Facilitation Centre for Industrial Plasma Technologies
Institute for Plasma Research

Plasma Processing Update

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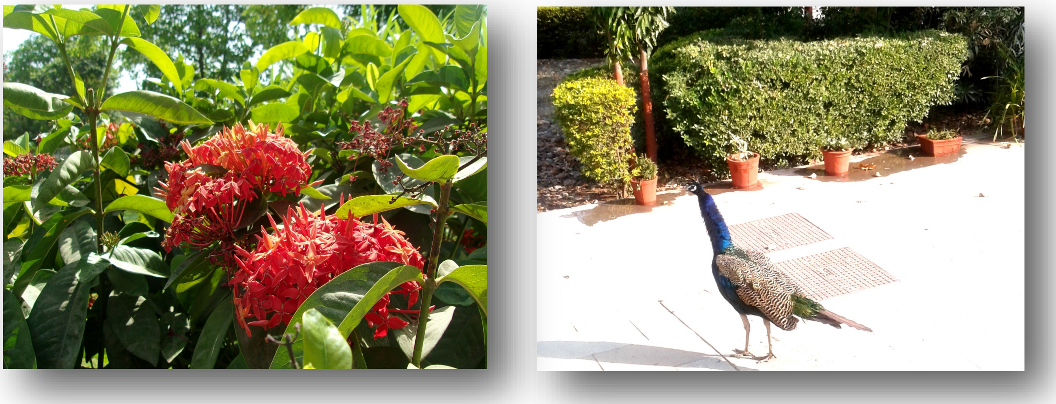

Flora and Fauna at FCIPT Campus

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Important Highlights

- **Inline treatment of textiles to improve functionality.**
- **Treatment @ 20-40 meters per minute**
- **System can be tuned for a wide variety of applications.**

Atmospheric pressure plasma system for Textiles

-Progress Report

Atmospheric Plasma Division (APD) of Institute for Plasma Research is working on a project funded by DST, New Delhi and MANTRA, Surat for developing a plasma treatment system to treat textile using atmospheric pressure DBD (Dielectric Barrier Discharge) plasma at the speed of nearly 20 to 40 meters per minute. In this system, the plasma discharge will take place between the air gaps (1.2 mm) of 72 pairs of cylindrical electrodes each of 2.5 meter width. This is being implemented to meet the requirement for improvement of functionality various kinds of textiles including technical textiles. Plasma generation has been successfully tested for a continuous period of 4 hours. In the coming months, 72 such mesh electrodes will be installed along with their mechanical switching mechanisms. The system will be installed and commissioned at MANTRA, Surat. A visit by the team from MANTRA, Surat to review the progress was held in March 2018.

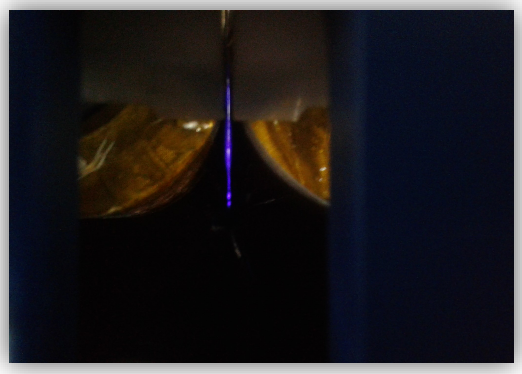

Figure 1: Atmospheric Pressure plasma formation between DBD rollers

From Left to Right : Dr. S.K.Basu, Dr. S.K. Nema, Mr. Vishal Jain, Shri. R.S. Bachkaniwala, Mr. Adam Sanghariat

Paschen's Curve and Striation System for teaching labs

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Important Highlights

- **Highly compact and light weighted**
- **Useful for teaching laboratories and research purpose**
- **Paschen's Curve of different gases can be studied.**

FCIPT, IPR has in the past developed five experimental setups (teaching -aids) for demonstration of plasma physics concepts to M.Sc. students based on UGC guidelines. Currently, Plasma physics is being taught as an elective subject with focus only on theoretical aspects in most of the Indian universities. Students can get hands on experience in working with Plasma using these setups which will help popularize Plasma science and also motivate students to take up Plasma science as a career.

