Investigation of Impurity Content Effects on Oxidation Behavior of Nuclear Graphite Using Synchrotron Microfocus XRD

Authors: Li Rongcheng, Yao Xianglei, Quan Xiaoyang, Zhou Xingtai, Huang Qing

Date: 2025-05-07T00:00:00+00:00

Abstract

With the widespread application of graphite in fourth-generation reactors, the oxidation behavior of graphite has become an important scientific issue. This study utilized synchrotron radiation micro-focused X-ray diffraction (micro-XRD) to analyze the oxidation gradient of graphite, and comparatively analyzed the microstructure of the oxidation layers of IG-110, IG-11, NG-CT-10, and NG-CT-1 with different impurity contents. The results indicate that impurities significantly increase the oxidation rate of graphite and alter its microstructure. When oxidation at 700°C reaches approximately 20% weight loss, the oxidation layer depths of IG-110 and IG-11 graphite are approximately 3.2 mm and 1.9 mm, respectively. Within the oxidation layer, regions with small grain sizes are preferentially oxidized due to their abundant active sites, while some aggregate particles with larger grain sizes oxidize slowly and remain intact. Further scanning electron microscopy observations revealed that IG-11 graphite also produced large-sized oxidation pores at locations beyond the oxidation layer depth, and enrichment of elements such as V, Ti, Fe, and Ca was found in these regions. The enrichment of impurities promotes the oxidation of the surrounding graphite structure.

Full Text

Investigating the Impact of Impurity Content on Nuclear Graphite Oxidation Behavior via Synchrotron Micro-XRD Analysis

Li Rongcheng[1,2], **Yao Xianglei**[3], **Quan Xiaoyang**[1,2], **Huang Qing**[1], ***Zhou Xingtai***[1]

[1]Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201800, China
[2]University of Chinese Academy of Sciences, Beijing 100049, China
[3]Shandong Jianzhu University, Jinan 250101, China

Abstract

With the widespread application of graphite in fourth-generation nuclear reactors, the oxidation behavior of graphite has become a critical scientific concern. This study employs synchrotron micro-focused X-ray diffraction (micro-XRD) to analyze oxidation gradients in graphite, comparing the microstructural characteristics of oxidized layers in IG-110, IG-11, NG-CT-10, and NG-CT-1 graphite with varying impurity contents. Results demonstrate that impurities significantly enhance graphite oxidation rates and alter microstructural evolution. At 700°C and approximately 20% weight loss, the oxidation layer depths in IG-110 and IG-11 graphite reach about 3.2 mm and 1.9 mm, respectively. Within the oxidation layer, regions with small grain sizes are preferentially oxidized due to their abundant active sites, while some larger aggregate particles with bigger grain sizes oxidize slowly and remain intact. Further scanning electron microscopy reveals that IG-11 graphite develops large oxidation pits even beyond the oxidation layer depth, with enrichment of V, Ti, Fe, and Ca elements detected in these regions. This impurity enrichment promotes oxidation of the surrounding graphite structure.

Keywords: Graphite; Impurity; Oxidation; Microstructure; micro-XRD

Introduction

Since its successful application in the first nuclear reactor (Chicago Pile I), graphite has been utilized in nuclear energy for over eight decades. Currently, nuclear graphite remains the neutron moderator and reflector material for fourth-generation nuclear reactors—molten salt reactors and high-temperature gas-cooled reactors [1]. Graphite occupies most of the reactor core volume, forming channels for coolant and control rods. Its performance and structural integrity directly affect reactor operational safety and service life. Recent studies have enhanced control rod sleeve graphite performance through carbon fiber cloth winding reinforcement, supporting reliable graphite application in molten salt reactors [2,3]. With the emergence of fourth-generation reactor concepts, research on nuclear graphite has gained renewed attention. During service, nuclear graphite faces challenges from high-temperature oxidation, which occurs in two scenarios: slow oxidation by impurities in the coolant during normal operation, and severe oxidation of graphite structures under accident conditions [4,5]. High-temperature oxidation alters graphite material properties including microstructure, density, dimensions, mechanical and thermal characteristics, consequently affecting the service safety of graphite components [6-9]. Understanding graphite oxidation behavior is fundamen-

tal for safety analysis of graphite components, and investigating oxidation mechanisms is necessary for reliable prediction of graphite service life.

