REFLECTOMETRY
EVALUATION OF THE NEW HIGH TEMPERATURE CHAMBER

The new high temperature high vacuum chamber HTC-Reflectometry provides a unique design for reflectometry measurements: With the environmental heater, sample temperatures up to 800°C can be realized in a high vacuum inert gas. With the **KEC** (**K**nife **E**dge **C**ollimator), the chamber fulfills the needs for precise reflectometry measurements on surfaces and thin film structures. The HTC-Reflectometry chamber is the result of a joint-development of Bruker AXS with its years of experience in diffraction techniques and **mri** Physikalische Geräte GmbH (**m**aterials **r**esearch **i**nstruments) with its detailed know-how in high temperature chamber design.

The HTC-Reflectometry chamber was tested on a D8 ADVANCE diffractometer in the temperature range between 30°C and 800°C. The chamber is equipped with an environmental heater made from Ta. The diffractometer was equipped with a Cu FL line-focus X-ray tube and a primary beam Göbel Mirror with an angular divergence of about 0.03° in the scattering plane (Fig. 1).

Fig. 1 Reflectometry chamber mounted on a D8 ADVANCE with twin Göbel Mirror TGM arrangement.

lattice type: hexagonal	lattice constant (nm)	1st Order thermal expansion coefficient ($10^{-6}K^{-1}$)	2nd Order thermal expansion coefficient ($10^{-9}K^{-1}$)
a	0.4758	5.431	2.150
b	1.2991	5.927	2.142

Table 1 Physical properties of sapphire

With a perfect single crystal sample, one can dispense with secondary optics. Instead, a wide-open detector can be used, thus avoiding any influences of the diffracted beam optics on the results. Since the primary beam Göbel Mirror conditions a parallel beam, the angular positions of the perfect crystal Bragg reflections depend on the incident angle only. Sample stage height variations, which may occur during heating, do not influence the results of the measurement.

The measurements shown in figure 2 are rocking curves at the 006 reflection of Al_2O_3 (00.1). The rocking curves are executed from Θ=20.65° up to 20.95° in steps of 0.002°. The measurement time/step was 0.2s. The temperature steps were: 30°C, 70°C, 100°C, 150°C, 200°C, 250°C, 300°C, 350°C, 400°C, 450°C, 500°C, 550°C, 600°C, 650°C, 700°C, 750°C, 800°C. During the measurements, the pressure inside the chamber was stable at $5*10^{-5}$ mbar. For each temperature, the rocking curve was started approx. 2 minutes after the set-point temperature had been reached.

Each rocking curve consists of two Bragg-reflections components caused by $CuK\alpha_1$ (λ=0.154062nm) and $CuK\alpha_2$ (λ=0.154439nm). The Bragg-reflections show an intensity ratio of 2:1. The maximum intensity of the $CuK\alpha_1$-line was $I_0=5*10^6$ cps, whereby each curve is normalized.

The angular position of the Bragg-reflections was used to calculate the lattice parameter c. Figure 3 shows the evaluated lattice parameter change versus temperature. The mean square deviations of the lattice parameter c are plotted as vertical bars. Since the angular positions of the Bragg-reflections are determined with a mean square deviation of about 0.0016°, the mean square deviations of the lattice parameter results in $9*10^{-5}$ nm. The line in figure 3 shows the theoretical change of the lattice parameter c with increasing temperature.

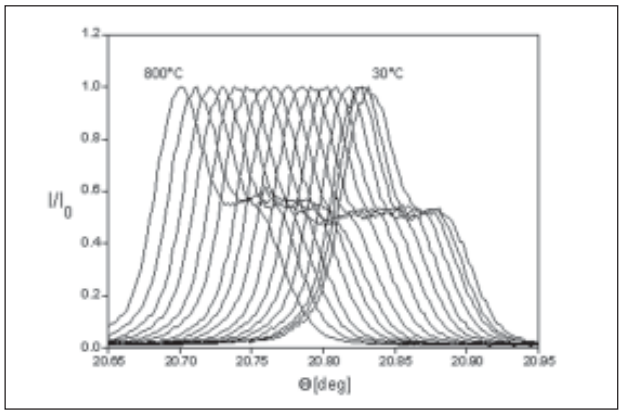

Fig. 2 Rocking Curve measurements on Al_2O_3 executed for temperatures between 30°C and 800°C.

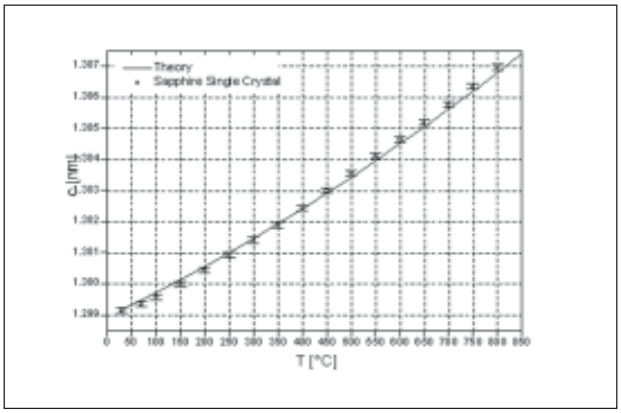

Fig. 3 Determined lattice parameter c of sapphire versus temperature. The line corresponds to the nominal behaviour of c.

Conclusion

The new HTC-Reflectometry chamber with an environmental Ta heater allows accurate high temperature investigations from room temperature up to 800°C. Temperature dependent lattice parameter investigations were used to show the agreement between the nominal and the real temperature of the sample in the HTC-Reflectometry chamber.

Technical Data

Tube:	CuKFL 0.4 x 12 mm line
System:	D8 ADVANCE Theta/Theta
Primary Optic:	Parallel beam Göbel Mirror
Sample Stage:	HTC-Reflectometry
Diffracted Beam Optics:	None
Detector:	NaJ Scintillation

BRUKER AXS GMBH
ANALYTICAL X-RAY SYSTEMS

D-76181 KARLSRUHE
GERMANY

TEL. (+49) (721) 595-2888
FAX (+49) (721) 595-4587
http://www.bruker-axs.de
Email: info@bruker-axs.de

BRUKER AXS, INC.
ANALYTICAL X-RAY SYSTEMS

5465 EAST CHERYL PARKWAY
MADISON, WI 53711
USA
TEL. (+1) (800) 234-XRAY
TEL. (+1) (608) 276-3000
FAX (+1) (608) 276-3006
http://www.bruker-axs.com
Email: info@bruker-axs.com

BRUKER AXS, LTD.
ANALYTICAL X-RAY SYSTEMS

MEADOWSIDE
MOUNTBATTEN WAY
CONGLETON
CHESHIRE CW12 1DN
GREAT BRITAIN

TEL. (+44) (1260) 296-900
FAX (+44) (1260) 296-909