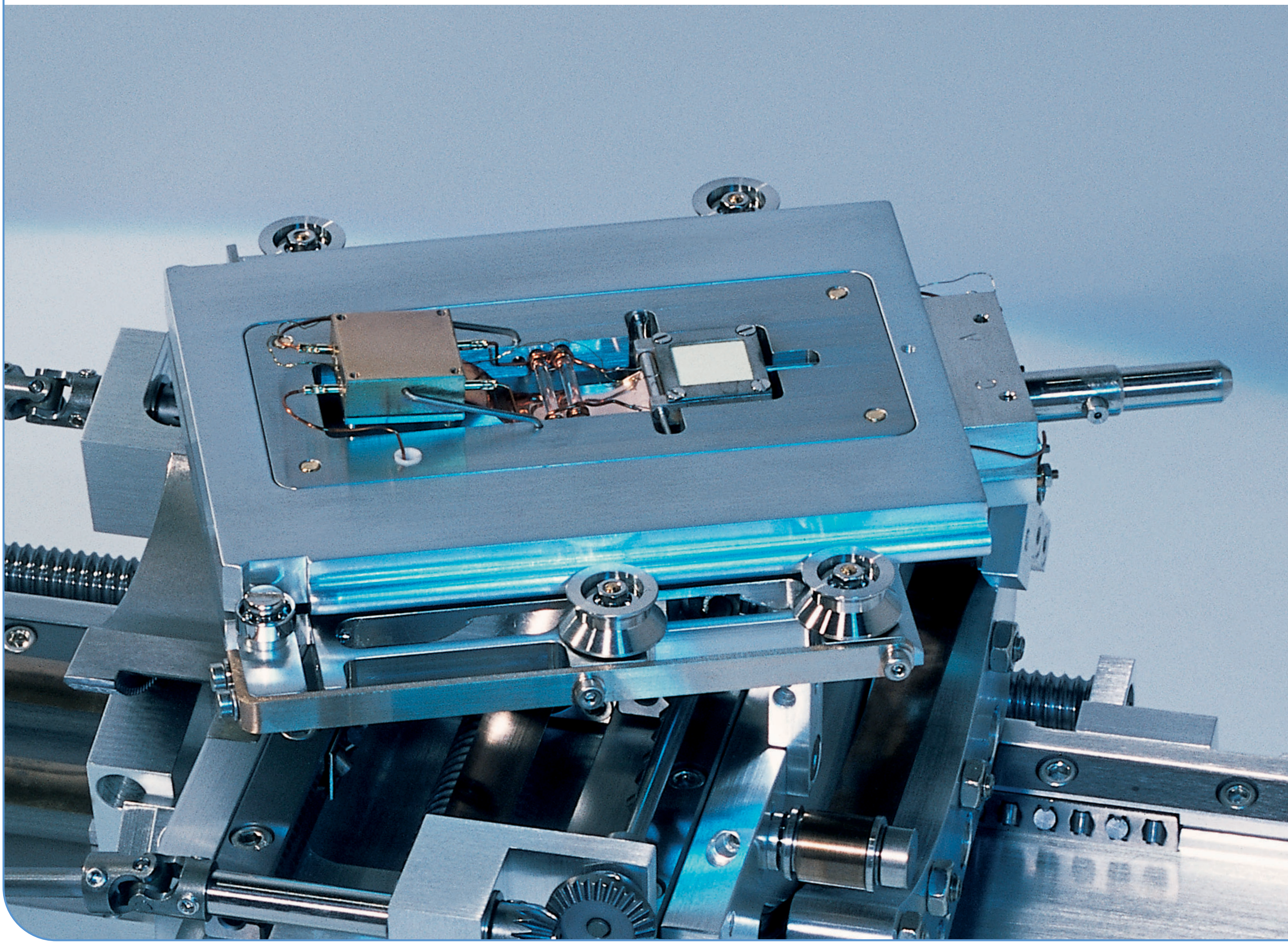

Analysis of Volatile Compounds

Changes in composition of materials with temperature can be determined using the sample stage heating and cooling systems of the TOF.SIMS 5. The system is compatible with the 100 mm, 200 mm and 300 mm models of the TOF.SIMS 5. The sample temperature can be regulated between -130° C and +600° C with a 1° C precision. In order to extend the application of the TOF.SIMS 5 to the analysis of volatile compounds, a similar system can be fitted to the loadlock to cool volatile samples before evacuation.

