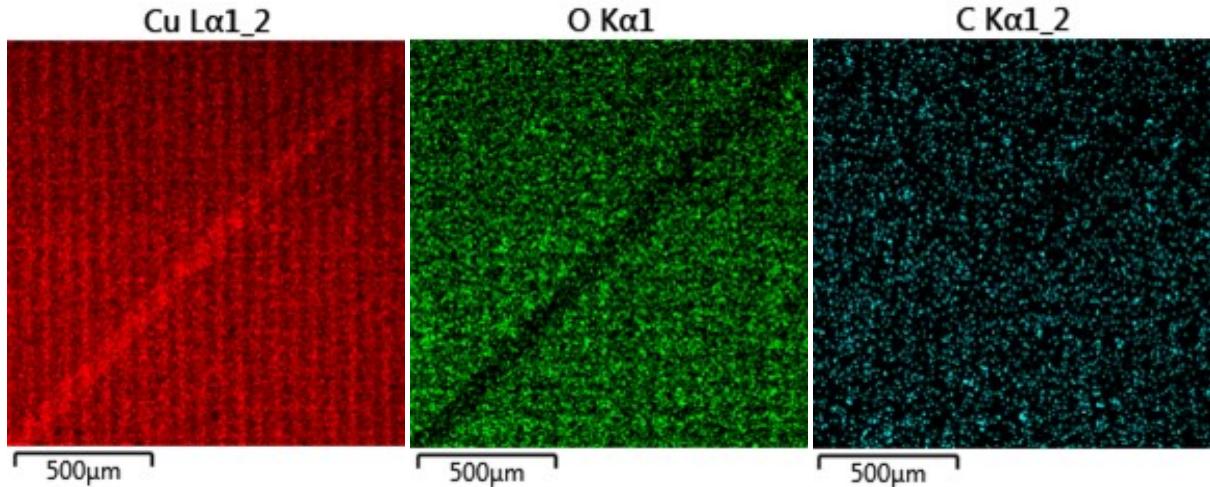


Figure 22: Cu presence in Cu sample  
Figure 23: O presence in Cu sample  
Figure 24: C presence in Cu sample

The SEM images showcase the microstructure of the ZrC under the ZrO<sub>2</sub> coating. The expected property is a uniform and continuous coating, and it is apparent from the smooth and homogeneous surface. Variations in depth and texture are observed which indicates areas where the coating might be too thin or non-uniform, potentially exposing the ZrC substrate. For the Cu sample, the SEM images show the quality of the laser etching. We observe well-defined edges and consistent depth of the engraving, which are crucial for applications like microcircuit fabrication or decorative finishes. EDX analysis helps confirm the chemical makeup of the ZrC-ZrO<sub>2</sub> interface. Expected properties include a clear split of ZrO<sub>2</sub> and ZrC layers with distinct peaks for Zr and O in the ZrO<sub>2</sub> layer and C and Zr in the ZrC layer. The presence of unexpected elements like Al and Ca could suggest contamination or secondary phases that could affect the material's properties like electrical conductivity or corrosion resistance. Also on the Cu sample, we can see some oxidation from the O atoms and some C atoms, potentially from coating.

### 3.2 Optical Profilometry (OP)

We used the Profilometer to image the surfaces of four samples: Copper (Cu), typical sample with laser etching, Glass ( $\text{SiO}_2$ ), Aluminum (Al). A step height provided by the manufacturer was used to calibrate the Profilometer.