Graphite oxidation behavior research primarily considers two processes: diffusion of oxidizing gases in graphite and carbon oxidation reactions. Based on the relative magnitude of diffusion and oxidation rates, graphite oxidation behavior can be divided into three regimes: chemical reaction control, internal diffusion control, and boundary layer control [10-12]. At low temperatures (approximately 500°C or lower), graphite oxidation rate is much slower than oxygen diffusion rate, resulting in uniform oxygen concentration throughout the sample—this is the chemical reaction control regime. At very high temperatures (>900°C), oxidation rate far exceeds diffusion rate, and oxygen is consumed completely at the sample surface—this is the boundary layer control regime. Between these two extremes, oxygen exhibits a diffusion gradient within graphite, constituting the internal diffusion regime.

Numerous studies have reported high-temperature oxidation behavior of various nuclear graphite grades [13-18]. Research indicates that graphite oxidation behavior, beyond its temperature dependence, is closely related to graphite microstructure [13-15] and is also influenced by sample size and gas flow rate [17,18]. Based on the service temperatures of molten salt reactors and high-temperature gas-cooled reactors, graphite oxidation behavior primarily occurs in the internal diffusion regime, forming an oxidation layer within graphite. The pore structure within the oxidation layer exhibits gradient distribution. Accurate characterization of this oxidation gradient is crucial for predicting macroscopic property changes. Early studies employed layer-by-layer machining to obtain density distribution with depth in high-temperature oxidized graphite [19]. Later research combined mechanical polishing with microscopic observation to determine oxidation gradients through statistical pore distribution analysis [20]. However, since oxidized graphite material is extremely fragile, damage from mechanical polishing poses significant obstacles to characterization of oxidation pores [6]. Due to the strong penetration capability of X-rays in graphite, synchrotron-based techniques have been gradually applied to probe graphite microstructures [21-23,27]. In recent years, the authors have utilized micro-focused X-ray diffraction (micro-XRD) at the Shanghai Synchrotron Radiation Facility to characterize high-temperature oxidized graphite, successfully measuring oxidation layer depth and lattice structure distribution within the oxidation layer by extracting graphite diffraction peak information [24]. Further adoption of ion sputtering instead of mechanical polishing revealed the complex oxidation pathways formed by high-temperature oxidation in graphite [25]. These methods provide powerful tools for investigating graphite oxidation behavior.

Impurity content significantly affects graphite oxidation rates [13,26], yet few reports exist on the influence of impurity content on oxidized graphite microstructure. This paper employs micro-XRD to conduct comparative studies on oxidation behavior of purified and unpurified graphite, then utilizes ion sputtering sample preparation to directly characterize microstructural changes induced by

oxidation.

1.1 Experimental Materials

This study employed IG-110 and IG-11 grade graphite manufactured by Toyo Tanso Co., Ltd. (Japan), and NG-CT-10 and NG-CT-1 grade graphite produced by Chengdu Carbon Co., Ltd. (China). Both IG-110 and IG-11 graphite use petroleum coke filler with an average particle size of 20 m. IG-11 has an ash content of 614.20 mg/L, while purified IG-110 nuclear-grade graphite has an ash content of 22.50 mg/L. Matrix impurities were quantitatively analyzed using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). Key impurities in IG-11 and IG-110 graphite are shown in Table 1 [24,25]. NG-CT-10 is a further purified product of NG-CT-1 graphite, with filler average particle size of 25 m [28]. All four graphite types were manufactured through isostatic pressing. Clean diamond tools without lubricants were used to machine the graphite into cylinders with diameter and height of 25.4 mm. Prior to oxidation experiments, samples underwent ultrasonic cleaning in acetone (purity >99.5%) for 10 minutes, repeated twice, to remove potential surface contaminants. Finally, samples were dried in an oven at 200°C for two hours, then cooled to room temperature for weight and dimension recording.

