Correlative Raman Imaging and Scanning Electron microscopy (RISE) combined with Energy-Dispersive X-Ray Spectroscopy (EDXS) for the Investigations of Polymer Materials

R. Schmidt[1], A. Zankel[1,2], H. Fitzek[2], C. Mayrhofer[2], M. Nachtnebel[2], H. Schröttner[1,2], Martina Dienstleder[2], Sabrina Mertschnigg[1,2], M. Poteser[3], H.-P. Hutter[3], W. Eppel[4]

[1] Institute of Electron Microscopy and Nanoanalysis, NAWI Graz, Graz University of Technology, Steyrergasse 17, 8010 Graz, Austria
[2] Graz Centre for Electron Microscopy, Steyrergasse 17, 8010 Graz, Austria
[3] Centre for Public Health, Medical University Vienna, Abteilung für Umwelthygiene und Umweltmedizin, Kinderspitalgasse 15,1090 Vienna, Austria
[4] Department of Obstetrics and Gynecology, Medical University of Vienna, Währinger Gürtel 18-20, 1090 Vienna, Austria

Introduction

With the system RISE (Raman Imaging and Scanning Electron microscopy) at the FELMI-ZFE, Graz, a new method opens new fields of possibilities to combine high resolution imaging by the scanning electron microscope Zeiss Sigma 300 VP (Oberkochen, Germany) and chemical analysis with the attached Raman microscope from WITec (Ulm, Germany). Combined with energy dispersive X-ray spectroscopy (EDXS - X-Max 80 from Oxford Instruments), here the power of the new system to characterize different polymeric layers as well as fillers and additives in polymeric materials and the possibilities finding and detecting nanoparticles is demonstrated.

RISE - Raman Imaging and Scanning Electron microscopy

The Raman investigation is achieved by consecutive measurements enabled by the specimen stage movement (semi-automatically) between the SEM and the Raman measurement position in the specimen chamber. Thus chemical mapping (Raman) can be combined with high resolution SEM images.

