Spectroscopy (Raman, XPS, and GDMS) and XRD analysis for studying the interaction between nuclear grade graphite and molten 2LiF-BeF₂ (FLiBe) at 700 °C

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Abstract

FLiBe-exposed IG-110 graphite and a control IG-110 sample were analyzed by Raman, XPS, GDMS, and XRD, and the complete raw data sets are provided in the Supplementary Information. These data sets enable full reproducibility and transparency of the data analysis we reported in the accompanying research paper titled “Fluorination of Nuclear Graphite IG-110 in Molten FLiBe salt at 700 °C”, published in the Journal of Fluorine Chemistry, and facilitates quantitative comparison with future similar studies by other research groups. In this data article, we provide plots of the peak fitting for all Raman spectra from each sampling point on the graphite surface. We also provide the measured impurity concentrations of the IG-110 samples, as measured by GDMS; this data was not reported nor discussed in the accompanying research paper. The method and software used for peak fitting for the spectra from Raman, XPS, and XRD are listed separately.

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Specifications Table

| Subject area                           | Engineering/Nuclear Engineering/Material Science |
|----------------------------------------|--------------------------------------------------|
| More specific subject area             | Microstructural analysis and elemental analysis of graphite |
| Type of data                           | Table, graph, figure                             |
| How data was acquired                  | Raman spectroscopy                               |
|                                        | X-ray Diffraction (XRD)                          |
|                                        | X-ray photoelectron spectroscopy (XPS)           |
|                                        | Glow Discharged Mass Spectrometry (GDMS)         |
| Data format                            | Raw data and peak-fitted plots                   |
| Experimental factors                  | Nuclear grade graphite IG-110, and FLiBe-exposed IG-110 |
| Experimental features                 | Elemental analysis and crystal structure analysis were performed to study the chemical interaction between IG-110 graphite and molten FLiBe |
| Data source location                  | Raman Spectra was recorded using a Thermo Scientific DXR Raman microscope in Soft Material Lab (SML), UW-Madison |
|                                        | XRD diffraction pattern was recorded using a Bruker D8 Discover X-ray Diffractometer in Material Science Center (MSC), UW-Madison |
|                                        | XPS spectra were recorded using a Thermal Scientific K-alpha X-ray photoelectron spectrometer (XPS) in Material Science Center (MSC), UW-Madison |
|                                        | GDMS was performed by the EAG laboratories |
| Data accessibility                    | Raw data is shared in the Supplementary Information |
| Related research article              | This data article is submitted as a companion paper to a research article: “Fluorination of Nuclear Graphite IG-110 in Molten FLiBe salt at 700 °C”, the research article is submitted to Journal of Fluorine Chemistry [1]. |

Value of the data

- All raw data are presented in this data article as Supplementary Information, which helps the reader to perform their own data analysis with the raw data sets.
- The method used to analyze the data is described in this data article, which helps the reader to reproduce the peak fitting of the XPS, XRD and Raman spectra.
- The full data and data analysis shown in this data article and in the Supplementary Information can be used for quantitative comparison with data from other studies of similar materials exposed to different experimental conditions.

1. Data

The interaction between IG-110 and fluoride salt FLiBe (2LiF-BeF2) was studied by immersing graphite samples into molten FLiBe at 700 °C and 1 atm for 12 h. The IG-110 sample was then taken out for elemental analysis (XPS, GDMS) and crystal structure analysis (Raman, XRD). This article reports the detailed data analysis from Raman and GDMS, and a repeatability test for XPS, as a supplemented data article to the journal paper published in Journal of Fluorine Chemistry [1]. The raw data for Raman, XPS, XRD, GDMS are reported in the Supplementary Information.
2. Experimental design, materials, and methods

Control sample of the same shape and similar size was prepared from the same type of graphite. Both the control and test sample were baked under 700 °C for 3 h to remove moisture and gaseous impurities. The IG-110 test sample was characterized together with IG-110 control sample.

Both samples were characterized by Raman spectroscopy, XPS, GDMS and XRD. All spectra from Raman, XPS and XRD were normalized and peak fitted for peak location, peak height, and FWHM by different software and different methods. Peak fitting of Raman normalized spectra were performed in PeakFit v4 software. The software was used to perform data smoothing and background
Fig. 2. Raman spectra peak fitting plot for test sample (8 points).
subtraction, then peak fitting with Gaussian function. XRD diffraction data were first analyzed using Diffrac.Eva v3.1 software for stripping the Kα2 component and removing background. The diffraction patterns were normalized and peak fitted by PeakFit v4 software using a deconvolution method. Data processing of XPS spectra were performed by SDP v7.0, a specialized software from PS International. The software is designed for XPS spectra analysis using Gaussian function peak-fitting.

The original data from these characterization techniques are shared as Supplementary Information.

2.1. Raman spectra peak fitting

Figs. 1 and 2 are the peak fitted Raman data for control sample and test sample. Raman spectroscopy for control sample were performed at five points on the same surface and Raman spectroscopy for test sample were performed at eight points on the same surface. Each spectrum was peak fitted separately. Peak location, amplitude and FWHM were recorded.

2.2. GDMS data analysis

Fig. 3 shows the impurities in graphite that decreased in concentration after FLiBe exposure, reported in wppm and μmol/g. Fig. 4 shows the elements that increased in concentration after FLiBe exposure, reported in wppm and μmol/g.

![Graphite impurities that decreased in concentration after salt exposure.](image1)

![Graphite impurities that increased in concentration after salt exposure.](image2)
3. XPS repeatability check (F 1s peak)

Fig. 5 is the normalized F 1s spectra from two different points of the surface of test sample. The FWHMs and peak locations of the two spectra are shown in the plot.

Acknowledgments

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Transparency document. Supporting information

Transparency data associated with this article can be found in the online version at https://doi.org/10.1016/j.dib.2018.08.079.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at https://doi.org/10.1016/j.dib.2018.08.079. The supplementary data includes: 1) GDMS_IG110; 2) Mercury Porosimetry_IG110; 3) XRD raw data_IG110; 4) Raman raw_IG110 Test Sample; 5) Raman raw_IG110 Control Sample; 6) XPS raw_IG110 Test Sample; 7) XPS raw_IG110 Control Sample; 8) DG for possible reactions.

Reference

[1] H. Wu, F. Carotti, et al., ‘Fluorination of nuclear graphite IG-110 in molten 2LiF-BeF2 (FLiBe) salt at 700 °C’, J. Fluor. Chem. 211 (2018) 159–170.