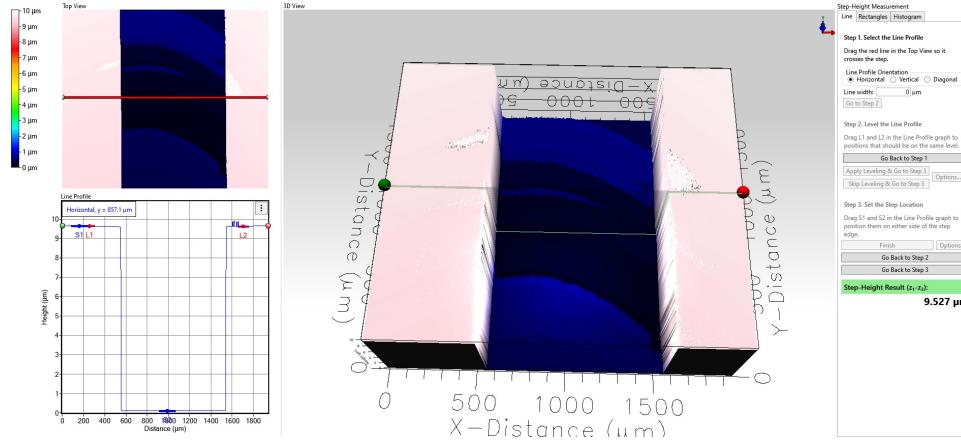


Figure 25: Step Height

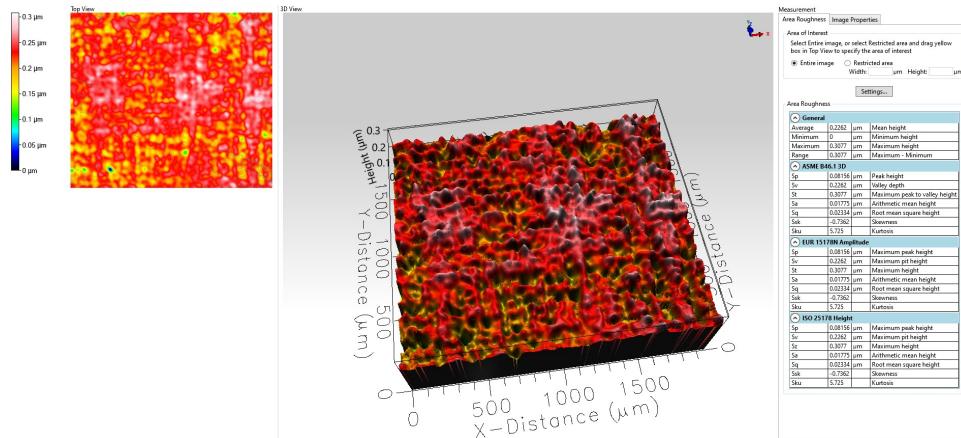


Figure 26: Copper

The step height standard was used for calibration, ensuring the accuracy of height measurements across these samples. The profiler provided detailed 3D topographical maps that highlighted features such as the surface roughness of Al and Cu, the smoothness of Glass, and the details of the laser etching. It's clear that post processing is required due to the nature of the apparatus. The metals Cu and Al exhibit uniform and smooth surfaces even after manufacturing with only minimal surface roughness. For glass, we can see how flat the surface is before processing, and even after processing the variations are in the range of  $0.01 \mu\text{m}$ . The laser etched sample highlights the precision and repeatability of the applied etching process, as evident by the consistent depth and apparent pattern.

