DOI: 10.1017/S1431927605500990

Microsc Microanal 11(Suppl 2), 2005

Nondestructive, High-Resolution Materials Characterization with the CR-AFM

U. Schmidt, A. Jauss, W. Ibach, K. Weishaupt, and O. Hollricher

WITec GmbH, Hoervelsinger Weg 6, Ulm 89075, Germany, www.witec.de

Materials research, biomedical research, and semiconductor manufacturing can all benefit from nondestructive, high-resolution methods of analysis. The CR–AFM combines Raman spectroscopy, a chemical analysis technique, with high-resolution imaging methods such as Confocal Microscopy and Atomic Force Microscopy (AFM). With this instrument it is possible to analyze heterogeneous materials with respect to their chemical composition and surface structure without laborious sample preparation. The materials can be analyzed under ambient conditions or in a liquid environment.

With the Confocal Raman Microscope (CRM), it is possible to obtain Raman spectra from extremely small sample volumes (down to 0.02 μm^3) and to collect high resolution Raman images. In the Raman spectral imaging mode, a complete Raman spectrum is acquired at every image pixel and the images are extracted by analyzing spectral features (sum, peak position, peak width, etc.). Fig. 1 shows a color-coded Raman spectral image of a living epithelial rat cell in its physiological surroundings. To obtain such an image, no labeling of the cell is required. Characteristic Raman spectra of the cell are represented in Fig. 1 B. By analyzing characteristic peaks of the spectra, the components of the cell can be distinguished with a resolution of about 200 nm.

By simply rotating the microscope turret, the CRM is transformed into an AFM. With this technique, a sharp tip is scanned over the sample, providing high resolution 3D images below the diffraction limit. The highly resolved topographic structures observed with the AFM can then be linked to the chemical information obtained by the CRM. Coating technology can benefit from both the chemical identification of phases within a film and the surface roughness determination down to the nanometer scale. Fig. 2 A shows a 20x20 μm^2 AFM image of a thin film of the polymer blend PMMA-PET spin-coated onto a glass slide. Three topographic levels become visible in the AFM image: a netlike structure with an average height of 170 nm, a film with an average height of 80 nm, and the dark holes within the film. The data acquired with the Raman spectral imaging mode from these polymer blend films reveal that the netlike structure corresponds to PET, whereas the lower film is the PMMA phase. The dark topographic holes correspond to the plain glass substrate.

In addition to the identification of different chemical components within a material, slight changes in the crystallographic structure which lead to stress in materials can also be detected with Raman spectroscopy. The stress field around a Vickers indent is represented in Fig. 3 A. It was analyzed using the relative shift of the 520/cm Si-Raman line. As expected, the stress field mirrors the geometry of the Vickers pyramid. The tensile strain appears at the edges of the pyramid, while the compressive strain appears at the flat sides. Fig. 3 B shows a top view AFM image of the Vickers indent and in Fig. 3D cross sections through the indent are represented. In the high resolution cross sections, the asymmetric shape of the indent is highlighted.

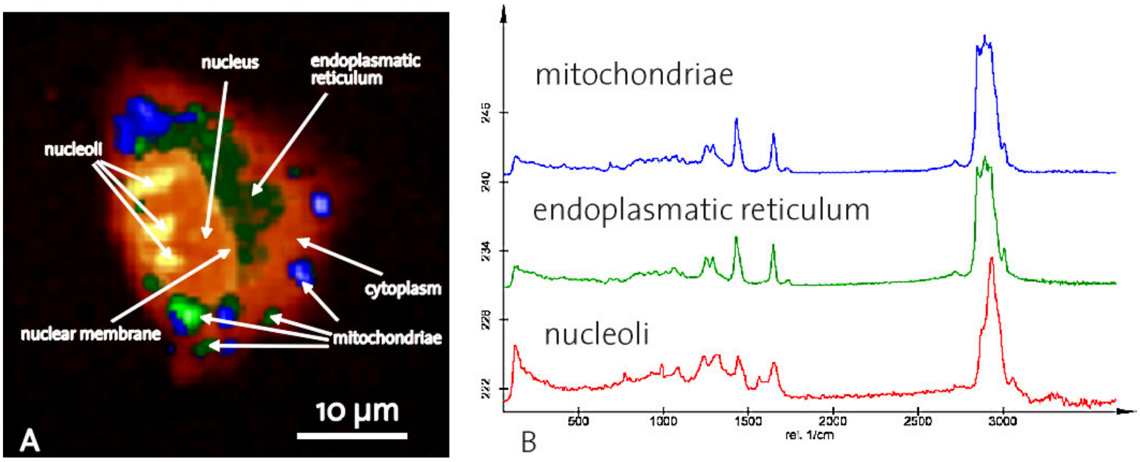

Fig. 1: Chemical composition of a living cell imaged in Raman spectral imaging mode (A) and characteristic spectra (B).