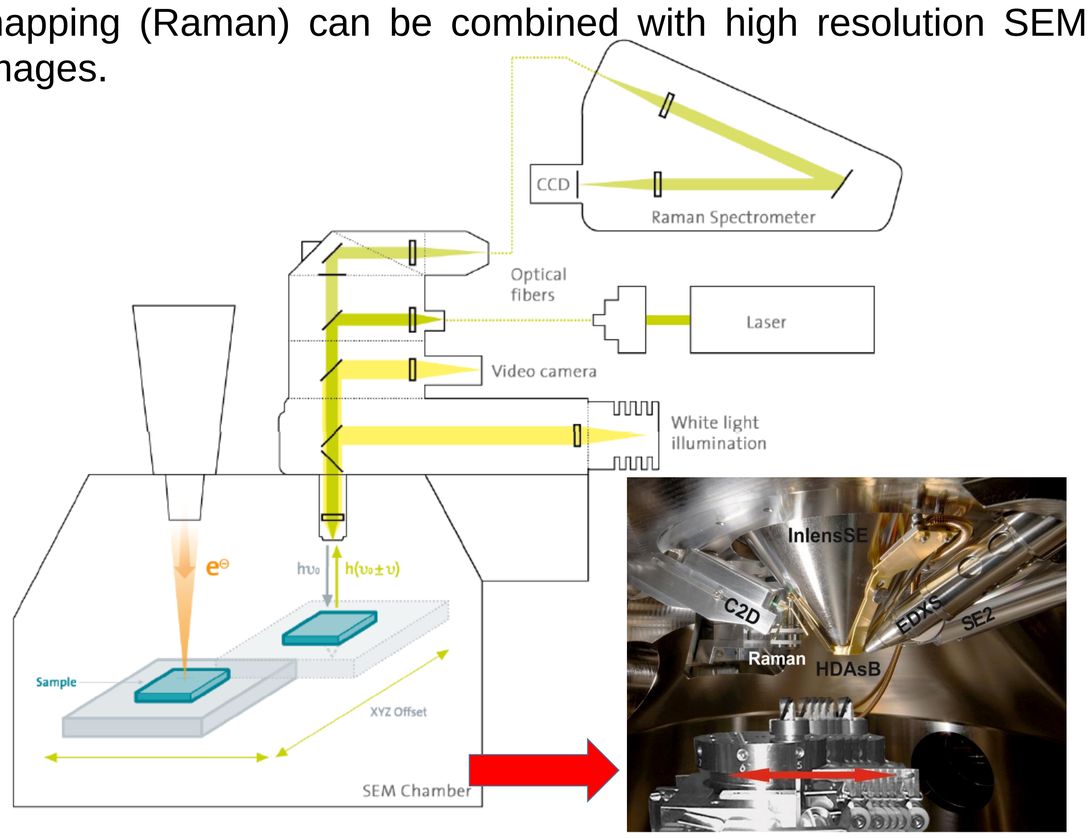

Fig. 1: Schematic of correlative Raman Imaging and Scanning Electron (RISE) Microscopy (Copyright by WITec GmbH) [1] and close up inside the microscope [2].

Application samples

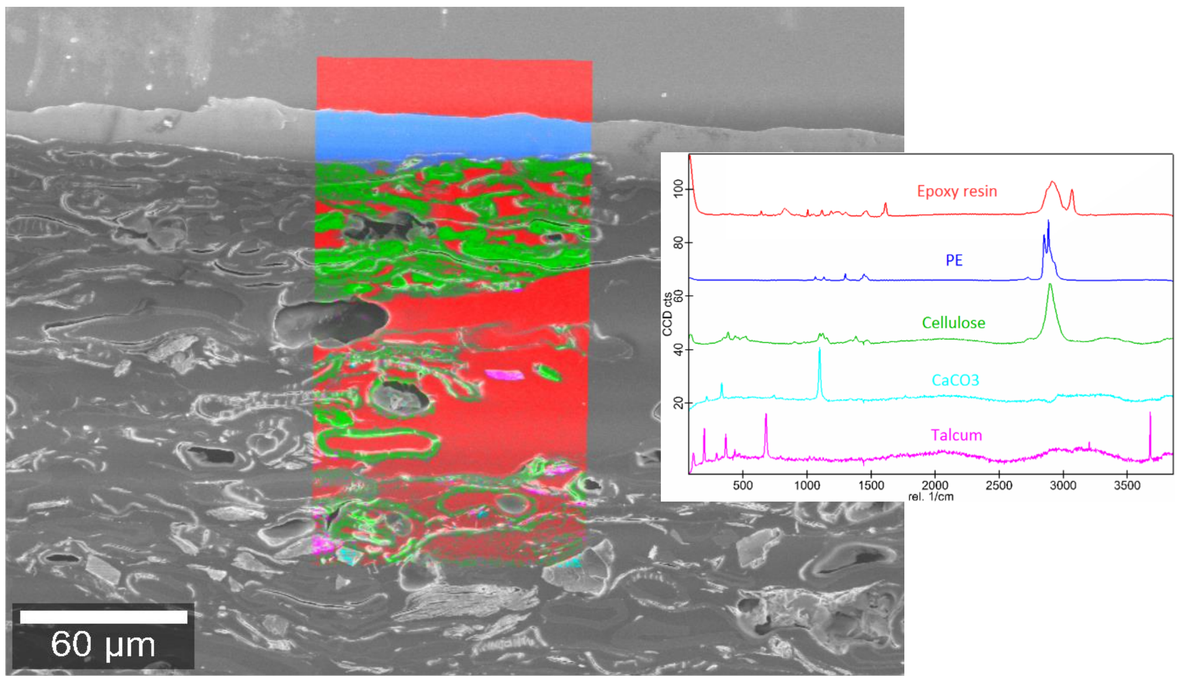

Fig. 2: Left: Correlative Raman-SEM image of a paper coffee cup; Right: Raman spectra and component identification [3].

