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The impact of neutron radiation and high temperatures on graphene-based magnetic field sensors

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I. INTRODUCTION

Magnetic confinement thermonuclear reactors are considered the power source of the future. One of the most promising candidates for practical fusion reactors is the tokamak, implemented through large-scale projects such as ITER or DEMO. The primary challenges these projects face are of an engineering nature, one of which involves magnetic field diagnostics [1].

Existing tokamaks are designed for short-pulse operation, where inductive sensors effectively monitor the magnetic field. However, as research progresses toward long-pulse and steady-state operation, inductive sensors become inadequate due to spurious voltages induced by high neutron fluence. They lead to significant measurement errors during time integration over long pulses. To address this issue, Hall effect sensors are being explored as a more viable alternative [1, 2]. Their objective is to measure the magnetic field of magnetic induction 6 – 12 T in an AC regime. The primary challenges for any magnetic sensor in such an environment include exposure to intense neutron radiation (with cumulative fluence ranging from 10^{18} – 10^{22} n/cm^2 over a five-year period) and operating temperatures reaching up to 350°C [3].

For this purpose, metallic and semiconductor-based hallotrons have been proposed [4 – 8]. In the case of metallic-based sensors, issues with the temperature dependence of the Hall constant and the low-level Hall signal must be resolved. More recently, research has emerged on graphene-based magnetic field sensors for tokamaks [9, 10]. Graphene is particularly attractive due to its 2D structure, which makes it nearly transparent to neutron radiation. However, depending on the fabrication method, sensor characteristics vary significantly. One approach involves exfoliating graphene and transferring it onto a substrate, a quick and inexpensive technique that unfortunately yields worse results compared to direct monolayer growth on the substrate. The latter method – epitaxial growth, allows for the fabrication of high-quality and reproducible samples [11, 12]. The primary issue with this technique is the strong interaction between the graphene layer and the substrate, which reduces its electrical properties. This problem is mitigated by intercalating the sensors with hydrogen atoms, resulting in quasi-free-standing graphene (QFS) [13].

Using the infrastructure of the National Centre for Nuclear Research (Poland), it was possible to expose epitaxial graphene on silicon carbide (G/SiC) to the fluence of fast neutrons. The studies showed that a fluence of 0.7×10^{18} n/cm^2 affected the electrical properties of the graphene. However, thermal treatment above 200°C resulted in partial recovery of these properties. Notably, thermal treatment without prior irradiation had no significant effect on the electrical characteristics of the sensors exhibiting excellent thermal stability up to 770 K [14, 15].

Further studies explored the effects of different neutron fluences on the graphene. Exposure to a lower fluence of 0.2×10^{18} n/cm^2 followed by thermal treatment resulted in complete recovery of the electrical properties. In contrast, exposure to a higher fluence of 2.0×10^{18} n/cm^2 led to permanent degradation that could not be reversed by thermal treatment [16]. The degradation observed at higher fluences is attributed to the depletion of the hydrogen intercalation layer during irradiation, which causes the graphene to rebond with the substrate. At elevated temperatures, residual hydrogen atoms can diffuse across the surface, potentially restoring separation in mildly damaged samples. This effect, called effective self-healing, has, however, a fluence threshold above which it does not occur [16]. These findings are consistent with Density Functional Theory (DFT) calculations, which verified the mechanism of effective self-healing effect, proposed based on experimental findings [9, 16].

This paper presents new findings on the electrical characterisation of epitaxial graphene-based radiation-resistant magnetic field sensors following exposure to a neutron fluence of 4.0×10^{18} n/cm² — twice the highest fluence previously examined. The collected results have been framed in the context of existing research.

Additionally, the paper outlines future research directions aimed at further investigating the performance of these sensors under varying radiation conditions and exploring ways to enhance their resilience for practical applications.

II. METHODOLOGY

This section provides a detailed description of sample preparation, irradiation procedures, and electrical measurements.

