High-temperature oxidation of graphite

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Graphite is used in extreme environments, such as high-temperature gas-cooled reactors and anodes for high-temperature fuel cells, because of its outstanding irradiation performance and oxidation resistance. Its oxidation resistance at high temperature depends on the porosity, ash content, oxidizer concentration, burn-off degree and degree of graphitization. The rate of oxidation was determined for different graphite grades (GLM50, GLM and SPSS) at the isothermal temperature range from 773 to 1273 K in a controlled dry oxygen environment. The degree of graphitization was quantitatively measured using selected area electron diffraction patterns and Raman spectra and was shown to affect the oxidation resistance of the graphite grade. The lowest degree of graphitization (0-67) and a high average pore radius (15.4 nm) in GLM decrease the rate of oxidation due to limited active sites for oxidation. The pore radius and the high graphitic structure reduce the average activation energy of GLM50 (41.8 kJ/mol) compared to those of GLM (48.5 kJ/mol) and SPSS (58.5 kJ/mol) in the boundary-layer-controlled regime in the temperature range 1073–1273 K.

Notation

A	pre-exponential factor
A_s	unit surface area
E_a	activation energy of oxidation
I_D	D-band Raman intensity
I_G	G-band Raman intensity
k	isothermal rate constant at temperature T
R	universal gas constant
$\Delta m/A_s$	normalized weight loss

1. Introduction

Graphite is an ideal solid moderating material with good irradiation performance in high-temperature gas-cooled reactors (HTGRs) and is also used as a high-temperature electrode (synthetic electrode grade graphite) for fuel cells and batteries.^{1–7} It has ideal properties for electrode and nuclear applications, such as being chemically inert, highly conductive, resistant to corrosion and machinable, and has good mechanical properties at elevated temperatures. The first-generation solid oxide fuel cell (SOFC) includes graphite as one of the electrodes.^{8–10} The imminent use of yttria-stabilized-zirconia-based solid electrolytes, due to their oxide conduction, becomes an obvious choice, with following efforts to reduce the working temperature of SOFCs.^{11–16} In first-generation SOFCs, graphite corroded electrochemically apparently due to water vapor evolution at the interface of metal oxide.^{10,16,17} Moreover, in HTGRs, graphite is used as structural material, reflector material and fuel element matrix material.¹⁸ The impurity in the helium coolant may lead to graphite outgassing in the heat-exchanger tube.¹⁹ Ingress of air or water causes graphite oxidation, which leads to degradation of its structural and functional properties.²⁰ The oxidation behavior of graphite can be correlated to mechanical and thermal properties. Thus, an analysis of the oxidation of graphite is an important aspect in

order to ensure the safety of chemical reactors. Protective inert ceramic coatings, such as boron nitride (BN)-, silicon carbide (SiC)-, aluminum oxide (Al₂O₃)- and zirconium diboride (ZrB₂)-based composites, have been used to improve the oxidation resistance and structural properties of graphite from the oxidative environment.^{21–26} Qiu *et al.*²⁷ studied the oxidation kinetics of most graphite components. Among graphite components, petroleum graphite had the lowest oxidation rate, followed by natural graphite and resin carbon. Moreover, the graphite oxidation in fuel handling tubes and hot gas duct rupture accidents were examined using the Oxide-RE code,²⁸ and the maximum graphite burn-off of fuel elements and burn-off flux were found to be ~32.9 kg and 328.8 mol/(m² h), respectively. Also, the oxidation of graphite is controlled by different regimes at different temperatures. The chemical regime, the boundary-layer-controlled regime and the in-pore-diffusion-controlled regime^{29–32} are the three different graphite oxidation regimes. At temperatures <873 K,³³ the reaction between graphite and the oxidizing gas occurs very slowly. The concentration of the oxidizing gas and the resulting oxidation are uniform through the whole depth of the penetration in graphite. Initially, the oxidation rate is controlled by only chemical reactivity. At elevated temperatures (>873 K), the chemical reactivity is high and the oxidizing gas reacts with the surface of graphite.³³ Simultaneously, the concentration of the oxidizing gas varies sharply at the graphite surface. Thus, the oxidizing attack is severe at the outside surface, which changes the geometry without any damage in the interior of graphite. At temperatures in the range 773–1173 K,³³ the in-pore-diffusion-controlled regime dominates, which falls in between the chemical regime and the boundary-layer-controlled regime. The oxidation rate in the in-pore-diffusion-controlled regime is controlled by a combination of in-pore diffusion and chemical reaction. Also, the concentration