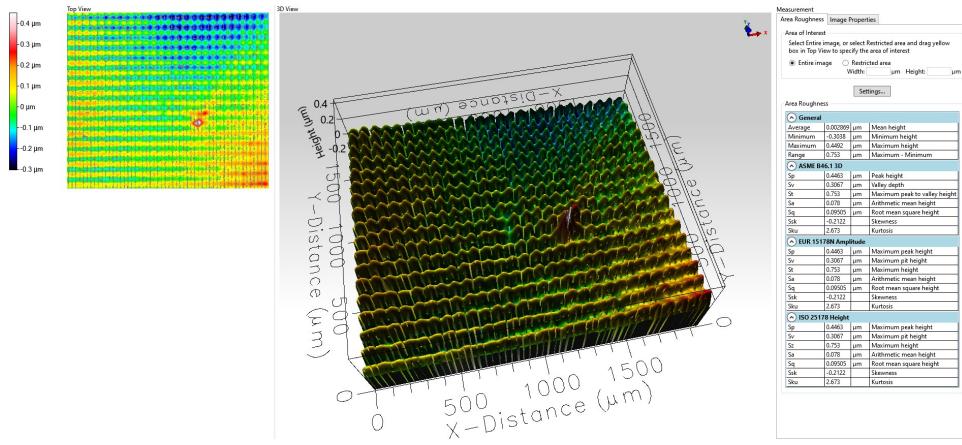


Figure 27: Sample With Laser Etching

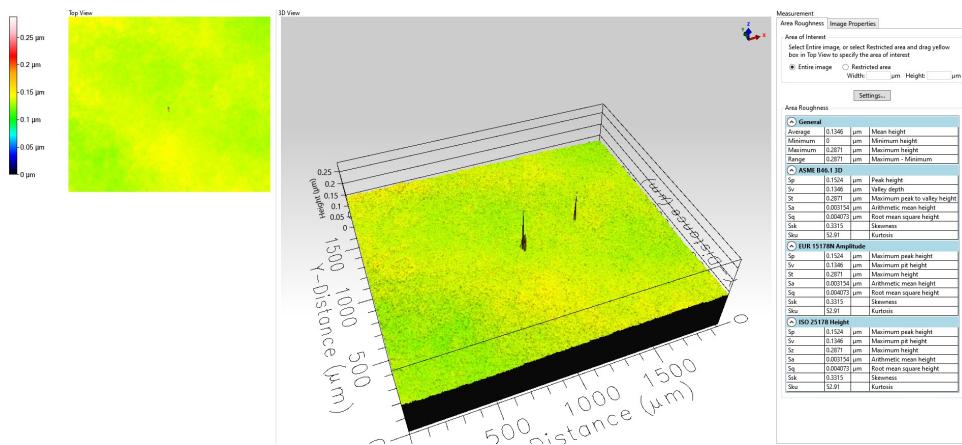


Figure 28: Glass Surface Without Image Processing

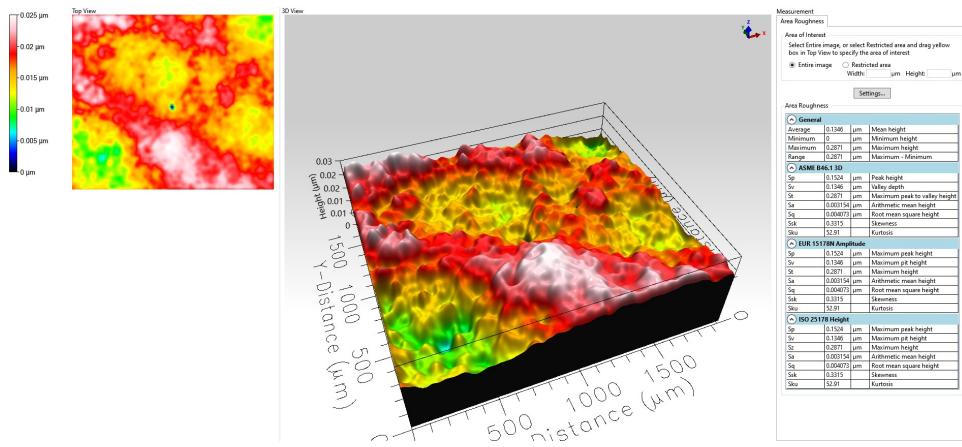


Figure 29: Glass Surface With Image Processing