A. Sample Preparation

Epitaxial graphene was synthesized on a semi-insulating silicon carbide (SiC) substrate using chemical vapour deposition (CVD), followed by in situ hydrogen intercalation to achieve a quasi-free-standing graphene (QFS) system. Propane served as the carbon precursor, and two SiC polytypes, high-purity 4H-SiC(0001) and vanadium-compensated 6H-SiC(0001), were utilized to examine variations in electrical properties [11, 12]. The substrate was processed into a 1.6 mm × 1.6 mm Hall effect structure featuring a cross-shaped 100 µm × 300 µm graphene mesa and four Ti/Au (10 nm/ 60 nm) ohmic contacts, all passivated with a 100-nm-thick aluminium oxide (Al_2O_3) layer synthesized from trimethylaluminum (TMA) and deionized water using Atomic Layer Deposition (ALD) method. A diagram of the structure is presented in Figure 1.

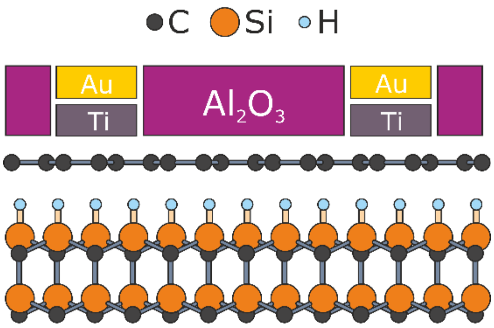

Figure 1. Diagram of the G/SiC sensor structure. Atomic layers of graphene, hydrogen intercalation, and surface layers of the substrate are marked with spheres. Bulk materials, such as the protective Al_2O_3 layer and gold pads with a titanium buffer layer, are marked with blocks.

On the edges of the graphene cross, the protective layer was etched with acid to form L-shaped ohmic contacts. This allows connecting the structure to 10 mm x 10 mm sapphire holders with golden wires. This step facilitates manual electrical characterisation.

One of the criteria for selecting individual components of the system was the activation time of each material after irradiation. Should the duration of deactivation last over a couple of months it disqualifies certain materials. For that reason e.g. silver, commonly used in electronics, cannot be utilized in this application.

B. Irradiation process

The neutron irradiation (NR) was performed using the MARIA research nuclear reactor. MARIA is a multi-purpose research reactor with 30 MW of nominal thermal power. It is used for scientific purposes as well as radionuclide production and for industrial applications. The neutron energy spectra range from thermalized (approx. 25 meV) up to fast neutrons (1-2 MeV) with respective fluxes of up to 2.5×10^{14} $cm^{-2}s^{-1}$ and 1.0×10^{14} $cm^{-2}s^{-1}$. In addition, there is a special facility located next to the reactor core where thermal neutron flux is significantly reduced compared to the centre of the core. This helps mimick the conditions inside fusion devices, where fast neutrons constitute a majority of the spectrum.

The sample was placed inside a quartz tube separated with quartz wool. Glass ampules were then put inside a metal capsule, which was tightly closed after filling up with helium. This step helps with leakage detection and enhances heat exchange. Lastly, a metal capsule was placed inside a collective metal container such that the maximum vertical distribution of fast neutron fluence during the irradiation was obtained. The properly sealed container was then placed inside the special structure where samples were irradiated. Each type of container used along the way is shown in Figure 2.

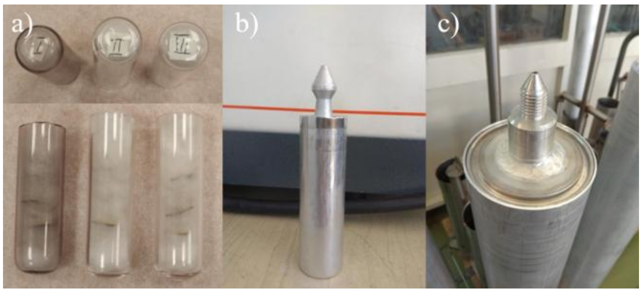

Figure 2. Overview of following steps required before irradiating samples in research nuclear reactor a) quartz tube, b) metal capsule, c) large collective container.

The combination of selecting a specific location and determining the duration for which the samples were placed there allowed for their exposure to fast neutrons of approx. 4.0×10^{18} n/cm^{-2} fluence. After extracting the ampule from metal containers it has been deactivating for about a year before further testing.

