Lab Report XRF 170

S2 PUMA Series 2

- Process Control of Si-Coated Paper and Cardboard

Silicone (Si) is the most widely used release coating material due to its ability to offer several desirable performance characteristics for different industrial applications. Silicone as additive helps provide resistance to slip, moisture, and abrasion, improved adhesion, anti-blocking properties, gloss, foam control, and many more. Large scale Si-coated paper, cardboard, and thin film production requires a fast and accurate analytical technique for process and quality control. The main requirements for the Si-coated materials are its purity, stability, and homogeneity of the layer on the paper or thin film surface.

Instrumentation

The S2 PUMA Series 2 energy dispersive X-ray fluoresence (EDXRF) spectrometer was used for this report. It is ideally suited for process control with its XY Autochanger for unattended analysis of up to 22 samples. The combination of a 50 Watt X-ray tube with closely-coupled beam optics and the new HighSense™ detector technology enables optimal sample excitation, resulting in outstanding analytical performance (Figure 1).

The S2 PUMA is equipped with Bruker's unique SampleCare™ technolgy, a multi-layer system that protects the instrument's vital components from accidental spills or sample breakages. This guarantees a high system uptime and easy maintenance. The intuitive TouchControl™ interface allows for independent routine operation in island mode without needing to connect a PC.

Standards and Sample Preparation

A set of six standard samples with varying Si coating thicknesses was used for this calibration, listed in Table 1.The samples only needed to be cut into shape – no other sample preparation was necessary. To avoid backscattering interference and to keep the light samples in place, inserts (or backscattering cups) were used, see Figure 2.

