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In-situ electric resistance measurements and annealing effects of graphite exposed to swift heavy ions

Sandrina Fernandes [a], Frederique Pellemoine [a,*], Marilena Tomut [b,c], Mikhail Avilov [a], Markus Bender [b], Marine Boulesteix [a], Markus Krause [b,d], Wolfgang Mittig [e], Mike Schein [a], Daniel Severin [b], Christina Trautmann [b,d]

[a] Facility for Rare Isotope Beams, Michigan State University, East Lansing, MI, USA
[b] GSI Helmholtzzentrum für Schwerionenforschung, Darmstadt, Germany
[c] National Institute for Materials Physics (NIMP), Bucharest, Romania
[d] Technische Universität, Darmstadt, Germany
[e] National Superconducting Cyclotron Lab (NSCL), Michigan State University, East Lansing, MI, USA

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ABSTRACT

To study the suitability of using graphite as material for high-power targets for rare isotope production at the future Facility for Rare Isotope Beams (FRIB) in the USA and at the Facility for Antiproton and Ion Research (FAIR) in Germany, thin foils of polycrystalline graphite were exposed to 8.6-MeV/u Au ions reaching a maximum fluence of 1×10^{15} ions/cm^2. Foil irradiation temperatures of up to 1800 °C were obtained by ohmic heating. In-situ monitoring of the electrical resistance of the graphite foils during and after irradiation provided information on beam-induced radiation damage.

The rate of electrical resistance increase as a function of fluence was found to decrease with increasing irradiation temperature, indicating a more efficient annealing of the irradiation-produced defects. This is corroborated by the observation that graphite foils irradiated at temperatures below about 800 °C showed cracks and pronounced deformations, which did not appear on the samples irradiated at higher temperatures.

1. Introduction

Rare isotope production at the Facility for Rare Isotope Beams (FRIB) [1] in the USA and at the Facility for Antiproton and Ion Research (FAIR) [2] in Germany relies on fast heavy ion beams impinging on high-power production targets and in-flight separation of the reaction products. The realization of a production target is challenging, because the beam will produce very high-power densities in the target material. These are caused by the combined effects of high energy-loss of heavy ions passing though the target material and the need for a small beam spot to achieve high resolution separation of reaction products. For both facilities, FRIB and FAIR, a radiation-cooled rotating solid carbon wheel was chosen as suitable target concept [3,4]. For FRIB, a multi-slice graphite target is being developed in order to meet the extreme requirement of operating a 200 MeV/u uranium beam of 400 kW being focused in a spot of 1 mm in diameter. For a graphite static target this corresponds to power densities of up to 60 MW/cm^3 [3].

In addition to the high-power density challenge, continuous irradiation of the target material with swift heavy ions will result in radiation damage of the graphite material, leading to changes in its structural and thermo-mechanical properties and limiting target lifetime. This lifetime may also depend on the specific grade of graphite material, its manufacturing process, and the operational temperature. Overheating and excessive thermo-mechanical stress load variations caused by the high beam intensity and high rotation speed can lead to fatigue [5] and creep failure [6] effects. Successful operation of high-power production graphite targets at FRIB and FAIR will benefit from an improved understanding how graphite material properties change with heavy-ion irradiation. It is known that radiation damage effects produced by neutrons can be controlled to some extent by annealing graphite during or after irradiation. This annealing process has been shown to promote thermal diffusion and defect recombination. Previous studies demonstrating these effects have mostly been performed at temperatures below 1300 °C [7–9].

This paper presents irradiation experiments on thin graphite foils bombarded with an intense high-energy (1.7 GeV) gold beam reaching fluences up to 1×10^{15} ions/cm^2 at the GSI UNILAC accelerator. Additional ohmic heating of the samples was used to attain foil irradiation temperatures of up to 1800 °C during irradiation. Data from electrical resistance measurements before, during, and after irradiation, combined with thermal simulations provide

* Corresponding author.
E-mail address: pellemoi@frib.msu.edu (F. Pellemoine).

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new insights into graphite performance under ion beam irradiation conditions similar to those expected at the FRIB and FAIR facilities.

2. Experiment

2.1. Experimental set-up

The experimental set-up allows in-situ electrical resistivity measurement of a thin graphite target made from a foil of purified polycrystalline isotropic graphite (grade 2360, supplied by Mersen USA St. Marys – PA Corp with a bulk density of 1.89 g/cm^3) during ion bombardment (Fig. 1a), by using the 2-point probes method. The foils had a nominal thickness of 74 μm, a width of about 1.2 cm, and a total length of 2.54 cm. The apparatus consists of a water-cooled vacuum chamber with a quartz-window viewport for temperature measurements with a thermal camera. The target holder is mounted on a flange with two insulated high current vacuum feedthroughs. A tantalum beam dump behind the target stops the ions after passing through the graphite foil (Fig. 1b). The beam current, pressure in the irradiation chamber, DC power supply current, and voltage were monitored using Labview software.

