

Determination of L_c in calcined petroleum coke according to ASTM D5187 with ARL EQUINOX 100 X-ray diffractometer

Introduction

Calcined Petroleum Coke (CPC) is produced from “green” petroleum coke, which is a byproduct of oil refining. The electrodes used in metal smelting industry are usually made of CPC, because of the sponge-like structure which improves the contact between electrode and metal. The electrical conductivity of the CPC material is strongly influenced by the crystallite size and therefore also the quality of the electrodes, which limits the effectivity of the whole smelting process. For this reason it is crucial to evaluate the crystallite size of the raw CPC material.

Instrument

Thermo Scientific™ ARL™ EQUINOX 100 employs a custom-designed Cu (50 W) or Co (15 W) micro-focus tube with mirror optics. The low power consumed by the unit allows it to be completely transportable, not requiring an external water chiller. The same unit is capable of being transported between laboratories without the need for special infrastructure.

The ARL EQUINOX 100 provides very fast data collection times compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time and is therefore well suited for both reflection and transmission measurements (Figure 1).

Experimental

An A508 graphite anode sample from RD-Carbon was measured in reflection mode. The sample was analyzed under Cu K α (1.541874 Å) radiation for 2 minutes with the sample rotating during the analysis. L_c was determined from the FWHM of the graphite [002] reflection using MAUD, JADE and according to the method described in ASTM D5187.

Results

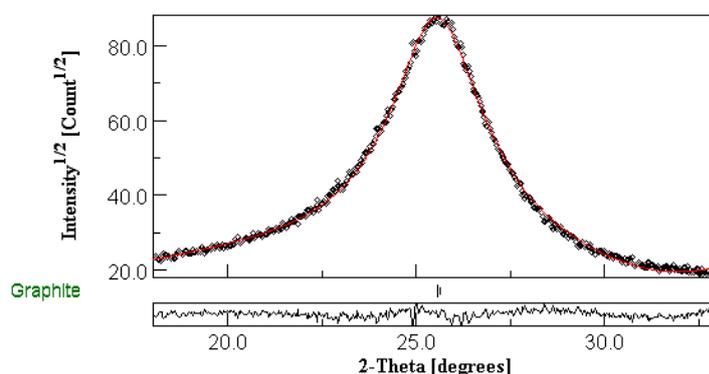
Fitting the [002] reflection of graphite using Rietveld's method implemented in MAUD and JADE yields the mean crystallite size of the graphite particles. Both programs additionally

Figure 1: ARL EQUINOX 100 X-ray diffractometer



account for the influence of micro-strain. The methods according to ASTM D5187 uses a simplified version of the Scherrer equation. All three methods yield similar results (MAUD: 48.1 Å; JADE: 42.0 Å; ASTM D5187: 18.5 Å), whereas the differences are most likely due to slightly different algorithm used by the programs and the susceptibility to errors of the FWHM determined according to ASTM D5187.

Figure 2: Rietveld fit (MAUD) of graphite [002] reflection



Conclusion

The ARL EQUINOX 100 is a suitable instrument to evaluate the crystallite size in CPC samples using Rietveld's method with MAUD and JADE as well as according to ASTM D5187.

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