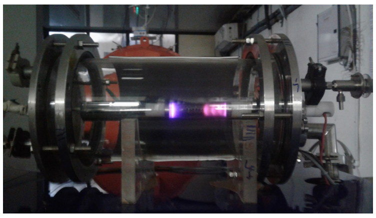


Figure 2: Striations inside glass chamber

On of the experiments includes Paschen's law that relates the voltage applied across the electrodes with the pressure of the gas contained between the electrodes and the distance between electrodes. This can be used to understand the basic discharge mechanism for creating Plasma.

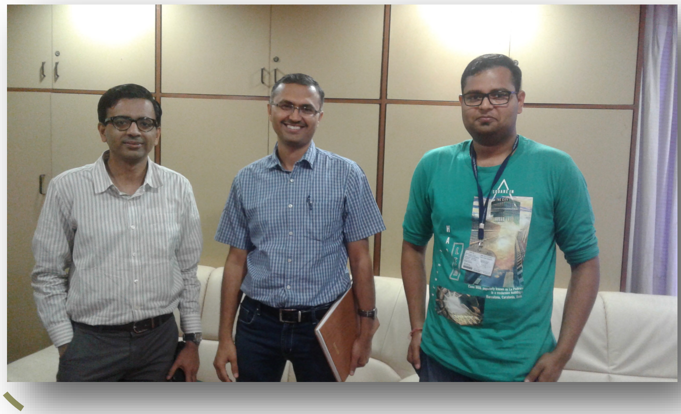
Figure 3: Paschen's Curve and Striations System

We have developed a new system for Centre of Excellence in Basic Sciences, Mumbai (UM-DAE CEBS) that is highly compact and light weight.

Our setup involves a glass tube contained within a glass chamber . The glass chamber is sealed by Stainless Steel (SS) flanges that sit on the top of O-Rings. The inner glass tube houses a cathode and an anode. The anode and cathode are connected to a fixed voltage output power supply with a resistor in series. Thus a single such system can be used to demonstrate both Paschen's law and striations.

Now, if we apply voltage between cathode and anode, an electric field is established between them. As we increase the voltage at fixed pressure and fixed inter electrode distance, a point is reached where Townsend avalanche breakdown of gas happens and the plasma is formed. In our case the gas is air which contains nitrogen, oxygen, carbon dioxide, argon, moisture and some other gases in minor percentage. We also can use different gases like Argon, Helium, Oxygen, Nitrogen and study their Paschen's Curve.

This system will be transferred to the CEBS campus in Mumbai for teaching labs amongst undergraduate and graduate students. Dr. Brijesh Prithvi (faculty at CEBS) recently visited us to see the final system and to discuss the details of transfer and installation of the system at CEBS.



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Important Highlights

- **Advanced characterization equipment**
- **300 kV acceleration voltage**
- **EDX, STEM, EELS attachments**
- **Imaging, Crystallography, Chemical composition**

300 kV TEM facility at FCIPT campus, IPR

A microscope may be described as an instrument that can magnify things that are too small to see with the naked eye. The smallest distance between two points/lines that human eyes can resolve is about 0.1 to 0.2 mm (100 to 200 µm). This distance is generally called as “resolution”. The “useful magnification” of a microscope is governed by its resolution. So, technically, any instrument that can help in revealing details finer than 0.1 mm, by magnifying them, can be described as a microscope.

Based on the type of illumination, microscopes can be divided into three major categories viz.

- Light Microscopes (Light)
- Confocal Microscopes (Laser)
- Electron Microscopes (Electrons)

Electron microscopes use a beam of electrons as a source of illumination and very high resolutions can be achieved as the wavelength of the illuminating radiation is very small. There are two types of electron microscopes viz.

- Scanning Electron Microscope (SEM)
- Transmission Electron Microscope (TEM)

The basic difference between SEM and TEM is that the SEM can provide topographical and chemical information of the samples’ surface only (with best possible depth of field), where as a TEM can also provide the bulk information, beyond the surface. The imaging and analysis in SEM depends on the back-scattered & secondary electrons, and x-rays, while in the case of a TEM it is transmitted electrons, and x-rays. Electron microscopes in general are known for their capabilities in achieving very high magnifications with high resolution. These microscopes find applications in both materials science and in biological sciences. In this article, we discuss about the TEM that is installed & commissioned at FCIPT campus, IPR.