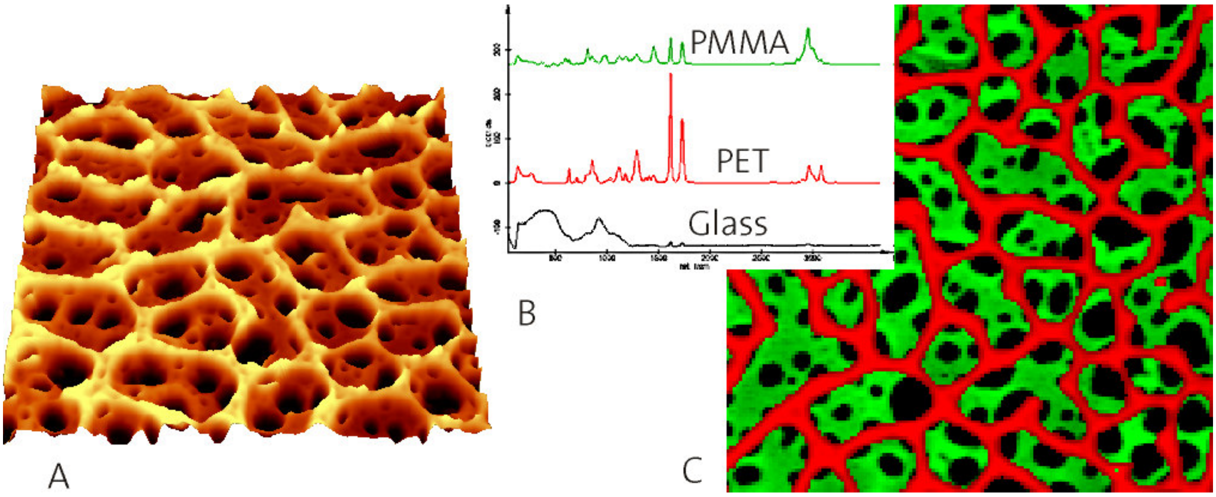

Fig. 2: PMMA-PET thin film spin coated on a glass substrate: high resolution AFM image (A), characteristic Raman spectra of components (B), and color coded Raman spectral image (C).

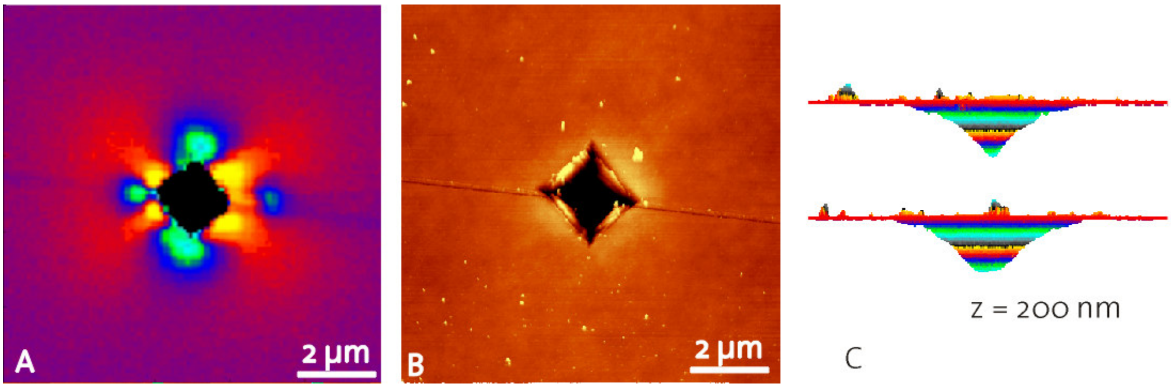

Fig. 3: Stress measurements around a Vickers indent on a silicon wafer: stress map (A), high resolution AFM image (B), and cross sections through the asymmetric shape of the indent (C).