

Application Report XRD 7

D2 PHASER Desktop XRD: Anode Coke (L_c Value) Analysis

The D2 PHASER is a portable desktop XRD instrument for research and quality control. It is easy to operate and independent of external media such as cooling circuits. Thanks to the LYNXEYE detector it is the fastest desktop XRD system on the market. This report demonstrates its use for quick and reliable crystallite size determination.

X-ray powder diffraction is a fast method for determining the average size of crystallites. The crystallinity of petroleum coke (expressed as L_c value) is a measure of quality affect-

ing suitability for the end use of the coke, and is a function of the heat treatment of the coke. The ASTM norm D 5187 describes how to obtain the crystallinity of coke by evaluating the shape of a carbon X-ray peak. This peak is scanned over a wide range and the L_c value calculated from the full width of the X-ray peak at half maximum intensity.

A coke powder specimen was prepared according to ASTM D 5187 and measured with the D2 PHASER. Experimental details are summarized in Table 1. Figure 1 shows a typical diffraction scan from petroleum coke. After automatic subtraction of the base line, DIFFRAC.EVA estimates the crystallite size by means of the Scherrer equation from the full width of the peak at half maximum.

The data presented in figure 1 exceeds the maximum peak intensity (in cps) shown in ASTM D 5187 by a factor of 50. Notably, this data was collected in less than 1 min, compared to 20 min mentioned in the norm. The 1-dimensional LYNXEYE detector makes this intensity and speed gain possible, even for a low powered XRD system.

To conclude, our cost-effective desktop XRD system D2 PHASER allows for extremely fast and precise quality control of anode coke material.

Tab. 1: Experimental settings.

D2 PHASER, LYNXEYE detector
Cu radiation (30 kV, 10 mA), Ni filter
Continuous scan from 14.0 to 38.0° 2Theta Step width 0.2° counting time 0.1 sec per step
Total scan time about 30 sec.
2.5° Soller collimators, 1.0 mm divergence slit, anti-scatter screen
LYNXEYE detector opening 5° 2Theta

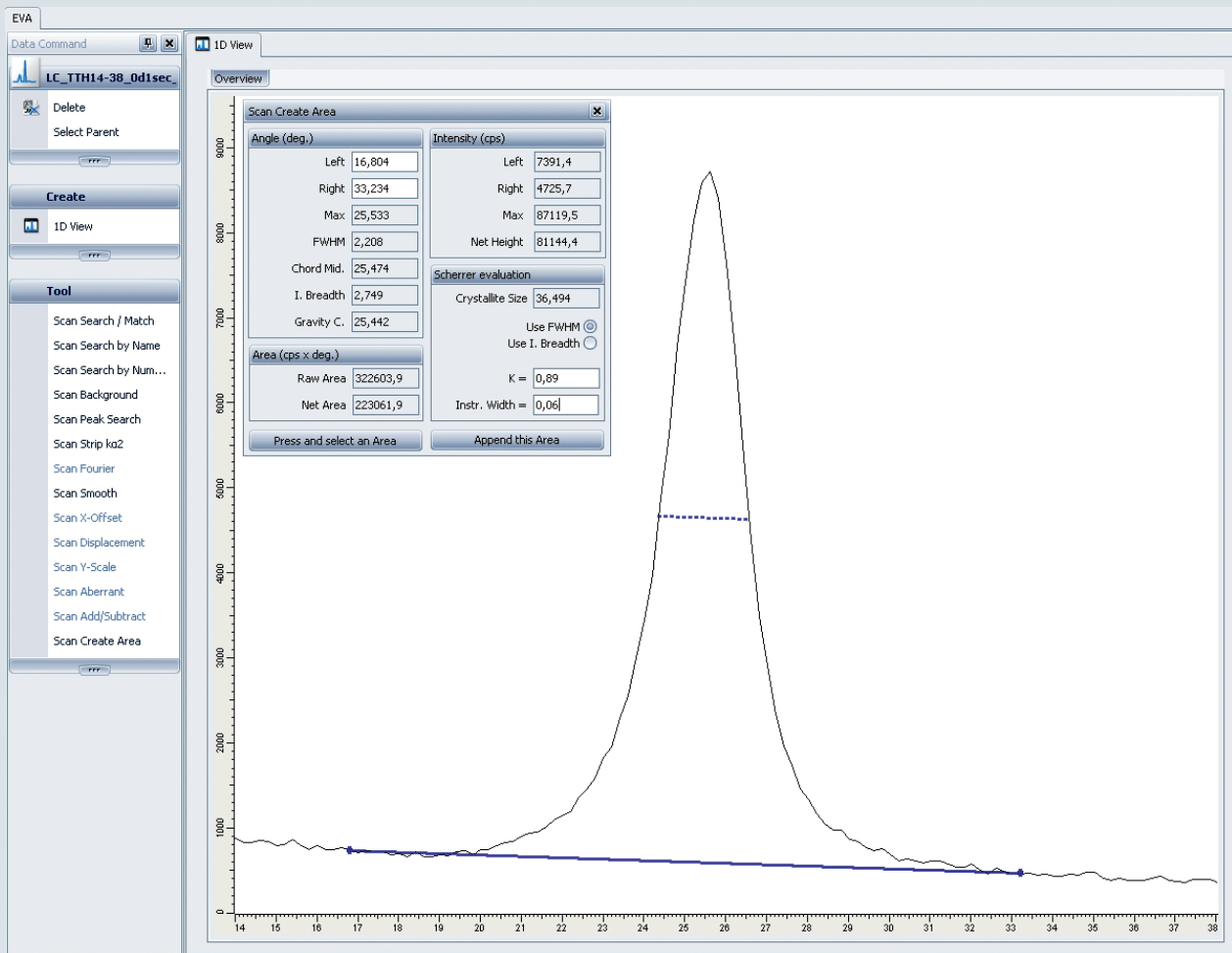


Fig. 1: Determination of the full width at half maximum of the 002 peak of petroleum coke using DIFFRAC.EVA. The software evaluates the peak shape and automatically calculates the crystallites size (36.5 Å) using the Scherrer equation.

All configurations and specifications are subject to change without notice. Order No. DOC-R88-EXS007. © 2009 Bruker AXS. Printed in Germany.

● **Bruker AXS GmbH**
 Karlsruhe, Germany
 Phone +49 (7 21) 5 95-28 88
 Fax +49 (7 21) 5 95-45 87
 info@bruker-axs.de
 www.bruker-axs.de

Bruker AXS Inc.
 Madison, WI, USA
 Phone +1 (800) 234-XRAY
 Phone +1 (608) 276-3000
 Fax +1 (608) 276-3006
 info@bruker-axs.com
 www.bruker-axs.com