As already stated, a TEM uses a highly energetic beam of electrons that are allowed to transmit through the sample to be analyzed. These transmitted electrons, carrying the information of the sample, are projected onto the fluorescent screen – where the image is formed – using various electro-magnetic lenses. The interaction of these electrons with the ‘sample matter’ contributes to the image contrast.

When the electrons pass through the sample, due to their interaction with the 'sample matter', they may either be scattered by a variety of processes or they may remain unaffected and just pass through. While the incident beam of electrons is highly uniform both in their energy and phase, the transmitted beam will have a non-uniform distribution that contains all the structural and chemical information of the sample. This non-uniform distribution of electrons is displayed in a TEM in two different ways. The *angular distribution* of electrons, due to scattering, can be visualized in the form of diffraction patterns that give crystallographic information of the sample being analysed; whereas, the *spatial distribution* of the scattered electrons is observed as contrast in TEM images of the sample. Therefore, whatever the information that we obtain out of a sample in a TEM can be attributed to some or the other form of electron scattering. The outcome of a TEM can be divided into three major categories:

- Imaging (high magnification with high resolution)
- Crystallographic information (such as crystal structure identification, symmetry and space group information etc.) of even nano features
- Chemical/elemental information (qualitative and quantitative) of even nano features

Though TEM can provide very rich information of the sample being analysed, it requires a tremendous effort to prepare the sample. As it is described earlier, the image/contrast formation in a TEM is based on the non uniform distribution of the scattered electrons after they are transmitted through the sample. However, if they experience very many scattering events before they exit the sample, the information they are carrying cannot be attributed to one particular feature of the sample. Therefore an important objective is to have less number of scattering events by the electrons inside the sample being analyzed. This can be achieved by having high energy electrons and by having very low sample thickness. In general, for simple imaging purpose, a TEM requires a sample of 3 mm diameter with a thickness less than 200 nm. For analytical electron microscopy, the thickness of the sample should be even less i.e. 50-100 nm.

TEM available at FCIPT, IPR

The TEM that is commissioned at FCIPT campus of IPR is a 300 kV TEM equipped with a Field Emission Gun (FEG) based electron source. Photograph of the TEM instrument is shown in figure 1.

Major specifications of the TEM are given below:

- Make & Model : FEI make, Tecnai G2 F30 model
- Electron Source : Field Emission Gun
- Accelerating Voltage : 50 kV – 300 kV
- Point resolution : 0.2 nm
- Line resolution : 0.102 nm
- Additional detectors : SDD detector (for EDXS)
  STEM – HAADF detector
  EFTEM/EELS

Figure 4: Photograph of the TEM instrument

Typical results of TEM

At FCIPT campus, TEM is being effectively used in nano-technology and thin film development technologies. A few of the images are shown below for better understanding.

Figure 2 shows the high resolution image of a typical nano-particle. The size of the particle is of the order of 20 nm and the atomic planes are clearly seen. This clearly manifests the ability of TEM to obtain images at very high magnification with very high resolution.

Figure 3 shows the TEM images of Silicon Carbide (SiC) particles, synthesized at FCIPT using a thermal plasma arc. All the three different images shown are of SiC synthesized at different operating parameters. Feedback from the TEM has helped to identify and optimize the process parameters for obtaining a particular sized and shaped nano particles. Figure 3 (d) shows the EDX spectrum of these nano particles confirming their chemical composition.