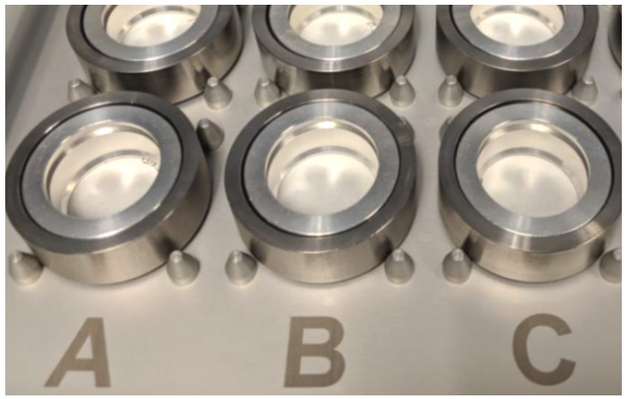

Figure 2: Samples with backscattering cups placed on an EasyLoad tray

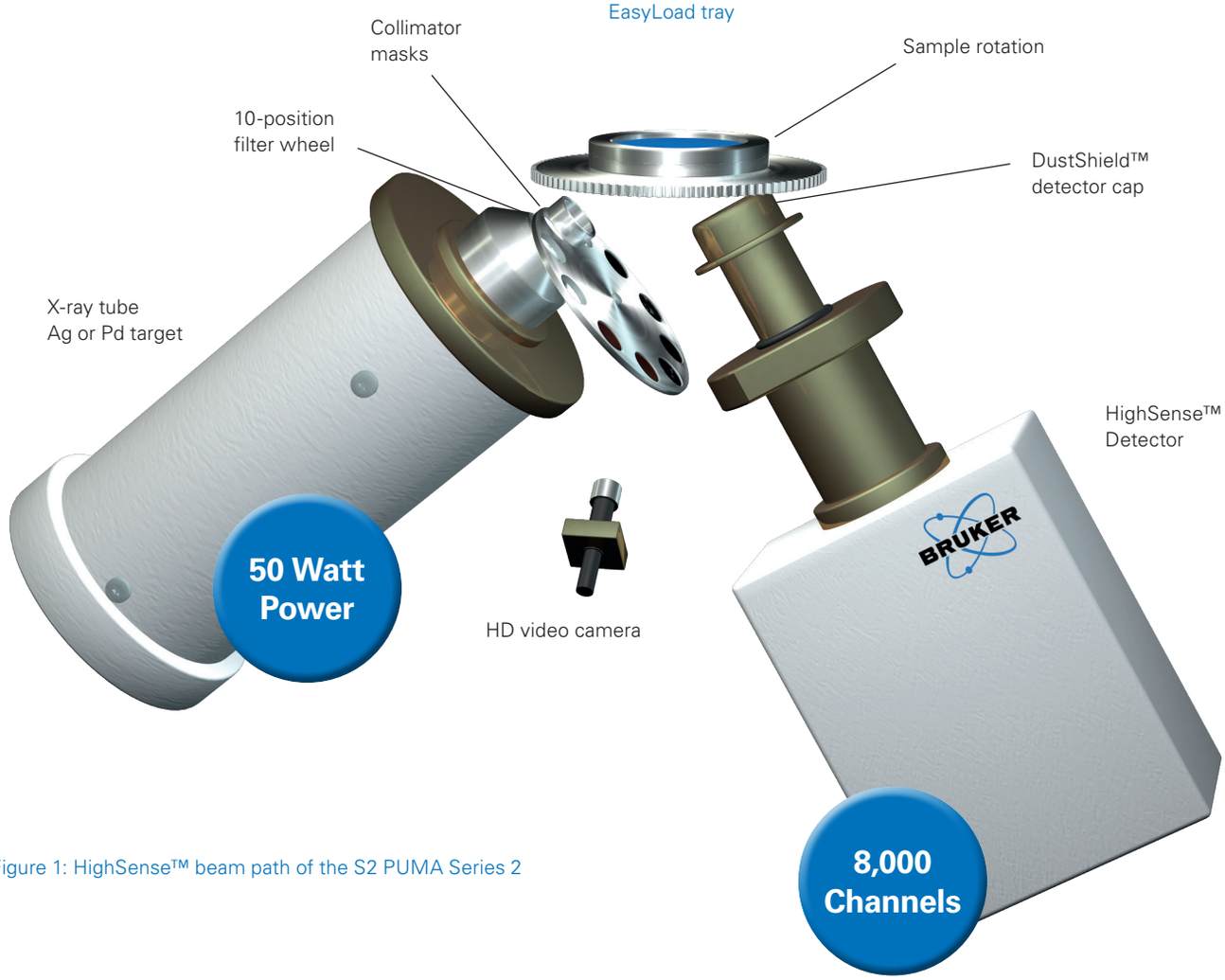

Figure 1: HighSense™ beam path of the S2 PUMA Series 2

Measurement Method

The samples were measured in vacuum mode. The parameters for this method are listed in Table 2. The gross measurement time was approx. 2:15 min, which includes sample handling, pumping and venting, setting the current, collecting the spectrum, dead time correction, and processing the results. The sample rotation allows to obtain representative results, even for heterogeneous samples. The automatic current setting adjusts the current such that the optimal count rate is reached.

The spectra of the standards are shown in Figure 3.

Calibration

The calibration curve uses the integrated Si KA1 peak, and the background is calculated from enveloping the spectra. Integrating over the peak increases the detected intensities and improves the measurement statistics. The calibration curve shows excellent correlation (see Figure 4) without the need for any matrix corrections. The achievable detection limit is 0.0002 lbs/ream (0.0003 g/m²).

Standard	Concentration [lbs/ream] (g/m²)
Std A	0.71 (1.16)
Std B	0.85 (1.38)
Std C	1.04 (1.69)
Std D	1.18 (1.92)
Std E	1.51 (2.46)
Std F	1.77 (2.88)

Table 1: Standards list.

	Voltage [kV]	Current [mA]	Mode	Filter	Rotation	Time [s]
Si	20	Auto-matic	Vacuum	None	Yes	100

Table 2: Measurement Parameters

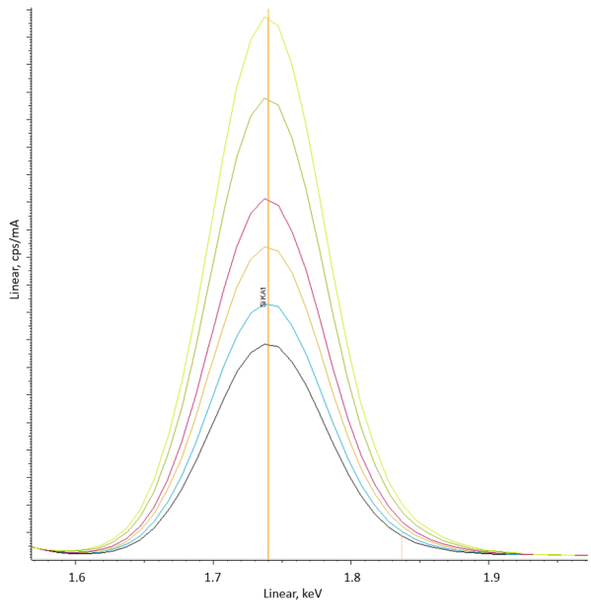

Figure 3: Spectra of the standards

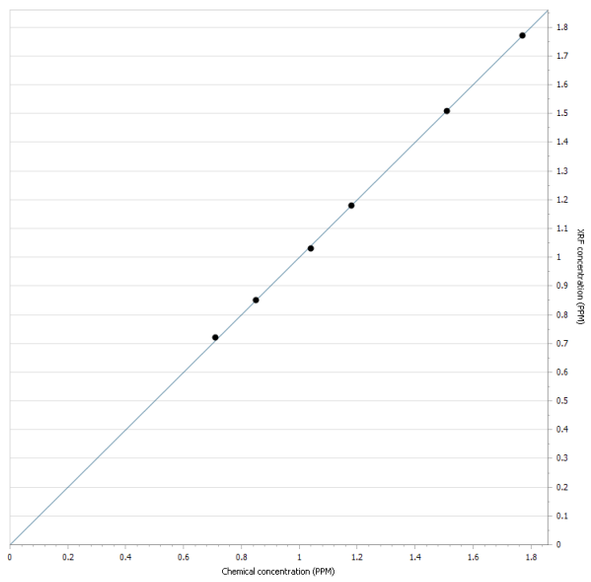

Figure 4: Calibration curve for Si

Precision Testing

Ten preparations of a sample, that was not part of the calibration set, were measured against the calibration. The low standard deviation of the results (Table 3) demonstrates the suitability of the sample preparation and the excellent stability of the S2 PUMA.

Conclusion

This lab report highlights the outstanding performance of the S2 PUMA Series 2 for process control and elemental analysis of Si additives in paper production (Figure 5). The high-power 50 Watt X-ray tube, combined with the HighSense technology and vacuum mode provide excellent analytical performance, short measurement times, and low operating cost (no helium consumption!). Industry-leading features, such as the XY Autochanger sample handling with optional integration into automated environments and TouchControl™, make continuous process control as well as product quality assurance easy and quick.

Figure 5: Disposable paper cups are typically Si-coated

Repetition	Si Weight [lbs/ream]	Intensity [cps/mA]
01	0.378	3265.8
02	0.377	3257.4
03	0.378	3269.0
04	0.378	3267.6
05	0.378	3266.9
06	0.399	3271.5
07	0.378	3267.5
08	0.378	3268.6
09	0.377	3260.9
10	0.379	3275.4
Average	**0.380**	**3267.1**
Std. Dev.	**0.006**	**4.8**
Rel. Std. Dev.	**1.67 %**	**0.15 %**

Table 3: Analytical precision

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