C. Electrical Measurements

Before irradiating G/SiC system was subjected to 4-point Hall effect measurements at room temperature (RT). For this purpose, Linseis Analyser L79 HCS 1 equipped with two permanent magnets of a magnetic field induction equal to ± 0.65 T, was used. Measurements were carried out using the van der Pauw method which allows for characterisation of electrical parameters like sheet resistance, hall coefficient, sheet carrier density, and carrier mobility.

Following neutron exposure, the same measurement protocol was applied to assess radiation-induced changes. Additionally, thermal annealing cycles were performed to investigate potential recovery effects. The annealing process consisted of five cycles, each beginning at room temperature (RT). In each cycle, the temperature was incrementally increased in 10°C steps until reaching a maximum temperature, which was 50°C higher than the maximum temperature of the previous cycle. The final cycle reached a maximum of 350°C. Once the maximum temperature was attained, the samples were annealed for 30 minutes before cooling back to RT, during which electrical measurements were conducted. The results of these analyses are presented in Section III.

III. DISCUSSION ON IMPACT OF NEUTRON RADIATION AND TEMPERATURE

In this section, the results of the electrical characterisation of G/6H-SiC sensor after irradiation in fast neutron fluence of 4.0 × 10^{18} n/cm^2 are shown. The results are compared to findings from the paper [9] describing the impact of neutron irradiation dose of 0.7 × 10^{18} n/cm^2 on the G/4H-SiC sensing platform. These findings were selected for comparison as they showed an intermediate irradiation dose after which, partial effective self-healing occurs under temperature treatment. Other fluences considered resulted in complete self-healing (0.2 × 10^{18} n/cm^2) and no effective self-healing (2.0 × 10^{18} n/cm^2) despite annealing [16].

Figure 3. compares sheet resistance measurements of two sensors (one grown on 4H-SiC and the other on 6H-SiC substrate) after irradiating in neutron fluence of 0.7 × 10^{18} n/cm^2 and 4.0 × 10^{18} n/cm^2. The blue diamond marks the average sheet resistance of a sample before irradiation. The first measurement after irradiation is shown as a red star. Black dots represent RT values after each cycle of temperature treatment.

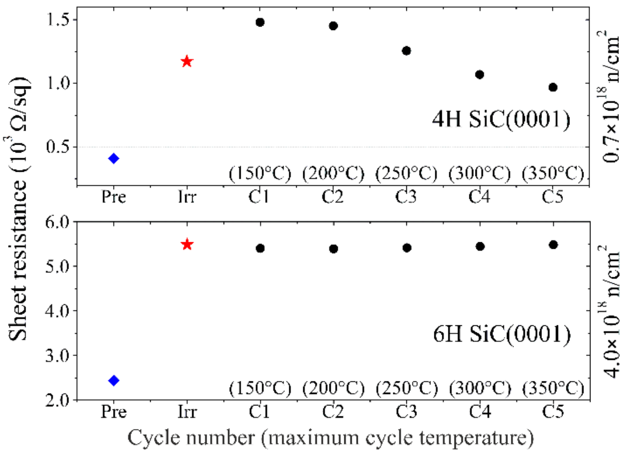

Figure 3. Sheet resistance measurements of irradiated G/6H-SiC structure compared to described in literature G/4H-SiC structure. Each sample was exposed to neutron radiation of respective fast neutron fluence of 4.0 × 10^{18} n/cm² and 0.7 × 10^{18} n/cm². The pre-irradiation sheet resistance value is represented by a blue diamond, while the first measurement at room temperature after irradiation is marked with a red star. Black dots indicate sheet resistance values measured at room temperature following a thermal cycling process, with annealing up to the respective temperatures specified in parentheses.

Initial values of the G/SiC sensors sheet resistance depend on substrate polytype and are consistent with the values reported in the literature [11,12]. Sheet resistance is expressed in Ohms per square, indicating that the value refers to the sheet measurements and not the bulk. It includes information about carrier mobility and concentration as defined by the formula (1):

$$R_S = \frac{1}{q n_S \mu}, \quad (1)$$

where q – carrier charge and n_S – sheet carrier density, μ - carrier mobility.