1.2 Oxidation Experiments

Oxidation experiments were conducted using a dual-temperature-zone tube furnace, with experimental setup shown in Figure 1 Figure 1: see original paper. Samples were placed on a quartz support frame (ensuring central positioning in the quartz tube) located in the central part of the right temperature zone. During experiments, both temperature zones were set to the same operating temperature. Gas flow entered from the left side, passing through the left zone for preheating. Oxidation temperature was controlled between 700-705°C. A thermocouple inserted from the right side of the furnace monitored temperature continuously with its measuring junction positioned near the sample. Air flow rate during oxidation experiments was 6 L/min. Nitrogen flow rate as protective gas during heating and cooling was also 6 L/min. At the start of each experiment, nitrogen was introduced first. After the furnace reached the preset temperature and maintained it for 30 minutes, the nitrogen valve was closed and the air valve opened to begin oxidation. Upon reaching the preset oxidation time, air was shut off, nitrogen was introduced, and heating was stopped. After cooling to room temperature, samples were removed and weighed three times to calculate weight loss rates. By controlling oxidation time, samples with different weight loss rates were obtained. Sample dimensions were measured using a dial caliper with resolution of 0.01 mm and error of $\pm 0.02 mm. Zero calibration was performed before measurement, with each sample measured three times and the a were cut for micro-XRD testing and scanning electron microscopy (SEM) characterization.

1.3 Micro-XRD Experimental Setup and Parameters

Micro-XRD experiments were performed at the BL17UM-High Performance Membrane Protein Crystallography beamline at the Shanghai Synchrotron Radiation Facility. The experimental design is shown in Figure 1(b). The experiments used X-rays with energy of 19.97 keV (wavelength 0.638588 Å) and spot diameter of approximately 10 m. Samples were fixed on the sample stage using a Compton film, with the stage moving vertically to enable line scanning of the sample by the X-ray spot. The stage movement step size was 20 m, with 2 s exposure time per step. Considering the complex microstructure of graphite, three line scans were performed on each sample, with 0.5 mm spacing between adjacent lines. Diffraction signals from air and Compton film were measured simultaneously for background subtraction during subsequent data processing.

X-rays transmitted through graphite samples produced diffraction rings on the detector, as shown in Figure 2 [Figure 2: see original paper]. The numbers in the figure indicate graphite diffraction rings. Additional spurious background signals were subtracted in subsequent processing. The distance between sample and detector was calibrated using Fit2D software and diffraction patterns from a standard AgB sample. Two-dimensional diffraction patterns were integrated to obtain conventional XRD spectra.

1.4 SEM Sample Preparation

After completing micro-XRD experiments, sample surfaces were processed using ion sputtering. This processing method has been described in detail in previous research [22] and is summarized here: Samples were polished with silicon carbide sandpaper, then treated twice with an ion milling instrument. The first stage used parameters of 5 keV particle energy and 10° incident angle; the second stage adjusted to 3.5 keV and 6° for surface sputtering. Finally, samples underwent rapid oxidation at 700°C for three minutes. This combined treatment achieves surface polishing while avoiding microstructural damage from mechanical polishing. The oxidized sample cross-sections were then observed using scanning electron microscopy to characterize pore distribution features.

2.1 Oxidation Rate and Dimensions

Table 2 presents weight and dimensional information for the four graphite grades before and after oxidation. The target weight loss rate was approximately 20% for all samples, though oxidation durations varied. Under experimental conditions, IG-110 exhibited an oxidation rate of 39.56 $mg/cm^2 \cdot h$, while IG-11 showed 125.88 $mg/cm^2 \cdot h$—an absolute difference of 86.32 $mg/cm^2 \cdot h$ (relative difference of approximately 104.35%). NG-CT-10 had an oxidation rate of 43.87 $mg/cm^2 \cdot h$, while NG-CT-1 showed 46.50 $mg/cm^2 \cdot h$—an absolute difference of 2.63 $mg/cm^2 \cdot h$ (relative difference of approximately 5.67%). IG-11 demonstrated significantly higher oxidation rates compared to IG-110, while the difference between NG-CT-10 and NG-CT-1 was relatively small. At approxi-

mately 20% weight loss, the purified graphite grades (IG-110 and NG-CT-10) exhibited minimal dimensional changes, whereas the unpurified grades (IG-11 and NG-CT-1) showed noticeable size reduction.

Figure 3 [Figure 3: see original paper] shows optical photographs of oxidized samples. The surface morphology of purified IG-110 and NG-CT-10 graphite showed no obvious changes after oxidation. NG-CT-1 graphite developed a few visible pits, while IG-11 exhibited densely distributed pits. These pits are closely related to impurity content and are hypothesized to result from catalytic oxidation in impurity aggregation regions. Reports indicate IG-11 has high impurity content, with calcium and vanadium mass contents reaching 38.9 mg/L and 313.3 mg/L, respectively [24].