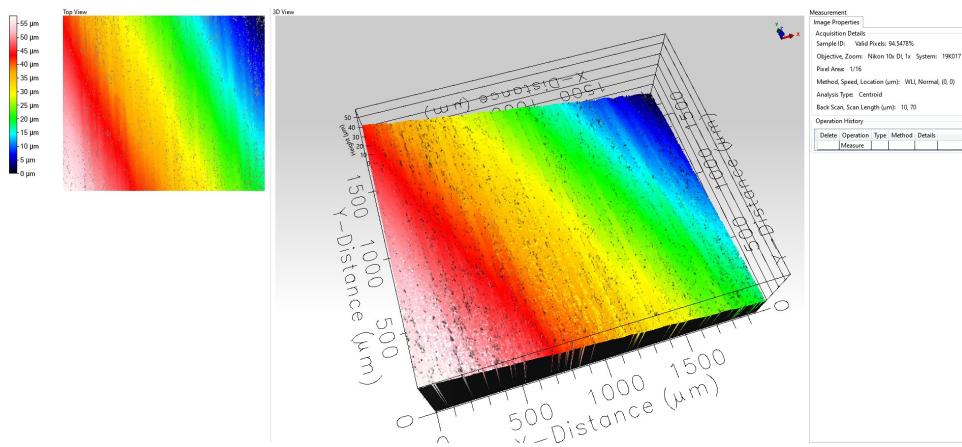


Figure 30: Aluminum Surface Before Flattening

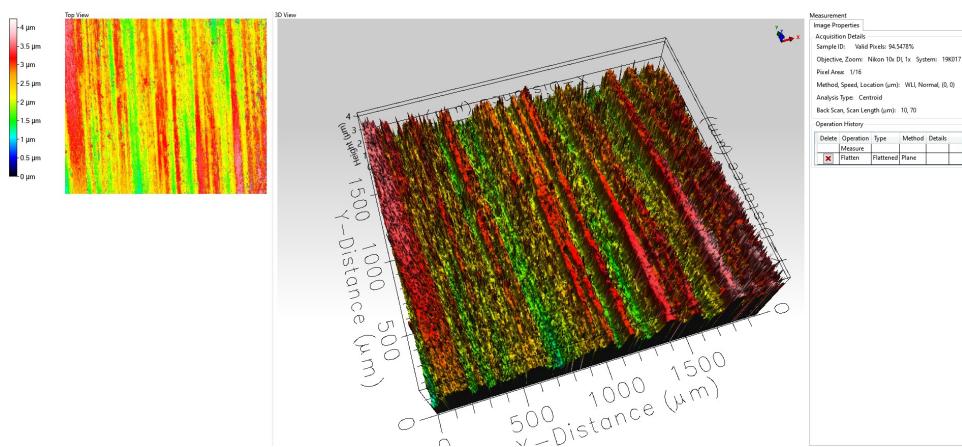


Figure 31: Aluminum Surface After Flattening

### 3.3 Atomic Force Microscopy (AFM)

We used AFM to image a Fluoride Tin oxide-coated glass substrate (FTO).