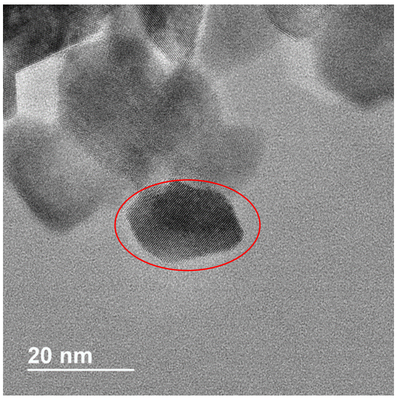

Figure 5: High resolution TEM image of a typical nano-particle

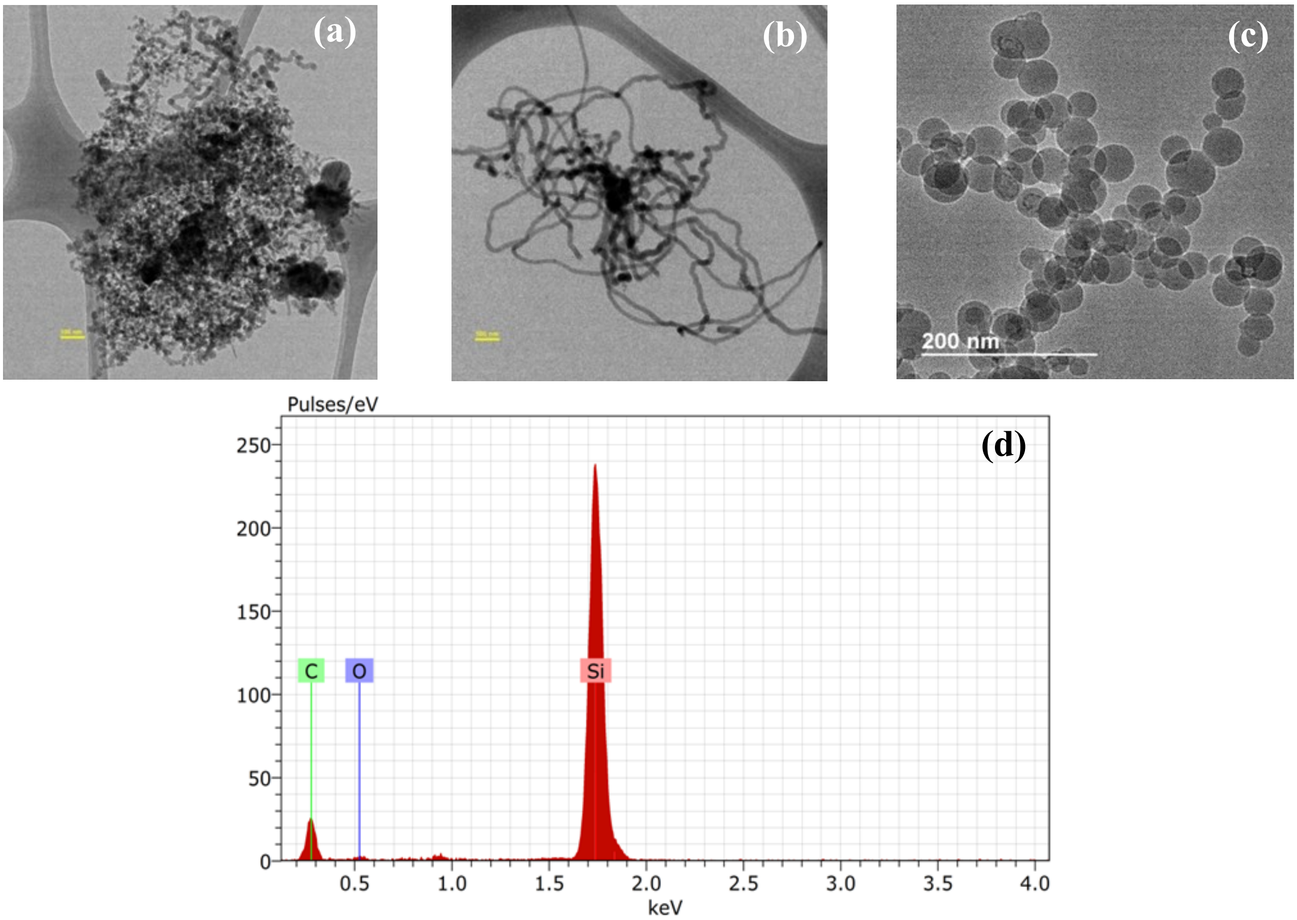

Figure 6: TEM images (a, b, c) of SiC nano particles synthesized at different plasma parameters, EDX spectrum (d) of the same nano-particles

In another application, Tungsten based nano particles were synthesized, in the form of colloids, using an under-water operating plasma torch. The size of the particles was observed to be very small (< 20 nm) as the same was confirmed using TEM. The TEM images showing the particle size, and the electron diffraction (ED) pattern are shown in figure 4.

