Transactions, **SMiRT-25**
Charlotte, NC, USA, August 4-9, 2019
Division I

CHARACTERIZATION OF VIRGIN ZXF-5Q AND AXF-5Q POCO GRAPHITE

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ABSTRACT

Graphite is an attractive choice for neutron moderation in nuclear reactors because of its small neutron absorption cross-section and high scattering efficiency. In this work, we perform Raman spectroscopy on virgin (un-irradiated) samples from two grades of POCO graphite – AXF-5Q and ZXF-5Q.

INTRODUCTION

In one of the designs of the proposed Very High Temperature Reactors (VHTRs), the reactor core is made of prismatic blocks of graphite. POCO graphite grades AXF-5Q and ZXF-5Q are potential candidates as moderating elements in VHTRs. The main difference between the two grades is the particle size; ~5 microns in diameter for the former (AXF-5Q, 1997), and ~1 micron for the latter (ZXF-5Q, 1997). In order to obtain base knowledge of both graphitic grades, we perform Raman spectroscopy to generate the characteristic fingerprint patterns of molecular structure and crystallinity.

RAMAN SPECTROCOPY

In Raman spectroscopy, a laser light source is used to irradiate a sample to generate a small amount of scattered light, which is detected as the Raman spectrum. Samples are first cut and mechanically polished, and then analyzed with Horiba XploRA PLUS Confocal Raman Microscope, available at the Analytical Instrumentation Facility (AIF) at NC State University. The parameters used in the spectroscopy analysis are listed in Table 1.

Table 1. Parameters used in the Raman spectroscopic analysis.

Filter	50%	**Objective Lens**	x100_VIS	**Laser**	785nm_Edge
Acq. time (s)	40 – 50	**Accumulations**	10 – 20	**Grating**	1200 (750nm)
Hole (µm)	500	**Slit (µm)**	100	**Range (cm^{-1})**	500 - 3500

AXF-5Q POCO Graphite

Figure 1 depicts the Raman spectrum for AXF-5Q grade. The main G peak at 1575 cm^{-1} arises from a single resonance process, proportional to the number of planar ring-like sp^2 carbon sites (Reich & Thomsen, 2004). Graphite with less crystallinity generates more peaks (besides the G peak), due to different types of defects present in the sample. For example, the peak at 1370 cm^{-1} is named the D peak, for disorder-induced modes. The D peak, which arises from the A_{1g} breathing mode, is attributed to an elastic scattering process primarily emanating from ring-like topological defects (Eapen, Krishna, Burchell, & Murty, 2014). The G′ peak, present at 2600 cm^{-1} is found to be an overtone to the D peak, where the electron backscatters by a second phonon; it lies exactly at twice the energy with double the slope of the D peak (Reich & Thomsen, 2004). The D′ peak, on the other hand, is attributed to an inelastic scattering event due to phonon absorption or emission (Zhang, et al., 2016). Both D and D′ peaks are associated with crystallite boundaries.