2.2 Micro-XRD Results

Figure 4 [Figure 4: see original paper]-a shows XRD patterns of pristine IG-110 and IG-11 graphite. Measurements revealed that IG-110 graphite has a slightly larger (002) peak width than IG-11, and the (002) peak angle is also slightly smaller. Figure 4-b presents XRD patterns of NG-CT-10 and NG-CT-1 graphite, whose (002) peaks overlap completely.

Parameters of the graphite (002) peak were extracted and analyzed from micro-XRD diffraction spectra. Figures 5a [Figure 5: see original paper]-c show the variation of (002) peak intensity, width, and position with oxidation depth in IG-11 and IG-110 graphite oxidation layers. Height loss measured in Table 1 is also indicated in the figures. The (002) peak intensity increases with depth and stabilizes upon reaching the unoxidized matrix. Since incident X-ray intensity was stable during micro-XRD experiments, the diffraction peak intensity shown in Figure 5-a is proportional to graphite density along the X-ray penetration path. Oxidation causes surface mass loss, resulting in decreased (002) peak intensity.

As shown in Figure 5b, the (002) peak full width at half maximum (FWHM) decreases with increasing oxidation weight loss. FWHM reduction generally indicates increased crystal grain coherent size. Figure 6 [Figure 6: see original paper] shows the average coherent length distribution with oxidation depth for IG-110 and IG-110 graphite calculated from (002) peak FWHM. The average coherent length was calculated using the Scherrer equation:

$$L = \frac{K\lambda}{B\cos\theta}$$

where λ is X-ray wavelength, B is FWHM, and is Bragg diffraction angle [29]. The average coherent length of pristine IG-110 was 25.2 nm, slightly lower than IG-11' s 26.9 nm. After oxidation, the average coherent length at IG-110 sample surface reached 33.5 nm, while IG-11' s reached 37 nm. Obviously, oxidation does not cause graphite grains to grow. The FWHM reduction occurs because

small-grain-size graphite structures are preferentially oxidized. These structures contain more defects and thus more active sites, making them susceptible to preferential oxidation. In contrast, large-grain-size graphite structures oxidize slowly and remain intact, leading to increased average measured grain size by XRD.

Figure 5-c shows that the (002) peak position in IG-110 graphite tends to shift to higher angles within the oxidation layer. The XRD-measured (002) peak represents the superposition of diffraction information from numerous grains in graphite. Regions with many active sites in graphite typically have lower graphitization degrees and larger interplanar spacing. During oxidation, these regions are preferentially eroded, causing their gradual disappearance. Meanwhile, regions with high graphitization degree and tight interlayer arrangement are preserved due to fewer active sites, increasing the average graphitization degree of remaining structures and causing the (002) peak to shift to higher angles. In contrast, IG-11 graphite, due to localized severe oxidation catalyzed by impurities, does not exhibit obvious selective structural evolution, so the (002) peak position shows no clear variation pattern within the oxidation layer.

Comparing the (002) peak intensity and width changes in IG-110 and IG-11 graphite reveals oxidation layer thicknesses of 1.9 mm and 3.2 mm for IG-11 and IG-110, respectively. The significantly lower oxidation layer depth in IG-11 graphite arises from multiple controlling mechanisms. Graphite oxidation involves oxygen diffusion, oxidation product transport, and kinetic control of graphite oxidation. Due to catalytic effects of impurities on graphite oxidation, IG-11 graphite contains regions of localized severe oxidation, which greatly affects oxygen diffusion into graphite and consequently influences oxidation layer depth. Since regions with impurities consume substantial oxygen, IG-11 graphite' s oxidation layer depth is expected to be smaller than IG-110' s, consistent with experimental observations.

Figures 7a [Figure 7: see original paper]-c show the distribution of (002) peak intensity, FWHM, and peak position with oxidation depth for NG-CT-10 and NG-CT-1 graphite. These parameters exhibit similar distribution patterns within oxidation layers as IG-110 graphite. Near the surface (within 1 mm depth), NG-CT-1 graphite shows greater (002) peak intensity reduction and slightly smaller FWHM than NG-CT-10. Beyond this depth in the internal oxidation layer, no obvious differences appear between the two graphite grades. Based on the depth distribution of (002) peak signals and sample surface morphology, NG-CT-1' s impurity content is significantly lower than IG-11' s.