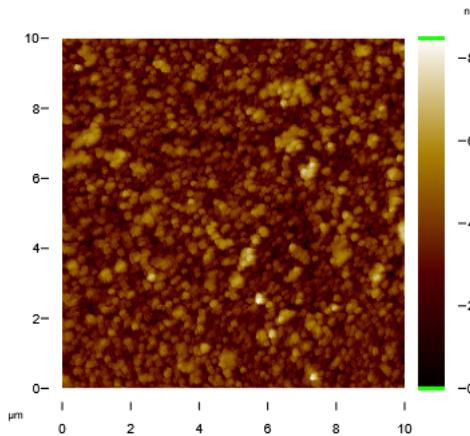


Figure 32: FTO AFM 2D

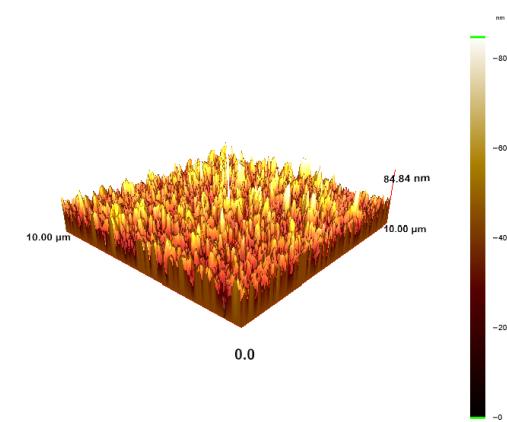


Figure 33: FTO AFM 3D

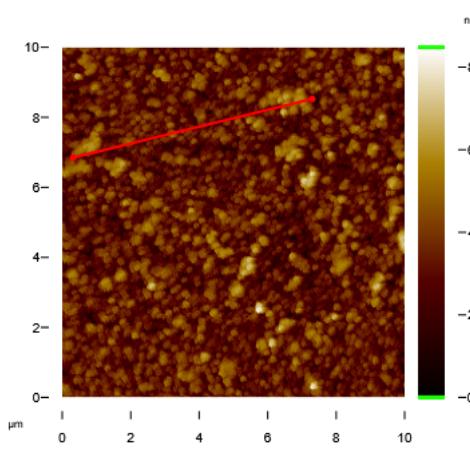


Figure 34: FTO AFM Line Image 1

Roughness Parameters	
Coefficient	Value
Average (Ra)	8.46 nm
Root Mean Square (Rq)	10.30 nm
Skewness (Rsk)	0.369
Kurtosis (Rku)	2.505
Maximum (Rp)	61.82 nm
Minimum (Rv)	10.51 nm
Peak To Peak (Rt)	51.31 nm
Ten Point Height (Rz)	42.70 nm