of the reactant and burn-off varies exponentially with depth in the in-pore-diffusion-controlled regime, which is known as an oxidation burn-off profile. Also, setting the range of different regimes was affected by the impurity level, microstructure, amount of porosity, size of pores and degree of graphitization.^{34–36} In the current work, oxidation of graphite grades (GLM50, GLM and SPSS) with different porosity levels and degrees of graphitization is studied. The oxidation rate of these graphite grades is analyzed. The effect of porosity, average pore size, degree of graphitization and activation energy on the oxidation rate of various types of graphite is quantified. Thus, the current work highlights the synergistic role of a higher degree of graphitization and lower activation energy in supporting an expedited oxidation of graphite.

2. Experimental procedure

Three different grades of graphite – GLM, GLM50 (both purchased from Graphite India Pvt. Ltd) and SPSS (extracted from die material of Fuji-SPS, Japan) – with dimensions of $100 \times 100 \times 3 \text{ mm}^3$ were used. These samples represent a range of densities and degrees of graphitization. The density of these

graphite grades was measured using the geometrical method, the water immersion method and image analysis. The nature of the graphitic phase and, thus, the purity of the graphite grade were analyzed from the Raman spectra collected from micro-Raman spectroscopy (Princeton Instruments, STR Raman, TE-PMT detector). Neodymium (Nd)-doped yttrium aluminum garnet green laser ($\lambda = 532 \text{ nm}$) with an accumulation time of 10 s in the backscattered mode was utilized to get the Raman spectra. Also, the microstructure of the graphite surface was analyzed using scanning electron microscopy (SEM; Zeiss Evo 50 and FEI Quanta 200). The porosity in the surface of graphite was calculated through image analysis (using the ImageJ software) of the SEM micrograph. Brunauer–Emmet–Teller (BET) surface area was measured with a Quantachrome Autosorb automated gas sorption system using nitrogen gas (N_2). Simultaneous thermal analysis (STA) – that is, thermogravimetric analysis and differential scanning calorimetry – is performed on graphite in order to study the oxidation mechanisms. Thermal analysis was performed using an STA-8000 (PerkinElmer, USA) where the graphite sample with an initial mass of 5–15 mg was placed in an alumina crucible. Isothermal studies were performed on samples

Figure 1. Normalized weight loss of graphite grades (a) GLM50, (b) GLM and (c) SPSS; (d) comparative normalized weight loss during isothermal holding for 60 min in the dry oxygen atmosphere at 773–1273 K. Insets in (a)–(c) show the comparative response at higher temperatures and lower times

from room temperature to 1273 K in the oxygen atmosphere (flow rate 20 l/min) at a pressure of ~1 bar for 60 min holding. At first, graphite samples were heated to final temperature (i.e. 773, 873, 973, 1073, 1173 and 1273 K) at a heating rate of 20 K/min in a nitrogen atmosphere. Then, dry oxygen was purged at final temperatures in order to attain isothermal response of graphite samples. The accuracy for STA was 0.2%, and the furnace had thermal stability of ± 0.5 K.

3. Results and discussion

The temperature dependence of graphite oxidation is revealed into three different temperature regimes with different rate-limiting steps:³⁶ the chemical regime (<873 K), in-pore diffusion regime (873–1073 K) and boundary-layer-controlled regime (>1073 K). The isothermal oxidation behavior of graphite grades GLM50, GLM and SPSS are shown in Figures 1(a)–1(c). The normalized weight loss per unit surface area ($\Delta m/A_s$) of graphite at isothermal holding for 60 min up to 873 K is found to be less (<14.4%) (Figure 1(d)), and it increases with increasing temperature. The GLM grade shows the lowest weight loss (39.3%) compared to GLM50 and SPSS (92.3 and 74.2%, respectively) at 973 K. At a higher temperature, ≥ 1073 K, complete oxidation occurred for all the graphite grades.