From the (002) peak intensity and FWHM changes, the oxidation layer depth for NG-CT-10 and NG-CT-1 graphite is approximately 3.7 mm. Figure 8 [Figure 8: see original paper] shows the average coherent length distribution with oxidation depth for NG-CT-10 and NG-CT-1 graphite. Both grades had identical original average coherent length of 26 nm. After oxidation, the average coherent length at sample surfaces reached 34-35 nm.

2.3 SEM Microstructure Analysis

Samples processed by ion beam sputtering and rapid oxidation were observed in SEM. Multiple images were stitched to obtain morphology pictures with both adequate resolution and large field of view, as shown in Figure 9 [Figure 9: see original paper] (image width: 3.5 mm). Both IG-110 and IG-11 sample surfaces were severely oxidized, having lost their original pore structures. Significant morphological differences existed within the oxidation layers.

Figure 9a shows a gradual contrast change in IG-110 graphite surface imaging due to oxidation weight loss, consistent with the oxidation layer damage gradient shown in Figure 5. No pits from non-uniform oxidation were observed in IG-110' s oxidation layer. Figure 11 [Figure 11: see original paper] shows magnified images of different oxidation depth regions in IG-110 graphite. Near-surface regions (Figures 11-a and 11-b) show severe oxidation where original pore structures are no longer visible. However, some typical filler particles remain exposed, indicating these large crystal-size graphite structures have few exposed active sites and thus oxidize slowly. In contrast, defect-rich structures, especially binder regions rich in pores, expose numerous active sites and are preferentially oxidized. This weakens the bonding force between filler particles and surrounding structures, making them loose and prone to detachment. The high-magnification image in the upper right corner of Figure 11-a shows quinoline insoluble (QI) particles marked by red arrows. QI is difficult-to-graphitize carbon with abundant pores and is thus hypothesized to be easily oxidized. However, studies show QI particles are generally protected from oxidation by dense spherical graphite lamellae encapsulation [20]. Further observation of Figures 11c and 11d reveals decreasing oxidation degree with increasing depth, where original pore structures remain undamaged. High-temperature oxidation creates very complex oxidation pathways within graphite structures.

Figure 10 [Figure 10: see original paper] shows cross-sections of oxidized NG-CT-10 and NG-CT-1 graphite. Both oxidation layers exhibit obvious gradient distribution characteristics, with no holes or cracks from local severe oxidation observed, indicating insignificant non-uniform oxidation behavior induced by impurities in both grades. Notably, their oxidation depth evolution patterns show high similarity: in the 0-1.2 mm surface region, original pore structures completely disappear, forming dense oxidation paths, though some filler particle outlines remain clearly visible (marked by red rectangles in Figure 10). In the 1.2-2.0 mm transition layer, oxidation paths extend along the binder phase (marked by red circles in Figure 10). In deep regions beyond 2.5 mm, original pore structures are completely preserved. This consistent oxidation behavior suggests similar impurity levels in both graphite grades.

Figure 9-b shows the cross-section of IG-11 graphite samples. Compared to IG-110 graphite, IG-11 developed oxidation pits reaching tens of micrometers or larger on both surface and interior. Particularly the internal oxidation pits indicate severe oxidation can occur even where oxygen concentration is low,

suggesting high local impurity content and non-uniform impurity distribution in graphite. Figure 12 [Figure 12: see original paper] shows magnified images of different oxidation depth regions in IG-11 graphite (non-pit areas). Like oxidized IG-110, high-temperature oxidation creates very complex pore structures in graphite. Some large filler particles survived severe oxidation loss due to few exposed active sites.

To verify the inference of non-uniform impurity distribution in IG-11, energy dispersive spectroscopy (EDS) analysis was performed on regions marked by red circles in Figure 9-b, as shown in Figure 13. EDS analysis detected trace Fe elements in pits near the oxidation surface, while V, Ca, and Ti elements were identified in deeper pits. These impurities correspond to those reported in IG-11 through chemical detection methods [25]. These impurities significantly accelerated high-temperature oxidation of surrounding graphite, enabling formation of large oxidation pits even in internal regions with low oxygen content.