The G/4H-SiC sample exhibited a temporary increase in the sheet resistance post-irradiation of 185% original value. Thermal treatment induced partial sheet resistance recovery (about 20%). G/6H-SiC sample, exposed to a sixfold higher neutron fluence experienced a 125% increase in sheet resistance, however analogous variable temperature annealing does not result in any effective self-healing effects.

Observations provided above align with findings from [16], where no self-healing effect was present after irradiating with neutron fluence of 2.0 × 10^{18} n/cm^2. Results of Raman spectroscopy measurements in *El-Ahmar et al.* suggest that the graphene layer itself was mildly affected by neutron irradiation. However, the electrical characterisation presented there implies a loss of the quasi-free-standing character of graphene and likely damage to the hydrogen intercalation layer. The DFT model explains the observed self-healing effect, suggesting that during thermal treatment lateral diffusion of the remaining hydrogen atoms facilitates the re-decoupling of the graphene layer from the SiC substrate, thereby restoring its quasi-free-standing properties. It is apparent that above a certain dose of neutron radiation, the hydrogen layer is too depleted, and an effective self-healing effect does not occur.

IV. SUMMARY AND FUTURE DIRECTIONS

As presented in this article, new findings in the field of radiation-resistant graphene-based magnetic field sensors align with current knowledge. Exposure of epitaxially grown graphene on silicon carbide intercalated with hydrogen to neutron fluence exceeding 2.0 × 10^{18} n/cm² alters unrecoverably electrical properties of the entire system. It was shown by *El-Ahmar et al.* that high-temperature treatment can fully or partially regenerate these properties, but only up to a certain radiation dose threshold. Probably, there is no strict threshold value, but it lies in 0.2 – 2.0 × 10^{18} n/cm² range. As expected, a higher dose of neutron radiation, presented in this paper, prevents the effective self-healing effect. This supports the idea that changes in electrical parameters are caused by the loss of quasi-free-standing properties of graphene, as a result of neutron radiation affecting the hydrogen intercalation layer.

Future research will focus on deepening the knowledge of the interaction mechanisms between neutron radiation and the G/SiC system. One of the objectives is establishing the critical fluence at which irradiation begins to significantly impact key material properties. Also investigating differences in 2D and 3D structures responses to neutron exposure. A fundamental objective is to identify radiation-induced modifications in individual layers of the system that could influence sensor performance in fusion-relevant environments.

To assess the integrity of graphene post-irradiation, techniques like Raman spectroscopy and high-resolution transmission electron microscopy (HRTEM) could be involved. Electrical characterisation might be used to support structural observations. Further analyses could focus on quantifying irradiation-induced defects, using Raman spectroscopy to evaluate defect density and assess its evolution with increasing neutron fluence. Changes in surface morphology could be investigated utilizing HRTEM analysis. Additionally, DFT simulations might be proposed to model the influence of neutron-induced defects, such as vacancies and adsorbates, on the electronic properties of graphene, particularly regarding self-doping and charge accumulation.

Further studies should also address the structural changes occurring within the SiC substrate under neutron irradiation. Raman spectroscopy could provide insight into bond restructuring within SiC, while electrical characterisation of unprocessed SiC substrates may help determine if conductivity is affected independently of the graphene layer. Identifying the fluence threshold at which SiC undergoes significant structural modifications could be a key focus, given that such changes may impact the stability of the overlying graphene.

Another critical area of investigation is the stability of hydrogen intercalation under neutron influence. Since hydrogen intercalation is essential for maintaining the QFS properties of graphene, understanding its resilience under irradiation is crucial. HRTEM imaging of the graphene/substrate interface could be useful for assessing how the decoupling of graphene from the SiC surface evolves with fluence. Electrical characterisation will provide additional insights into changes in intercalation effectiveness, while DFT simulations might help model hydrogen coverage dynamics and predict the stability of the intercalation layer under neutron bombardment. Establishing the critical fluence at which hydrogen retention is compromised would be an essential outcome.

Lastly, exploring differences in radiation-induced damage between 2D and 3D systems could provide valuable insights, particularly concerning whether the low dimensionality of graphene offers inherent advantages in radiation-rich environments. By comparing defect densities in graphene and bulk SiC as a function of fluence, future research may clarify whether 2D materials demonstrate greater resilience compared to their 3D counterparts. Additionally, investigating the effects of neutron irradiation on hybrid 2D/3D systems could shed light on how radiation-induced changes in the SiC substrate influence the overlying graphene layer, especially in terms of charge redistribution and defect formation.