Figure 35: FTO AFM Line Image 1 Roughness

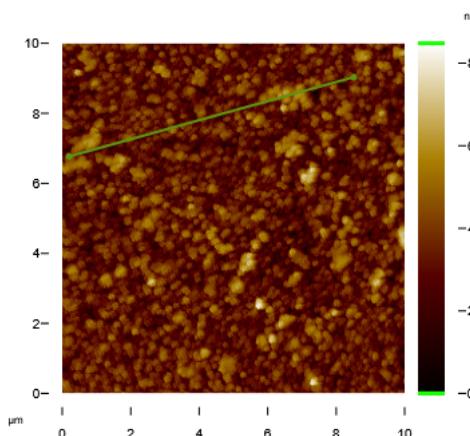


Figure 36: FTO AFM Line Image 2

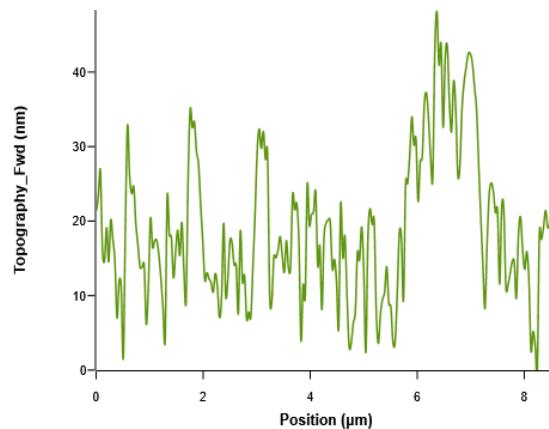


Figure 37: FTO AFM Line Image 2 Topography

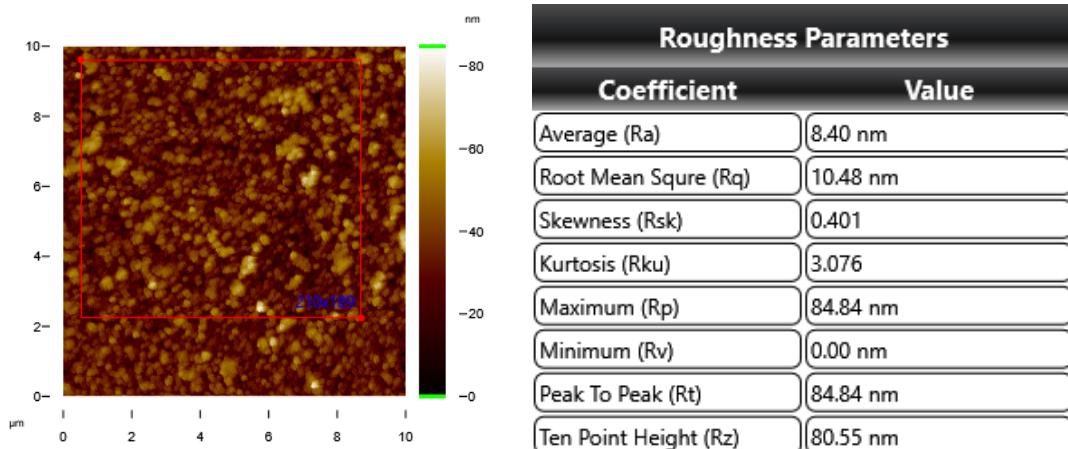


Figure 38: FTO AFM Area Image

Figure 39: FTO AFM Area Image Roughness

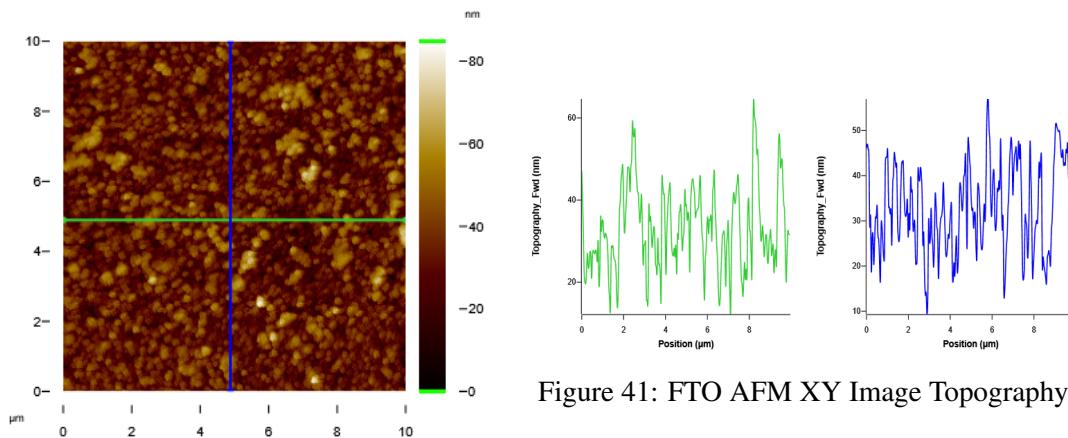


Figure 40: FTO AFM XY Image

Figure 41: FTO AFM XY Image Topography

It is clear that FTO has a smooth surface with minimal grain boundaries visible at the nanoscale.

The variations range between 10 nm and 60 nm.

## 4 Applications

- **Materials Science:**
  - Characterizing the surface roughness, texture, and morphology of materials.
  - Analyzing material defects such as cracks, corrosion, and other surface irregularities.
  - Investigating thin films, coatings, and surface treatments.
- **Semiconductor Industry:**
  - Inspecting semiconductor wafers for defects.
  - Measuring layer thickness and uniformity in multi-layer structures.
  - Surface roughness and topography assessment for quality control.
- **Biomedical Applications:**
  - Imaging and analyzing biological samples like cells and tissues at high resolution.
  - Characterizing the surface properties of biomaterials for implants and prosthetics.
- **Manufacturing and Engineering:**
  - Assessing wear and tear on mechanical components.
  - Examining the quality of machined surfaces to ensure compliance with specifications.

## **5 Conclusion**

The integration of SEM, EDX, OP, and AFM in this experiment has effectively shown the surface characteristics of various materials. SEM and EDX analyses revealed the microstructural details and elemental composition of ZrC-ZrO<sub>2</sub> and Cu, confirming the uniformity of coatings and the quality of laser etchings. OP provided quantitative data on surface roughness and topography, particularly highlighting the precision of surface treatments and manufacturing quality. AFM offered unparalleled insights into the nanoscale surface features of FTO-coated glass, showing minimal grain boundaries and surface roughness suitable for advanced optical applications. This study demonstrates the powerful synergy of combining multiple surface characterization techniques to obtain a comprehensive understanding of material surfaces, which is vital for the advancement of materials science and engineering.

## **6 References**

- Lab manuals