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Crystal damage and porosity evolution in proton irradiated POCO graphite

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We present post irradiation examination (PIE) results of a piece of fractured POCO ZXF-5Q graphite fin that was extracted from the NuMI beamline of U.S. FermiLab. This piece of specimen has been irradiated by 340 kW, 120 GeV pulsed protons for producing neutrinos for MINOS/MINERvA high energy physics experiment use. PIE was conducted on this specimen using a novel approach with micro-Raman spectroscopy to quantify total graphite crystal damage evolution across proton irradiation damage gradient. Alongside with this, porosity evolution across this gradient was examined by six high resolution focused ion beam-scanning electron microscopy tomography (FIB-SEM tomography). Results from both techniques purvey notable findings.
Specifically, G-band positions derived from a large number of spectra collected from the POCO graphite specimen possessing proton irradiation damage gradient were plotted with open literature Raman data on HOPG, BEPO, PCEA and IG-110 graphite as a function of carefully calibrated dpa levels. The total damage level within 2σ beam radius in this POCO graphite was estimated to be equivalent to ~ 2 - 5 dpa at ~ 350 - 370℃. Derived G-band positions were then mapped to the three-stage amorphization trajectory model indicating beam centre area has entered the second stage, i.e., transitioning from nanocrystalline graphite into amorphous carbon. G-band position relative shift (⊠G) curve with a 'turn-around' peak as a function of total damage can be used for damage monitoring and lifetime prediction at proton beamline in future. The developed methodology has the potential to 'unify' total damage levels across different grades of nuclear graphite subjected to different irradiation species including ions, neutrons and protons at different temperatures.
Porosity studied by FIB-SEM tomography from six locations across damage gradient has been segmented and quantified with a deep learning-based tomographic image segmentation technique. It has been found that there is a decrease in the total volumetric percentage of the porosity at proton beam centre (~ 8 - 8.4 vol.%), by comparing to un-irradiated POCO (~ 12 - 13 vol.%) and to beam 2σ and 5σ radii (~ 12 vol.%). This decrease in porosity volume percentage was found to be caused by the reduction in pores with volumes > 0.1 µm3. The underlying mechanism causing this reduction is not clear in such complicated proton beamline irradiation environment and further investigations are still needed.

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