The weight loss of GLM50 is higher than that of GLM and SPSS, which is attributed to its high degree of graphitization. The crystallinity of graphite is qualitatively revealed in the bright-

field transmission electron microscopy (TEM) images and corresponding selected area electron diffraction (SAED) patterns of graphite shown in Figures 2(a)–2(f). The diffused ring pattern in the SAED patterns (see the intensity profiles of the diffused ring in Figures 2(b), 2(d) and 2(f)) confirms a partial amorphous content of the graphite samples (GLM50, GLM and SPSS).

The degree of graphitization is quantitatively calculated using the relative intensities of the D-band and G-band of the Raman spectra (Figure 3(a)). The D-, G-, D'- and 2D-bands of Raman spectra are the signature peaks for the carbonaceous material. G-band is the characteristic peak for graphite and the D-band intensity (I_D) corresponds to the disordered carbon (defect peak). The relative intensity of G-band, I_G , in the Raman spectra dictates the degree of graphitization using the relation $I_G/(I_G + I_D)$. The deconvoluted D- and G-band intensities are measured using Figure 3(b), and the degree of graphitization (Table 1) is obtained as 0.81, 0.67 and 0.72 for GLM50, GLM and SPSS, respectively. Highly graphitized GLM50 with a lesser number of edge atoms possesses high active sites available in graphite due to the formation of covalent sp^2 bonds between the carbons.³⁷ Thereby, high graphitization undergoes enhanced oxidation and shows an increased oxidation rate relative to that of graphite with a low graphitization degree.³⁸

The least graphitized GLM is highly stable due to its strong covalent bonding, which causes a difference in the surface

Figure 2. Bright-field TEM images and SAED patterns of (a, b) GLM50, (c, d) GLM and (e, f) SPSS. The intensity of the line profile across the SAED patterns confirms the high amorphous content of graphite samples

Figure 3. (a) The Raman spectra and (b) deconvoluted Raman spectra of graphite grades GLM, GLM50 and SPSS

Table 1. D-, G- and D'-bands from the deconvoluted Raman spectra of graphite

Graphite grade	Position of the band: cm ⁻¹			I_D/I_G	Degree of graphitization $I_G/(I_D + I_G)$
	D-band	G-band	D'-band		
GLM50	1350.1	1582.6	1620.4	0.24	0.81
GLM	1353.6	1582.6	1618.2	0.50	0.67
SPSS	1352.1	1582.0	1617.6	0.38	0.72

energies compared to those of GLM50 and SPSS. This results in a lower gasification rate due to oxidation in GLM from the basal planes rather than the active surface (edge and prismatic surfaces³⁹). However, SPSS and GLM50 show high oxidation rates due to high graphitization, with the high active sites leading to an increase in gasification or burn-off. Moreover, the degree of graphitization and different regimes during oxidation of graphite also depend on the porosity, pore volume and active surface area for oxidation. The characteristics of the pores in graphite are measured using the BET technique and are tabulated in Table 2. The open pores will react with oxygen that comes into contact with the active surface of graphite. The total active surface area decreases with an increase in the pore size.⁴⁰ Therefore, GLM50, with an average pore radius of 15.4 nm, possesses less active surface area, which results in a decreased graphite oxidation rate compared to those of GLM and SPSS (Table 2). It may be noted that the pore size is obtained for the initial samples. Consequently, during the oxidation, the pores may combine into larger pores depending on their oxidation kinetics, changing the active surface area at the surfaces of graphite (edge, prismatic and imperfect

site). In summary, the effective surface areas of GLM and SPSS (compared to that of GLM50) may decrease due to their slower kinetics of oxidation.

The oxidation rates for the graphite grades GLM50, GLM and SPSS at the isothermal temperatures of 1073, 1173 and 1273 K under a dry oxygen environment are shown in Figure 4(a). Although a denser graphite with a smaller pore size of 2.8 and 3.7 nm in GLM50 and SPSS, respectively, and a low surface area is expected to have a sluggish oxidation rate,⁴⁰ the oxidation rate obtained for GLM50 is higher compared to those of GLM and SPSS, which is attributed to the high degree of graphitization with sp²-bonded carbon, low average pore size and corresponding high active sites for oxidation. Although the starting pore volumes of GLM and SPSS are higher than that of GLM50 (Table 2), the oxidation reaction rate depends on the convective flow of oxygen that accesses the active surface area of graphite. Also, the oxidation is limited by the kinetics of the reactions at the subsequent time of the oxidation test and the availability of oxygen at the higher burn-off condition. The activation energy for

