

# Morphology of a PA / PTFE blend studied by Raman imaging



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## Introduction

The capabilities of Raman imaging as a new prospective tool for 2 dimensional characterisations have been recognised by many authors, which is demonstrated by the increasing number of publications during the last years. In this work we want to apply the advantages of this technique for polymer characterisation and morphological studies. A blend of 80% PA, 18% PTFE and 2% silicone oil used as friction bearing was under investigation. The obtained Raman spectra and images are compared to EDX elemental mappings to confirm the new results.

## Raman equipment

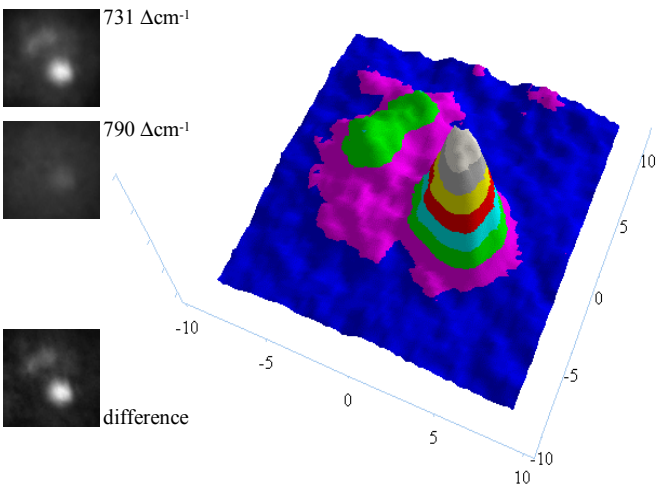
All Raman measurements were performed with a Renishaw 2000 system. The exciting laser wavelength was the 632,8nm line of a HeNe laser with 17mW output power. For the wavelength selection in the imaging mode dielectric bandpass filters with a resolution of about 20cm<sup>-1</sup> were used. The yellow bars in the spectrum indicate the spectral zones used for the images.

## Characterisation

The main components of the blend (commercial friction bearing) were already identified after acquiring one Raman spectrum. Polyamide [1] can easily be distinguished from PTFE [2] due to very different Raman spectra (see figure). The prove was done by recording FTIR spectra, which additionally brought evidence for silicon oil, which behaves as boundary lubricant and significantly reduces wear rates and coefficients of friction.

## Morphology studied by Raman imaging

To identify PTFE in the polymer blend we choose the mode at 731Δcm<sup>-1</sup> as the selective signal, because it is very strong and is not interfered by a PA mode. The image below was obtained by subtraction of a background measured at 790Δcm<sup>-1</sup>.



With this subtraction an eventual inhomogeneity in laser intensity over the sampled area, a slightly enhanced fluorescence in the PTFE clusters and spectral noise could be largely removed.

The final 3 dimensional image shows the sample area in x and y direction [scale in μm] and the intensity of the 731Δcm<sup>-1</sup> mode in the z direction. A high signal indicates a high local concentration of PTFE on the surface. The smaller cluster could be caused by a cluster lying beyond the surface.

## References

- R.L. McCreery, *Raman Spectroscopy for Chemical Analysis*, John Wiley&Sons, Inc., 2000
- D.I. Bower, W.F. Maddams, *The vibrational spectroscopy of polymers*, Cambridge University Press, Cambridge, 1989

## Results

Qualitative Polymer characterisation could be done without any problems. The following acquisition of a few Raman images lead unambiguously to the conclusion that PTFE clusters with diameters between 8 and 20μm are distributed in the PA matrix. The confirmation of the Raman results using

SEM /EDX elemental mappings is summarised in the chapter below.

**Specific chemical information from vibrational modes and the velocity of spectra acquisition allow a rapid and reliable characterisation of Polymer compounds.**

The disadvantage of a poorer lateral resolution, compared to SEM data is largely compensated by the factor of time needed for the analysis. For most applications a resolution of 1μm is sufficient anyway. Also fluorescence, normally a severe problem in Raman spectroscopy, can be overcome by simple spectra subtraction.

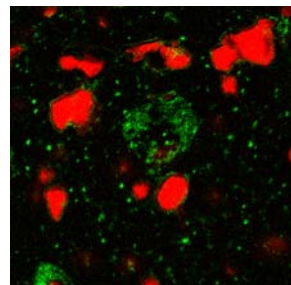
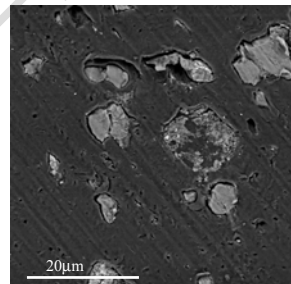
## SEM / EDX elemental mappings

To confirm the results of the Raman images we decided to employ Scanning Electron microscopy. High lateral resolution and the possibility for elemental mappings make it indeed a good choice as a complementary method. The same sample used for Raman imaging was carbon coated (200Å layer) and used for the investigations. The work was done on a Leo Gemini DSM 982 with an EDX Voyager Noran system.

A secondary electron picture with 7kV acceleration voltage shows the sample morphology. Energy dispersive spot analysis confirmed that the bright particles consist of PTFE (high fluorine concentration). The darker matrix contains no fluorine and is therefore supposed to be the PA matrix.

The elemental map to the right gives the distribution of fluorine (red) and silicon (green). The clusterlike structure and the spatial dimensions of PTFE are confirmed. Also the sizes of the cluster are comparable to the results obtained by Raman imaging. Due to the high contrast of silicon we could determine the distribution of silicon oil. It is concentrated on the PA / PTFE interfaces, which can be explained by the process of manufacture, where an initial mixture of PTFE and silicon oil is incorporated in the PA matrix.

SEM / EDX is a very good analytical tool for this kind of analysis. It's disadvantages are time consuming sample preparation, long acquisition times (in this case 5 hours) and the poor identification power for polymers, when no elements with higher Z contrast are present.



## Acknowledgement

The Raman instrumentation was financed by the Bundesministerium für wirtschaftliche Angelegenheiten in the context of the "Impulsförderung für ACR-Institute". The study was carried out in co-operation with "Innovation Business Steiner", Spielberg, Austria