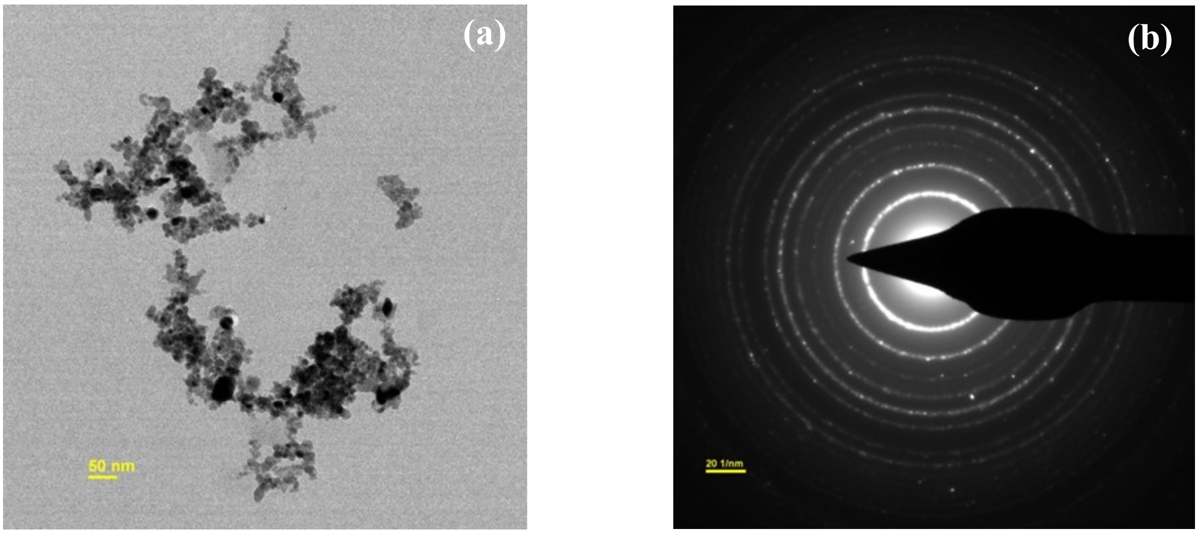

Figure 7: (a) TEM image of synthesized powder, and (b) ED pattern of the same powder

In yet another application, TEM was used for identifying the size and shape of ZnO nano-rods, synthesized at FCIPT using a thermal plasma arc. Further, TEM was also used for chemically mapping the nano-rods to show the distribution of individual elements. Figure 5 shows these TEM images.

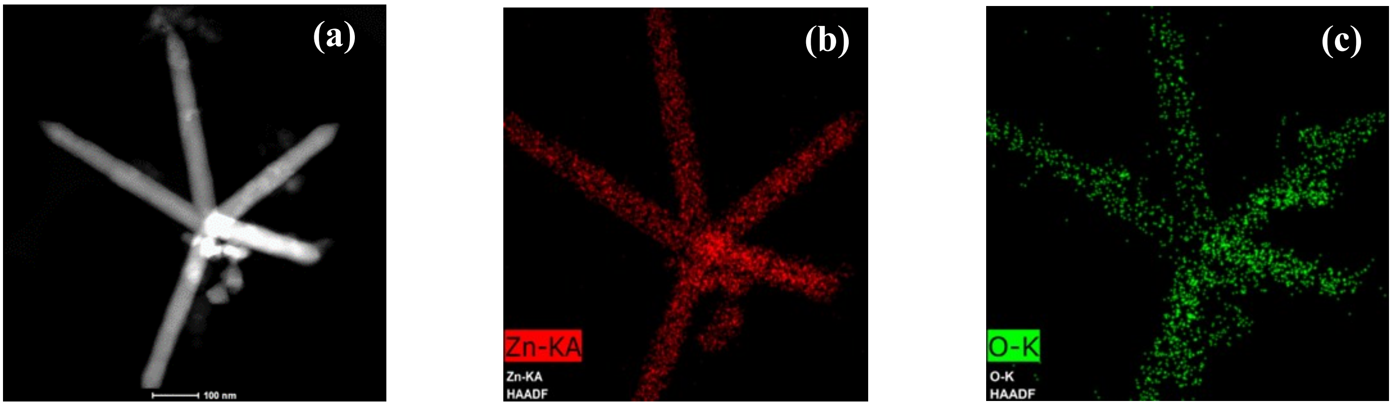

Figure 8: TEM images showing (a) the shape and size of the synthesized nano-rods; (b) and (c) elemental mapping of the same nano-rods

TEM was also used for studying thin-film deposited samples. Figure 6 shows the TEM image of the TiN thin film deposited on a glass substrate. The image 6a clearly indicates the columnar growth of the deposited coating. Further, the images 6b and 6c confirm the poly crystalline nature and chemical constitutes of the film respectively.