Table 2. Average pore radius, pore surface area and pore volume using the BET technique

Graphite	Average density: g/cm ³	Average pore radius: nm	Pore surface area: m ² /g	Pore volume: cm ³ /g
GLM50	1.80	2.8	1.0×10^{-3}	1.073×10^{-5} (pore radius <178.3 nm)
GLM	1.74	15.4	0.602	4.630×10^{-3} (pore radius <159.4 nm)
SPSS	1.81	3.7	4.816	8.805×10^{-3} (pore radius <225.1 nm)

Figure 4. Comparison of the oxidation rates of GLM50, GLM and SPSS graphite at the isothermal temperatures of 1073, 1173 and 1273 K

oxidation was calculated from the rate of normalized weight change during isothermal holding for 1 h. The activation energy of oxidation E_a can be obtained from the following equation, where k is the isothermal rate constant, T is absolute temperature, R is the universal gas constant and A is the pre-exponent factor

$$1. \quad k = A \exp(-E_a/RT)$$

The average activation energy for the different regimes, which is identified using Figures 1(d) and 4(b), is tabulated in Table 3. The activation energy of GLM50 in the chemical and boundary-layer-controlled regimes is marginally lower than those of GLM and SPSS. The in-pore diffusion regime at the intermediate temperatures (873–1073 K) shows a higher activation energy for GLM50 (103.6 kJ/mol) than for SPSS (94.9 kJ/mol), which may be attributed to the constraint of dry oxygen for convective/diffusive flow through the GLM50 sample due to its smaller average pore size (pore radius of 2.8 nm) compared to those of GLM and SPSS. The lower activation energy for the SPSS grade in the in-pore diffusion regime (Table 3) with a steep slope of

reaction rate (Figure 4(b)) confirms the rapid kinetics of oxidation. Although the initial active surface area for oxidation is known, the oxidation kinetics may change as the accounting of the instantaneous surface area is not done. Additionally, the higher extent of graphitization appears to be the main attribute in enhancing the oxidation rate. Thus, it may be inferred that the oxidation rate of GLM50 graphite becomes the highest (when compared to those of the GLM and SPSS graphite samples) as the pore volume, dynamic active surface area and oxygen pressure increase during in-pore diffusion and boundary-layer-controlled oxidation increase with time.

The overall contribution of porosity and degree of graphitization on oxidation of graphite is schematically depicted in Figure 5. The oxidation rate due to the chemical reaction is accelerated due to the increasing temperature in the in-pore diffusion and boundary-layer-controlled regimes.

The mean oxidation rate at 1273 K is 132–271 times higher than that at 873 K. Based on the oxidation rates of different graphite grades at three different regimes, GLM is suggested for the application in high-temperature environments and as a high-temperature electrode for fuel cells and batteries.

Table 3. Activation energy of different graphite grades (GLM50, GLM and SPSS)

Graphite	Average activation energy: kJ/mol		
	Chemical regime (<873 K)	In-pore diffusion regime (873–1073 K)	Boundary-layer-controlled regime (>1073 K)
GLM50	150.7	103.6	41.8
GLM	152.4	104.6	48.5
SPSS	173.2	94.9	58.5
Pure graphite ⁴¹	158.6	72.0	10–30

4. Conclusions

All graphite grades (GLM50, GLM, and SPSS) showed a highly temperature-sensitive behavior with a significant difference in the rate of oxidation at 773–1273 K. Thus, GLM and SPSS graphite showed similar and superior oxidation resistance due to their low degree of graphitisation when compared to that of GLM50. The effects of graphitization and porosity and corresponding active site for oxidation makes GLM50 prone to severe oxidation in a dry oxygen atmosphere. The average activation energy determined for GLM50 was 150.7 and 41.8 kJ/mol in the chemical regime and boundary-layer-controlled regime,

Figure 5. Schematic representation of GLM50, GLM and SPSS graphite on oxidation

respectively, showing an enhanced oxidation resistance (compared to that of GLM and SPSS) due to its low degree of graphitization and the limited availability of active sites for oxidation.

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