By integrating structural, spectroscopic, and electrical characterisation techniques with theoretical modelling, forthcoming studies might provide a deeper understanding of how neutron irradiation affects graphene-based sensors at both atomic and macroscopic scales. Such findings could ultimately contribute to optimizing radiation-resistant graphene devices, ensuring their long-term stability and functionality in extreme environments.

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VI. REFERENCES

[1] W. Biel et al., “Development of a concept and basis for the DEMO diagnostic and control system,” Fusion Engineering and Design, vol. 179, Jun. 2022

[2] F. P. Orsitto et al., “Diagnostics and control for the steady state and pulsed tokamak DEMO,” Nuclear Fusion, vol. 56, no. 2, Jan. 2016

[3] K. Kovarik, S. Entler, I. Duran, and T. Eade, “Analysis of Transmutation of Candidate Sensitive Layer Materials of Hall Detectors under DEMO Like Neutron Fluxes,” Fusion Engineering and Design, vol. 155, Mar. 2020

[4] I. Bolshakova et al., “Metal Hall sensors for the new generation fusion reactors of DEMO scale,” Nuclear Fusion, vol. 57, no. 11, 2017

[5] S. Entler et al., “Temperature dependence of the Hall coefficient of sensitive layer materials considered for DEMO Hall sensors,” Fusion Engineering and Design, vol. 153, Sep. 2020

[6] I. Ďuran et al., “Status of steady-state magnetic diagnostic for ITER and outlook for possible materials of Hall sensors for DEMO,” Fusion Engineering and Design, vol. 146, Sep. 2019

[7] J. Jankowski, R. Prokopowicz, K. Pytel, and S. El-Ahmar, “Toward the Development of an InSb-Based Neutron-Resistant Hall Sensor,” IEEE Transactions on Nuclear Science, vol. 66, no. 6, Jun. 2019

[8] S. El-Ahmar et al., “The Comparison of InSb-Based Thin Films and Graphene on SiC for Magnetic Diagnostics under Extreme Conditions,” Sensors, vol. 22, no. 14, Jul. 2022

[9] S. El-Ahmar et al., “Graphene on SiC as a promising platform for magnetic field detection under neutron irradiation,” Applied Surface Science, vol. 590, Jul. 2022

[10] I. A. Bolshakova et al., “Resistance of hall sensors based on graphene to neutron radiation,” in Springer Proceedings in Physics, vol. 244, pp. 199–209, Apr. 2020

[11] T. Ciuk et al., “Thermally activated double-carrier transport in epitaxial graphene on vanadium-compensated 6H-SiC as revealed by Hall effect measurements,” Carbon, vol. 139, Nov. 2018

[12] T. Ciuk et al., “High-Temperature Hall Effect Sensor Based on Epitaxial Graphene on High-Purity Semiinsulating 4H-SiC,” IEEE Transactions on Electron Devices, vol. 66, no. 7, Jul. 2019

[13] C. Riedl, C. Coletti, T. Iwasaki, A. A. Zakharov, and U. Starke, “Quasi-free-standing epitaxial graphene on SiC obtained by hydrogen intercalation,” Physical Review Letters, vol. 103, no. 24, Dec. 2009

[14] W. Reddig, M. Przychodnia, T. Ciuk, and S. El-Ahmar, “High-Temperature Stability of Sensor Platforms Designed to Detect Magnetic Fields in a Harmful Radiation Environment,” IEEE Sensors Letters, vol. 7, no. 8, Aug. 2023

[15] T. Ciuk et al., “High-temperature Thermal Stability of a Graphene Hall Effect Sensor on Defect-engineered 4H-SiC(0001),” IEEE Electron Device Letters, vol. 45, no. 10, Oct. 2024

[16] S. El-Ahmar et al., “Fluence and thermal threshold for an effective self-healing in high-energy-neutron-irradiated Al2O3/QFS-graphene/6H-SiC(0001) system,” Applied Surface Science, vol. 685, Mar. 2025