Analysis of Metal Traces in Polymers with Micro-XRF on SEM

Application Note # MXRF-SEM-04

Introduction

Polymers are a class of materials with an extremely wide spectrum of properties and have hence a large range of applications. These are e.g. constructive parts in vehicles, isolators in electrical and electronic components and even materials to produce toy figures.

Metal additives in polymers are used to tailor the properties for the aimed application. In the past various heavy metals e.g. Cd, Pb or Hg have been used, which might be hazardous for the human body. The 2005 issue of the RoHS directive defines 0.1 mass% as maximum content for Pb and Hg when applied in homogenous materials for electric or electronic components.

Sample analysis using micro-X-ray fluorescence spectroscopy (Micro-XRF) in the scanning electron microscope (SEM) and energy-dispersive spectroscopy (EDS) is especially promising through the sensitivity for trace elements. This advantage is used here for the investigation of acrylonitrile butadiene styrene (ABS) reference materials.

Samples

The measured polymeric material is an acrylonitrile butadiene styrene (ABS) with low metal content. The specimens were investigated to check the XRF detection limit. The BAM (Federal Institute for Materials Research and Testing in Germany) has developed a set of reference materials (Figure 1). Two of these ABS materials (ABS-3 and ABS-7) were selected for the measurements described in this application note.
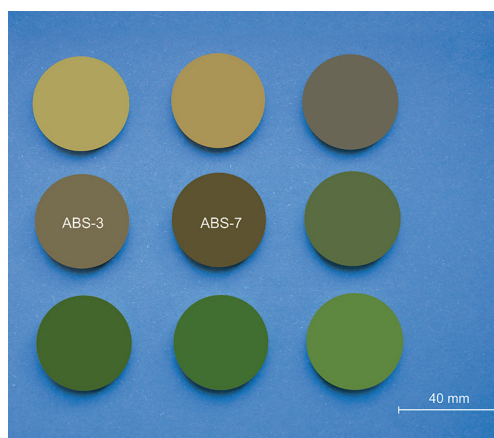

Figure 1

All samples of the ABS reference material (Acrylonitrile Butadiene Styrene) indicating the analyzed specimens ABS-3 and ABS-7.

Measurement conditions

XTrace is a micro-focus X-ray source equipped with a Rh target. It was mounted on a scanning electron microscope (SEM). For detection a silicon drift detector XFlash® 6 I 30 with 30 mm² active area and an energy resolution of 123 eV for Mn Kα was used. The micro-XRF spectra were acquired at 50 kV, 600 µA and 300 s live time.

Results

Table 1 shows the certified values of specimen ABS-3 and ABS-7. In sample ABS-7 all four elements are below the limit of detection for EDS. In sample ABS-3 only the Pb value is above the limit of detection for EDS. The spectra of both samples show the peaks of all four elements demonstrating that traces of Cr, Hg, Br, and Pb are detectable in polymers using XTrace (Figure 2 and 3). In the spectrum of sample ABS-3 small peaks of Hg and Br are visible (marked by a circle). The element concentrations are 0.0017 mass% (= 17 ppm) for Hg and 0.0050 mass% (= 50 ppm) for Br. The Pb peak of sample ABS-7 proves that also such small amounts like 0.0024 mass% (= 24 ppm) of Pb are detectable with XTrace as well as the 0.0007 mass% (= 7 ppm) of Cr.

Conclusion

Micro-XRF analysis of metals in polymers demonstrates XTrace's capability of the detection of elements in low concentrations. Mass fractions down to 10 ppm can be detected with XTrace. These low concentrations are below the limit of detection of EDS.

Element	Certified values	
	ABS-3	**ABS-7**
Cr	0.0101	0.0007
Br	0.0050	0.0466
Hg	0.0017	0.0878
Pb	0.1434	0.0024

Table 1

Certified values in mass% normalized for both specimens
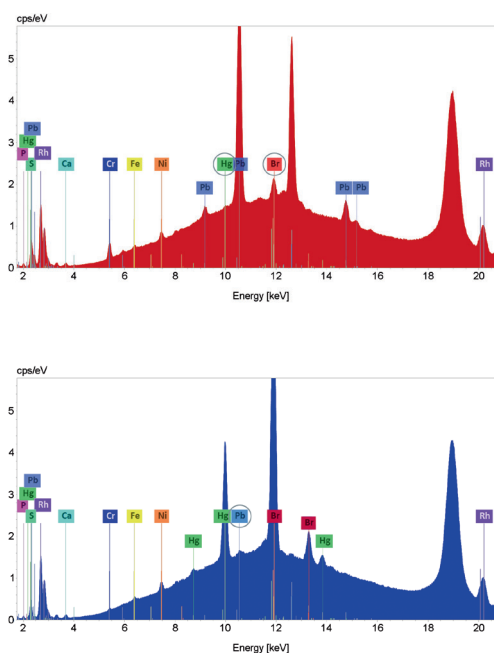

Figure 2

Spectrum of sample ABS-3. Even low concentrations of Hg (0.0017 mass%) and Br (0.0050 mass%) are visible as peaks.

Figure 3

Spectrum of sample ABS-7. Despite the low concentration of 0.0024 mass% the element Pb is visible as a peak in the spectrum.

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