The sample holder for the graphite foil was directly mounted on the leads of the current feedthrough as shown in Fig. 1c. A high-purity graphite laminate, Grafoil [10], served as spacer between the sample and the copper holder, also providing good electrical and thermal contact. The electrical total resistance was measured in-situ by determining the voltage drop across the sample as a function of the applied current. To get an accurate electrical resistance measurement without ohmic heating, a low current of 1 A was applied, limiting the temperature increase of the sample to a value of less than 20 °C. During the irradiation, the electric current for ohmic heating was adjusted to the desired irradiation temperature by applying currents up to 35 A.

The temperature distribution of the graphite samples was mapped before irradiation using a non-contact near-infrared thermal imaging camera, MIKRON M9200 [11] connected to a PC with MikroSpec RT9200 software (3.9200.117) supplied by LumaSense Technologies Inc. During irradiation the temperature of the samples was not measured due to concerns regarding the radiation hardness of the thermal camera.

2.2. Heavy-ion beam irradiations

The irradiation experiments were performed at the M3-beamline of the UNILAC accelerator facility at GSI [12] using a ^{197}Au beam of 8.6 MeV/u specific energy. According to calculations with the SRIM-2008 code [13] this beam energy is large enough that the projectiles do not stop in the foils. The linear rate of energy loss varies between 20 and 25 keV/nm along the ion trajectory in the sample.

The beam was pulsed at a rate of 42 Hz with a pulse length of 3 ms. To obtain a well-defined and uniform ion exposure area on the graphite foil the ion beam was adjusted to pass through a mask with a 10 mm × 10 mm aperture. The ion flux was between 3 and 4×10^{10} ions/cm^2·s. The highest accumulated fluence was 1×10^{15} ions/cm^2. A total of four samples were irradiated at different fluences and temperatures. The parameters of the different samples and of the irradiations are presented in Table 1.

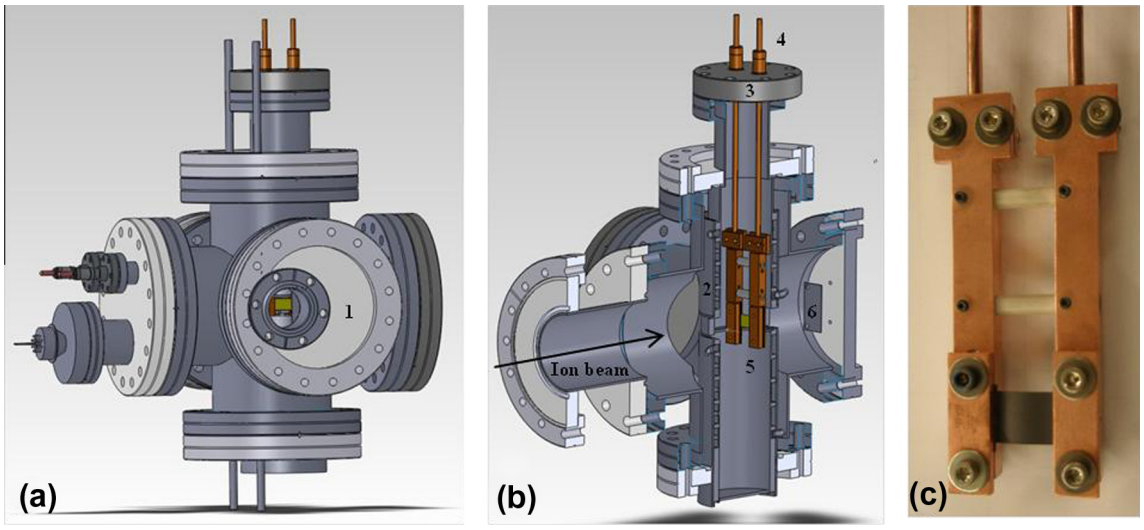

Fig. 1. Experimental setup for irradiation of graphite foils in combination with in-situ electrical resistance measurement. (a) Vacuum chamber with view port for thermal imaging (1). (b) Cross section of vacuum chamber displaying the water cooled system (2), sample holder flange (3), current feedthroughs (4), sample holder (5), and tantalum beam dump (6). (c) Photo of copper sample holder with a mounted graphite foil (beam side).

Table 1
Sample and their irradiation parameters. The sample name denotes the current (in A) for ohmic heating applied during irradiation. The uncertainty of dimensions *a* and *c* is about 0.05 cm.

Sample	Mean flux (ions/cm^2 s)	Fluence (ions/cm^2)	Foil thickness *e*[a] (μm)	Foil width *a* (cm)	Irradiated area Height *c* (cm)
1 A	4.3×10^{10}	10^{14}	77.1 (2.0)	1.24	0.92
11 A	3.1×10^{10}	10^{14}	72.6 (0.5)	1.22	0.88
25 A	4.2×10^{10}	10^{14}	77.6 (0.5)	1.18	0.56
35 A	3.9×10^{10}	10^{15}	77.2 (0.5)	1.20	0.85

[a] Measured by contact profilometry.