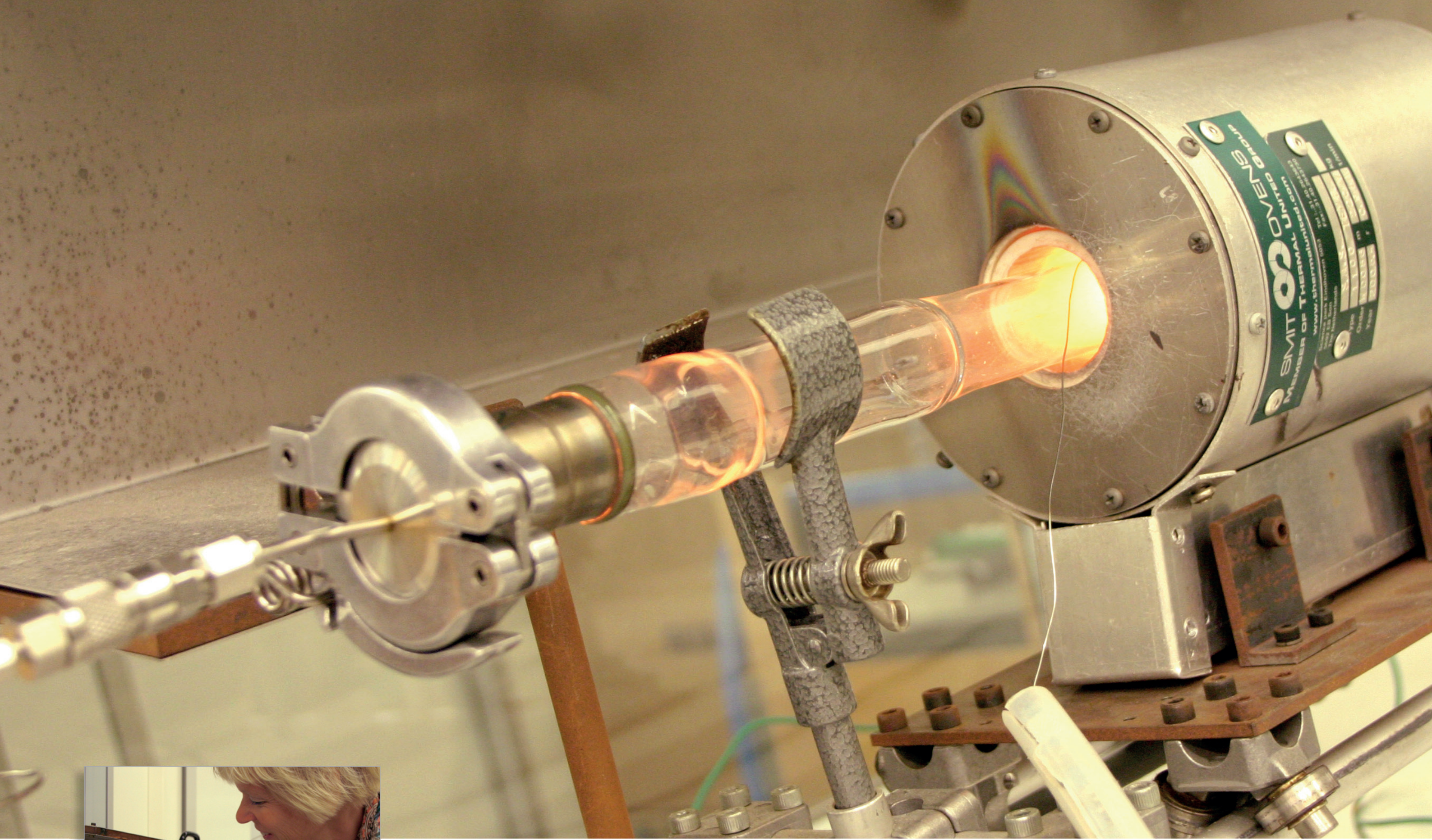

Thermal Desorption

- Outgassing
- Contamination control
- Process control
- Simultaneous detection of gasses

During manufacturing of many products, surface cleanliness of the individual components is of utmost importance. Degassing of e.g. adsorbed water or organic compounds during the production process can deteriorate the performance of the final product. Thermal desorption is a technique that is able to detect small amounts of desorbed molecules from a surface. The concentrations of volatilized molecules, such as H_2O, CO, CO_2 and organic compounds, can be analyzed quantitatively. In this way, information essential for product quality control and failure analysis can be provided.