References/ Literature

[1] C. Cardell, I. Guerra, An overview of emerging hyphenated SEM-EDX and Raman spectroscopy systems: Applications in life, environmental and materials sciences, Trends in Analytical Chemistry Volume 77, March 2016, 156-166

[2] R. Schmidt, H. Fitzek, M. Nachtnebel, C. Mayrhofer, H. Schroettner, A. Zankel, The combination of electron microscopy, Raman microscopy and energy dispersive X-ray spectroscopy for the investigation of polymeric materials, Macromolecular Symposia, Vol. 384, 1, 2019

[3] Zankel, A., Fitzek, H.: Seminar on Electron Microscopy and Nanoanalysis 2: (Raman + EDX) @ Zeiss Sigma 300 VP - Correlative Microscopy and Spectroscopy

[4] R. Schmidt, M. Nachtnebel, M. Dienstleder, S. Mertschnigg, H. Schroettner, A. Zankel, M. Poteser, H.-P. Hutter, W. Eppel, H. Fitzek, Correlative SEM-Raman microscopy to reveal nanoplastics in complex environments, Micron,accepted

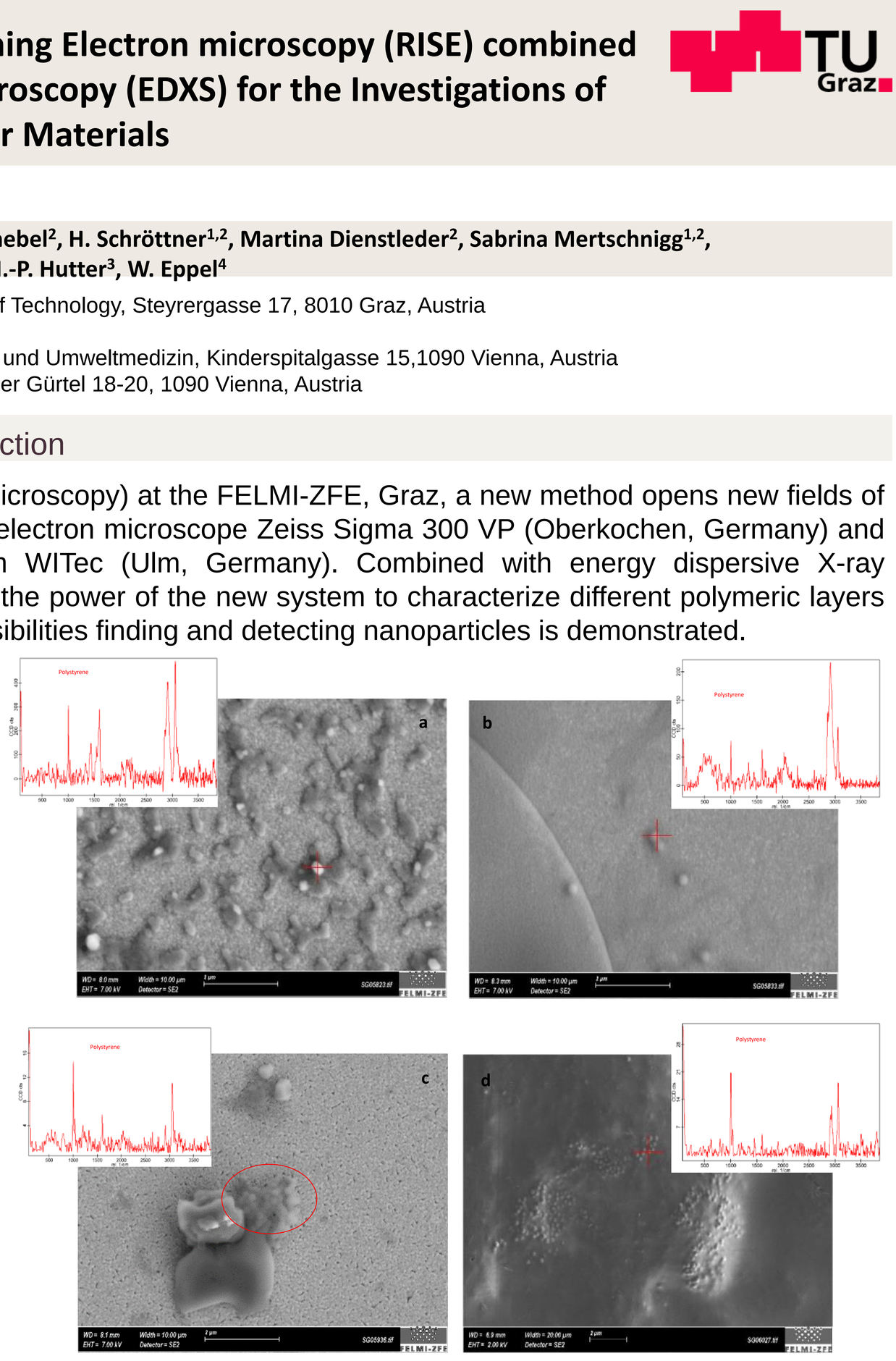