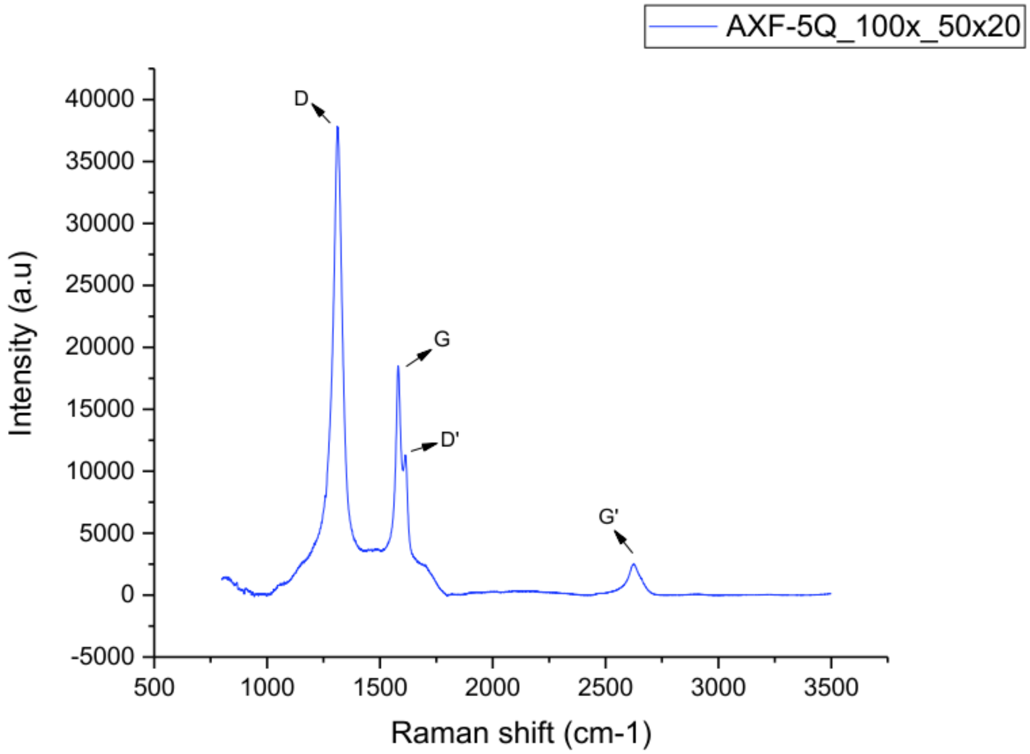

Figure 1. Raman spectra for AXF-5Q grade.

It is known that the relative intensity I_D/I_G gives a measure of the size of the crystallites (Ferrari, 2007). The crystallite size (L_a) can be calculated using the Tuinstra–Koenig (TK) empirical formula (Tuinstra & Koeing, 1970), which is given by:

$$L_a = C(\lambda)\left(\frac{I_D}{I_G}\right)^{-1} \tag{1}$$

where C, which is a constant dependent on the wavelength of the incident light, is $\approx 4.4\ nm$ here. Thus, the characteristic length-scale in graphitic carbon is inversely proportional to the I_D/I_G ratio. The TK relationship is generally valid for crystallite sizes greater than 2 nm (Zhang, et al., 2016). From Figure 1, for the relative intensity of I_D/I_G = 2.04, the crystallite size calculated from TK equation is 2.16 nm for the AXF-5Q grade.

ZXF-5Q POCO Graphite

As seen in Figure 2, the characteristic peaks of AXF-5Q are also present in the ZXF-5Q grade, except at different intensities. While the absolute intensities of the peaks are not pertinent, their relative intensities carry useful information. Interestingly, the relative intensity I_D/I_G for ZXF-5Q of 0.51 is smaller than unity, and it corresponds to a crystallite size of 8.62 nm through the TK equation. Thus the characteristic crystallite size is much larger than that of AXF-5Q implying fewer boundary defects in the ZXF-5Q grade.

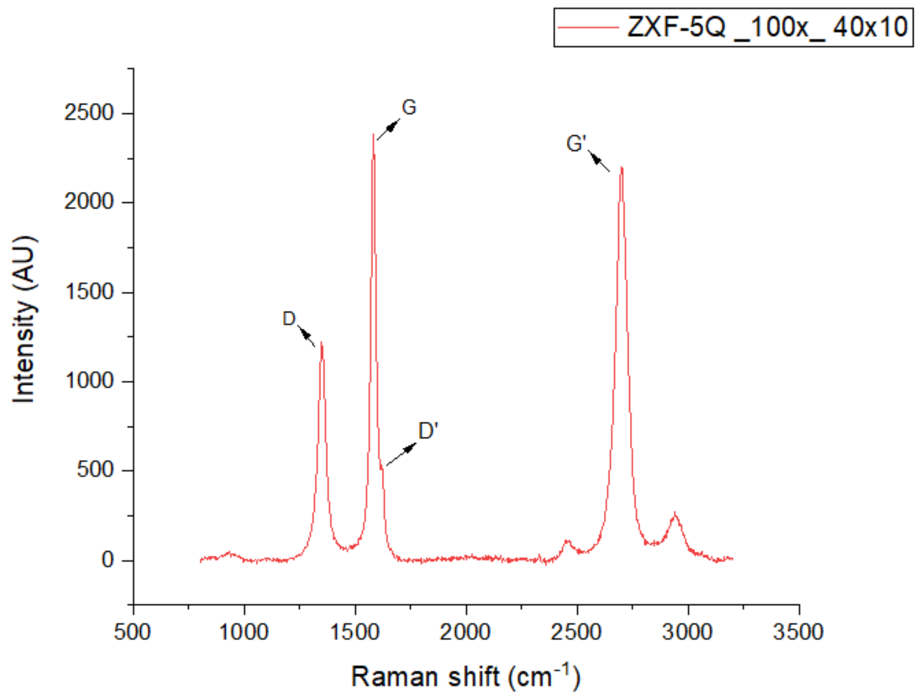

Figure 2. Raman spectra for ZXF-5Q grade.

CONCLUSION

In this work, we have performed Raman spectroscopy on virgin ZXF-5Q and AXF-5Q graphite grades. Spectral characteristics of the two grades reveal the existence of fewer boundary defects in ZXF-5Q than in AXF-5Q, as evidenced by the smaller I_D/I_G ratio and the inferred larger crystallite size in the former.

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