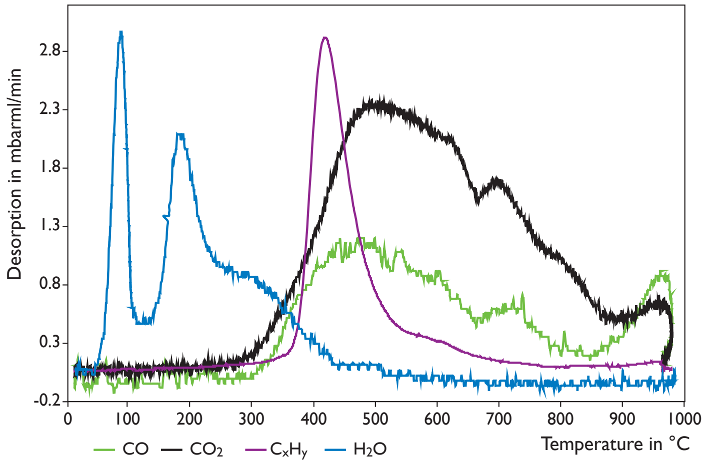

Fig. 1: typical desorption graph, displaying the amount of desorbed gas as a function of temperature.

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Principles of the technique

A sample is placed in a quartz tube with a quartz crucible. After flushing with a carrier gas (argon or synthetic air), the sample is heated with an appropriate ramp rate, leading to desorption of adsorbed gasses. The degassed molecules are transported by the inert carrier gas to a specific detector and analyzed. H_2O, CO, CO_2 are detected using infrared spectroscopy and C_xH_y compounds using a Flame Ionisation Detector (FID). Desorption of these compounds can be measured as a function of time under different heating rates and atmospheres. It is possible to detect the oxidation products of the materials that do not desorb under Argon, by heating the sample (again) while flushing with purified air.

In figure 1, a typical example is displayed. The four types of gasses all have their own temperature response. The desorption curve of water displays two peaks, the lower and higher temperature peaks representing physisorbed and chemisorbed water respectively. Response curves, like the one displayed in figure 1, yield valuable information for process optimization, providing input for e.g. necessary temperature pre-treatment steps or limits for the allowed temperature budget.

Process control

Galvanic deposition is a well-known method to deposit a zinc layer on metal strips. The quality of this Zn layer is essential for proper functioning of the metal. Upon under-perfomance of a batch of metal strips, a root cause analysis was performed. Figure 2 displays a DES analysis of the amount of desorbed H_2O from a series of production batches. A direct relation was found between the performance of the metal and the water content. Simultaneously detected CO, CO_2 and C_xH_y amounts did not yield this correlation. As a result, the variation in performance could be correlated to systematic variations in the cleaning procedure, allowing for optimization of this critical process step.

Typical applications

- Process control of surface cleaning steps
- Out-gassing of binder materials
- De-gassing of glue residues

Characteristics

Samples	All kind of solids (metal, glass, powders, glue residues, ..)				
Compounds	H_2O, CO, CO_2 and hydrocarbon compounds				
Sample preparation	Preparation of the sample in a glove box and subsequent transfer to the analytical set-up is possible				
Detection limits	Depends on the amount of material to analyse. For 1 gram material:				
	H_2O	5	mbar*ml	5	weight ppm H_2O
	CO	0.5	mbar*ml	0.2	weight ppm C
	CO_2	0.5	mbar*ml	0.2	weight ppm C
	C_xH_y	0.25	mbar*ml	0.1	weight ppm C
Maximum temperature	1000 °C				

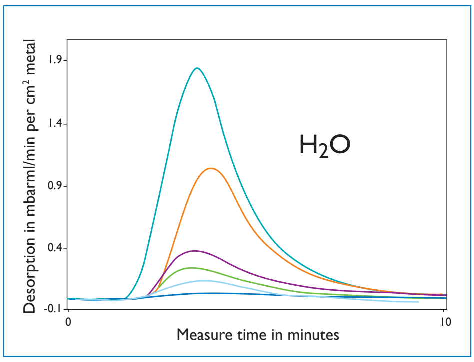

Fig. 2: Desorption curves from different batches of products, displaying large variations in the amount of desorbed water. These variations could be correlated to systematic differences in the cleaning procedure.

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