Fig. 3: SE images of analysed PS-particles in a) distilled water with a dilution of $2 \cdot 10^{4}$ µg/L; b) distilled water with a dilution of $2 \cdot 10^{-3}$ µg/L; c) Fleur de Sel salt saturated solved in distilled water with a dilution of 20 µg/L; d) amniotic fluid with a dilution of 200 µg/L. All samples were taken from the surface of the solutions and applied on a gold sputtered glass slide [4].

Additional EDXS options

With the additional EDXS investigations like conventional recording of spectra, mapping with conventional geometry, Large Area Mappings (LAM) and also automated particle analysis are possible as shown here in Figure 4.

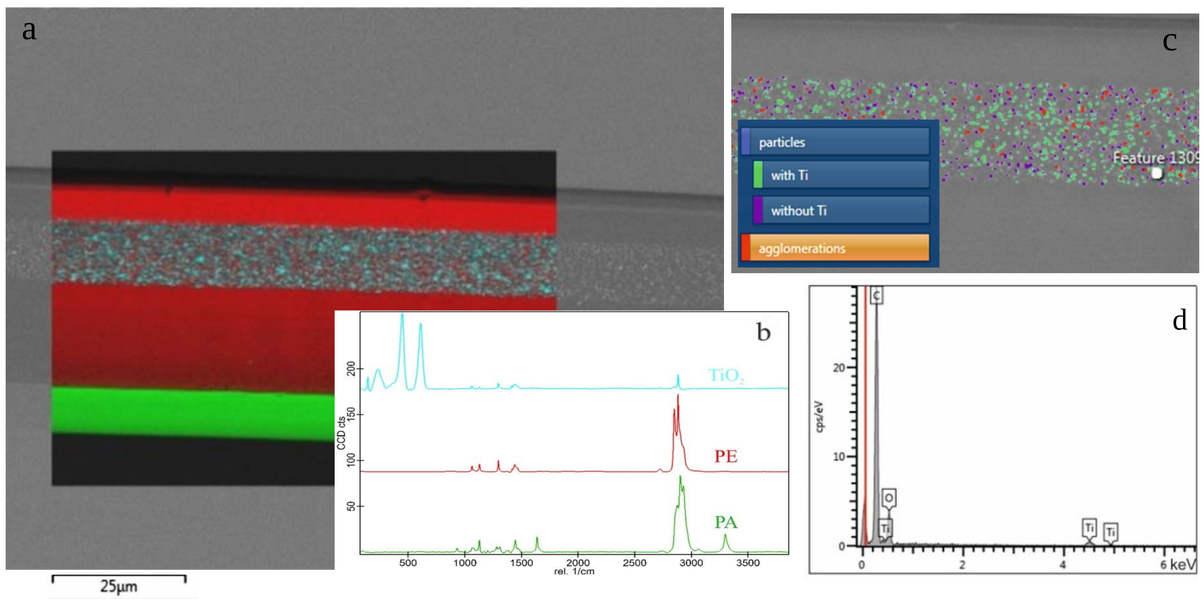

Fig. 4: a) Correlative Raman-SEM image of different polymeric layers of a packaging film; b) Raman spectra and component identification; c) TiO_2 particle layer; d) representative spectrum of one feature in this TiO_2-layer [2].

Contact

Email: ruth.schmidt@felmi-zfe.at
Web: www.felmi-zfe.at