TOF.SIMS 5

Heating and Cooling

Heating and Cooling Systems

The heating and cooling systems for the analysis chamber and loadlock comprise liquid nitrogen cooling, resistive heating and programmable computer controlled PID regulation. Both options are compatible with the 100 mm, 200 mm and 300 mm models of the TOF.SIMS 5.
The sample temperature can be adjusted between -130° C and + 600° C with a 1° C precision. The ground potential of the sample stage allows the thermocouple to be fitted onto the sample holder in direct contact with the sample for accurate temperature measurement. Computer programming of temperature and analysis provides the means of determining the changes in composition with changing temperature to give temperature profiles (so-called Temperature Programmed SIMS), of analysis at chosen temperatures, of determination of the temperature at which transitions take place, and of analysis of temperature sensitive materials.

Detection of BHT on Silicon

The TOF.SIMS 5 with sample cooling in the loadlock is used to analyse volatile compounds with high sensitivity. For example, BHT (Butylhydroxytoluene), used as an antioxidant in storage box polymers, has been found on Si wafers. The sample has been cooled in the loadlock to 150 K prior to pump-down and analysis. See figure 1.

Temperature Programmed SIMS

After introducing the cooled sample into the main chamber, a temperature profile was recorded with a temperature ramp from 150 K to 450 K. The temperature profile in figure 2 shows that the BHT M^+ intensity drops at 213 K while the Si substrate signal increases indicating the desorption of BHT. Note that sample cooling significantly below 213 K (-60° C) is essential for the analysis of volatile materials such as BHT.
Parallel mass detection allows a complete high mass resolution spectrum to be recorded at each point of the profile. Figure 3 shows the subtraction of the spectra reconstructed at temperatures before and after the desorption of BHT. Note that the peaks of all compounds without variation (e.g. hydrocarbons) in this temperature region are eliminated.

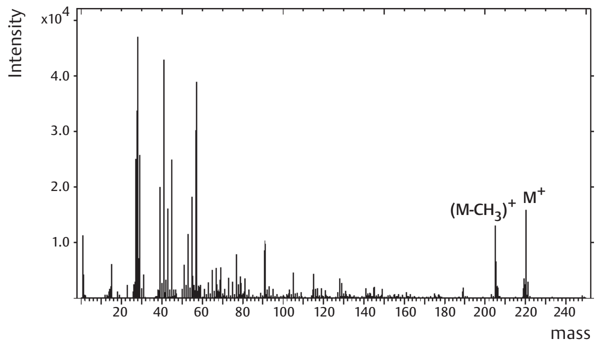

Fig. 1

Positive spectrum of **BHT** at 150 K (-123° C)

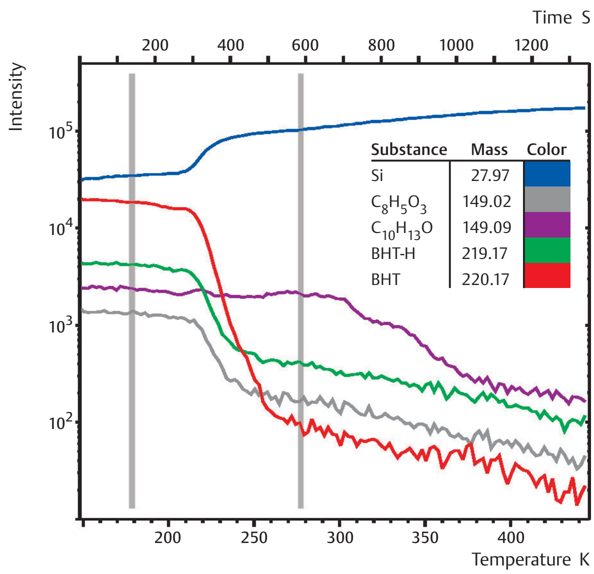

Fig. 2

Temperature profile recorded during temperature ramping from 150 K to 450 K

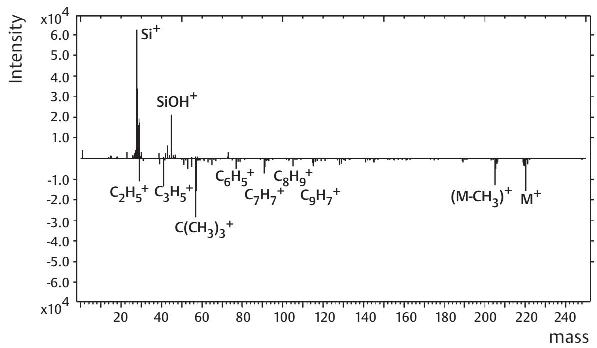

Fig. 3

Spectra difference (280 K - 180 K) of **BHT on Si**

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