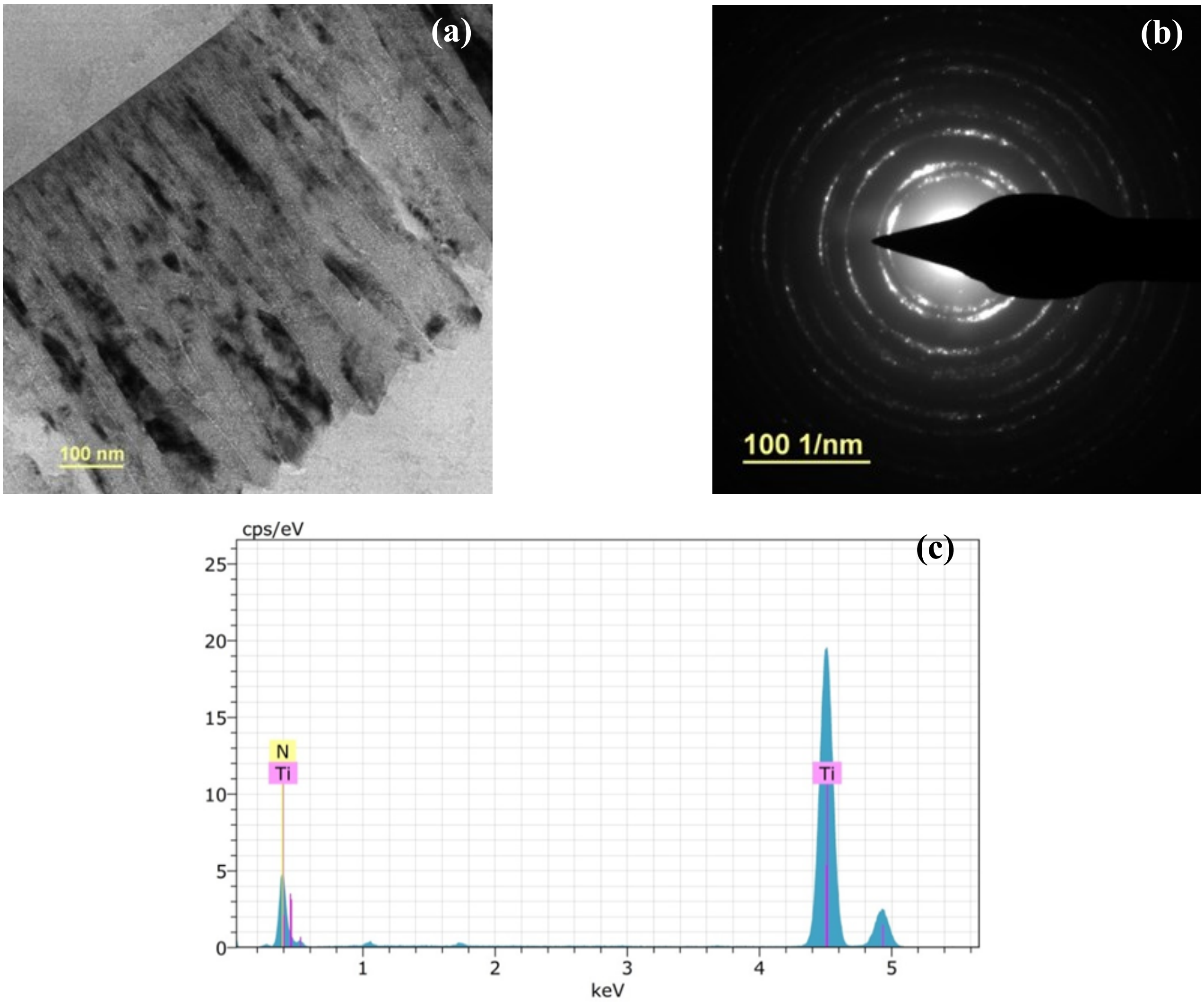

Figure 9: (a) TEM image of TiN film deposited on glass, (b) ED pattern, and (c) EDX spectrum of the same film

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