Conclusion

This study employed synchrotron micro-XRD to analyze oxidation layers in four graphite grades (IG-110, IG-11, NG-CT-10, and NG-CT-1), investigating the influence of different impurity contents on microstructural evolution during high-temperature oxidation. Oxidation caused (002) FWHM reduction. Combined with SEM characterization, this indicates that small-grain-size graphite structures with abundant active sites are preferentially oxidized, while large-grain-size structures oxidize slowly and remain intact. Impurity content significantly affects graphite oxidation behavior—IG-11 with high impurity content showed much higher oxidation rates compared to high-purity IG-110. Oxidation pits reaching 100 m in size were found on IG-11 graphite surfaces and in internal regions with low oxygen content. Enrichment of IG-11 impurities (V, Ti, Fe, Ca) was detected in these pits, demonstrating non-uniform impurity distribution in graphite and significant acceleration of oxidation reactions in surrounding graphite structures.

Acknowledgments

The authors thank the staff at the BL17UM-High Performance Membrane Protein Crystallography beamline of the Shanghai Synchrotron Radiation Facility for their experimental support.

Author Contributions

Li Rongcheng designed the experimental methodology, analyzed experimental data, and wrote the initial manuscript. Yao Xianglei and Quan Xiaoyang organized and managed data. Huang Qing reviewed and revised the manuscript and provided research funding. Zhou Xingtai modified the manuscript, finalized the version, and provided research funding.

References

1. Allen T R, Sridharan K, Tan L, et al. Materials challenges for generation IV nuclear energy systems[J]. Nuclear Technology, 2008, 162(3): 342–357. DOI: 10.13182/nt08-a3961.

2. Marsden B J. Managing nuclear graphite ageing[J]. Nuclear Energy, 2002, 41(2): 145–154. DOI: 10.1680/nuen.41.2.145.39019.

3. Huang B, Jiang H, He Z T, et al. Study on the reinforcement process and performance of carbon fiber fabric for graphite control rod guide tube of molten salt reactor[J]. Nuclear Techniques, 2021, 44(04): 81-88. DOI: 10.11889/j.0253-3219.2021.hjs.44.040602.

4. Paul R M, Contescu C I, Gallego N C, et al. On the thermal oxidation of nuclear graphite relevant to high-temperature gas cooled reactors[J]. Journal of Nuclear Materials, 2023, 573: 154103. DOI: 10.1016/j.jnucmat.2022.154103.

5. Luo X W, Yu S Y. Oxidation of graphite material used in reactor[J]. Atomic Energy Science and Technology, 2007, 41(6): 694–698. DOI: 10.3969/j.issn.1000-6931.2007.06.013.

6. Olasov L R, Zeng F W, Spicer J B, et al. Modeling the effects of oxidation-induced porosity on the elastic moduli of nuclear graphites[J]. Carbon, 2019, 141: 304–315. DOI: 10.1016/j.carbon.2018.09.051.

7. Eto M, Oku T, Konishi T. High temperature Young' s modulus of a fine-grained nuclear graphite oxidized or prestressed to various levels[J]. Carbon, 1991, 29(1): 11–21. DOI: 10.1016/0008-6223(91)90089-2.

8. Yang X, Wang X, Tsang D K L. The effect of thermal oxidation on the coefficient of thermal expansion of nuclear graphite[J]. Journal of Materials Science, 2020, 55(18): 7805–7815. DOI: 10.1007/s10853-020-04577-8.

9. Sato S, Hirakawa K, Kurumada A, et al. Degradation of fracture mechanics properties of reactor graphite due to burn-off[J]. Nuclear Engineering and Design, 1990, 118(2): 227–241. DOI: 10.1016/0029-5493(90)90060-B.

10. Kane J J, Contescu C I, Smith R E, et al. Understanding the reaction of nuclear graphite with molecular oxygen: Kinetics, transport, structural evolution[J]. Journal of Nuclear Materials, 2017, 343–367. DOI: 10.1016/j.jnucmat.2017.06.001.

11. El-Genk M S, Tournier J M P. Comparison of oxidation model predictions with gasification data of IG-110, IG-430 and NBG-25 nuclear graphite[J]. Journal of Nuclear Materials, 2012, 420(1–3): 141–158. DOI: 10.1016/j.jnucmat.2011.09.027.

12. Kim E S, No H C. Experimental study on the oxidation of nuclear graphite and development of an oxidation model[J]. Journal of Nuclear Materials,

2006, 349(1–2): 182–194. DOI: 10.1016/j.jnucmat.2005.10.015.

13. Smith R E, Kane J J, Windes W E. Determining the acute oxidation behavior of several nuclear graphite grades[J]. Journal of Nuclear Materials, 2021, 545: 152648. DOI: 10.1016/j.jnucmat.2020.152648.

14. Lo I H, Yeh T K, Patterson E A, et al. Comparison of oxidation behaviour of nuclear graphite grades at very high temperatures[J]. Journal of Nuclear Materials, 2020, 532: 152054. DOI: 10.1016/j.jnucmat.2020.152054.

15. Contescu C I, Guldan T, Wang P, et al. The effect of microstructure on air oxidation resistance of nuclear graphite[J]. Carbon, 2012, 50(9): 3354–3366. DOI: 10.1016/j.carbon.2012.01.040.

16. Lu W, Li M Y, Li X W, et al. Experimental study on the oxidation behavior and microstructural evolution of NG-CT-10 and NG-CT-20 nuclear graphite[J]. Nuclear Science and Techniques, 2019, 30(11): 165. DOI: 10.1007/s41365-019-0693-0.

17. Paul R M. Application of a three-dimensional random pore model for thermal oxidation of synthetic graphite[J]. Journal of Nuclear Materials, 2021, 543: 152589. DOI: 10.1016/j.jnucmat.2020.152589.

18. Chi S H, Kim G C. Effects of air flow rate on the oxidation of NBG-18 and NBG-25 nuclear graphite[J]. Journal of Nuclear Materials, 2017, 491: 37–42. DOI: 10.1016/j.jnucmat.2017.04.032.

19. Contescu C I, Azad S, Miller D, et al. Practical aspects for characterizing air oxidation of graphite[J]. Journal of Nuclear Materials, 2008, 381(1–2): 15–24. DOI: 10.1016/j.jnucmat.2008.07.020.

20. Wang P, Contescu C I, Yu S Y, et al. Pore structure development in oxidized IG-110 nuclear graphite[J]. Journal of Nuclear Materials, 2012, 430(1–3): 229–238. DOI: 10.1016/j.jnucmat.2012.07.015.

21. Huang Q, Li R C, Li C, et al. Depth profile analysis of oxidized nuclear graphite microstructures using micro-focused synchrotron X-ray diffraction[J]. Journal of Materials Science, 2022, 57(11): 6320–6334. DOI: 10.1007/s10853-022-07044-8.

22. Li R C, Huang Q, Yao X L, et al. Pore structure development of oxidized nuclear graphite[J]. Journal of Nuclear Materials, 2024, 601: 155342. DOI: 10.1016/j.jnucmat.2024.155342.

23. Qi W, Xia H H, Zhang C, et al. Effect of temperature on the microporous structure of nuclear graphite studied by SAXS[J]. Nuclear Techniques, 2017, 40(10): 100103. DOI: 10.11889/j.0253-3219.2017.hjs.40.100103.

24. Lee J J, Ghosh T K, Loyalka S K. Comparison of NBG-18, NBG-17, IG-110 and IG-11 oxidation kinetics in air[J]. Journal of Nuclear Materials, 2018, 500: 64–71. DOI: 10.1016/j.jnucmat.2017.11.053.

25. Yan D X, Cao X L, Shen K. Determination of impurity distribution in IG-11/110 nuclear graphite using TOF-SIMS[J]. Journal of Nuclear Materials, 2024, 588: 154814. DOI: 10.1016/j.jnucmat.2023.154814.

26. Wang J M, Feng S L, Yang Y G, et al. Microstructure evolution of IG-110 nuclear graphite with salt infiltration revealed by in situ tensile synchrotron-XRD[J]. Nuclear Techniques, 2019, 42(4): 040106. DOI: 10.11889/j.0253-3219.2019.hjs.42.040106.

27. Liu D, Zillhardt T, Earp P, et al. In situ measurement of elastic and total strains during ambient and high temperature deformation of a polygranular graphite[J]. Carbon, 2020, 163: 308–323. DOI: 10.1016/j.carbon.2020.03.020.

28. Jing S P, Zhang C, Pu J, et al. 3D microstructures of nuclear graphite: IG-110, NBG-18 and NG-CT-10[J]. Nuclear Science and Techniques, 2016, 27(3): 66. DOI: 10.1007/s41365-016-0071-0.

29. Zheng G Q, Xu P, Sridharan K, et al. Characterization of structural defects in nuclear graphite IG-110 and NBG-18[J]. Journal of Nuclear Materials, 2014, 446(1–3): 193–199. DOI: 10.1016/j.jnucmat.2013.12.013.

Note: Figure translations are in progress. See original paper for figures.