

A theoretical analysis for oxidation of titanium carbide

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Received: 14 February 2008 / Accepted: 4 August 2008 / Published online: 26 August 2008
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Abstract The oxidation kinetics of titanium carbide (TiC) powder has been studied based on the experimental data summarized as well as theoretical analysis in terms of our model. The effects of temperature, especially the oxygen partial pressure on the oxidation kinetics, are discussed not only qualitatively but also quantitatively in the light of our special theoretical approach. The calculated results show that our theoretical analysis is reasonable.

Nomenclature

α	Original TiC material phase
β	Oxide material phase
ΔE	Apparent activation energy of oxidation
ξ	Reacted fraction of oxidation
D_0^{β}	Diffusion coefficient of oxygen
V_m	Coefficient depending on substance and reaction
R_0	Radius of particle with original whole particle
R	Radius of particle with original TiC powder
x	Thickness of the oxide layer in the particle = $R_0 - r$
K	Equilibrium constant
P_{O_2}	Partial pressure of oxygen in gas phase
$P_{O_2}^{eq}$	Oxygen partial pressure in equilibrium with oxide
R	gas constant
t	Time in second
T	Absolute temperature with K
η	Temperature-increasing rate

Introduction

Titanium carbide (TiC) as engineering materials has attracted increased attention because of its extremely high melting point and strength as well as good corrosion resistance [1–3]. In comparison with other carbides, for example, tungsten carbide (WC), TiC has higher hardness (33%), lower density and thermal stability [4], it is the most popular reinforcing material after SiC and Al₂O₃ [5]. Although TiC has such attractive properties, poor oxidation resistance at high temperatures tends to restrict its range of applications.

Many oxidation studies on TiC materials have been reported [6–14], most of which have discussed the oxidation mechanism from a kinetic viewpoint. Among these studies, many techniques including X-ray diffraction, X-ray microanalysis, thermogravimetric analysis, auger spectroscopy, and scanning electron microscopy (SEM) have been employed to study the oxidation phenomenon. Shimada et al. have systematically investigated the oxidation behavior of TiC powder at low temperature range of 623 to 773 K and also studied the effect of oxygen partial pressure by using TG-DTA, XRD, SEM, and EMD methods. The results showed that the oxidation behavior was affected by temperature and oxygen partial pressure. It might be seen from the morphology development of the oxidation product, i.e., the formation of oxycarbide/titanium suboxides and the crystallization of anatase, followed by the generation of cracks in the grains. Lavrenko et al. [10] carried out the oxidation experiment with hot-pressed TiC materials at $P_{O_2} = 13 \text{ Pa} - 100 \text{ KPa}$ and temperature ranging from 973 to 1,473 K, and reported that the oxidation process followed the parabolic law within the first 40–50 min, after that oxidation proceeded parabolically. Voitovich [11] researched the oxidation of TiC ceramics in

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the temperature range of 873 to 1,473 K in air. Oxidation kinetics was multistage, i.e., three-stage parabolic law and linear stage. A two-layer scale was formed above 1,273 K. The outer scale consisted mainly of rutile (TiO_2). The inner scale consisted of various titanium oxides. These studies have revealed that the oxidized product formed the double layers consisting of rutile (TiO_2) (in the outer layer) and oxycarbide or titanium suboxide (in the inner layer).

On the other hand, there have been few works on the theoretical study and the rate changes of oxidation have not been well understood. The oxidation kinetic study in literature has not given a satisfactory explanation yet, especially in quantitative aspects.

The oxidation of TiC powder researched by Shimada and Kozeki [8, 9] and Stewart and Cutler [6] have been reviewed in this paper and based on which the theoretical analysis of the effect of oxygen partial pressure and temperature on oxidation have been systematically discussed and analyzed. The work presented here is intended to fill up the gap of theoretical analysis quantitatively.

Experimental facts

Stewart and Cutler [6] researched the oxidation behavior of TiC powder at the temperature ranging from 873 to 1,273 K using a thermogravimetric apparatus. TiC powder used in the experiment contained 0.28% impurities i.e., iron, zirconium, silicon, and vanadium, and the particle size was 106–150 μm . The powdered sample was treated with hot HCl for 15 min and was then washed with distilled H_2O to remove the impurities in TiC powder. Oxygen partial pressures were controlled by a regulated flow of oxygen and helium through capillary flow meters. The gases were dried through activated aluminum, BaO, and P_2O_5 and helium was treated with magnesium amalgam to remove oxygen. During the oxidation experiment, the sample was weighted and then plunged into the furnace, in which the atmosphere and the reaction temperature had been established. The mass increase due to oxidation was then monitored continuously. Figure 1 showed a series of curves of weight gain ($\Delta m/m_0$) versus time t under the temperature range of 873 to 1,058 K and the oxygen partial pressure of 85 KPa that were converted from Stewart and Cutler's data [6]. Stewart and Cutler also studied the oxidation kinetic and found that the parabolic oxidation proceeded with an activation energy of 192 kJ/mol.

Shimada and Kozeki [8] have systematically investigated the oxidation behavior of TiC powder at the temperature ranging from 623 to 773 K at oxygen pressure of 3.9, 7.9, and 16 kPa using TG-DTA, XRD, SEM, and EMD methods. TiC powder used in the experiments had the purity of 99.5% and the surface area was 9.9 m^2/g

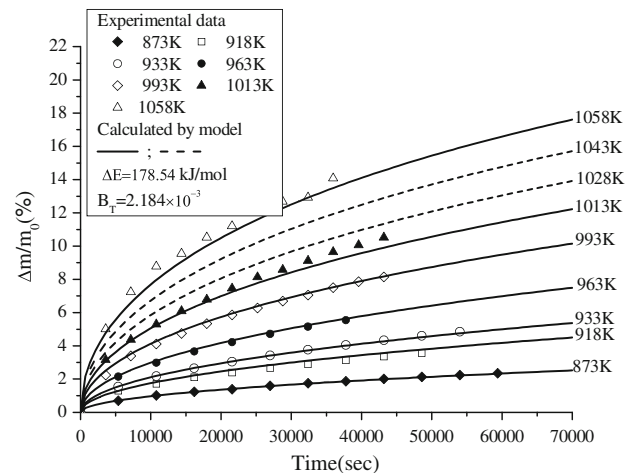


Fig. 1 Isothermal oxidation behavior of TiC powder in oxygen partial pressure of 85 kPa [6]

measured by the BET method. In the isothermal oxidation experiments, Shimada et al. plotted the curves of α^2 versus time (where α represents the reacted fraction). For convenience to our theoretical treatment, these curves had been converted to the plots of α versus time. According to the results of Shimada et al., it showed that: (1) the oxidation behavior was described by the one-dimensional diffusion equation. The oxidation consisted of four steps, i.e., I (fast step), II (slow step), III (fast step), and IV (slow step), at all pressures. Two activation energies were obtained in steps II–IV: 65–150 kJ/mol below 693 K or so and 32–71 kJ/mol above that temperature; (3) these phenomena can be explained from the viewpoint of the morphological development of the oxidation product. The oxidation morphological development at the experimental temperature range was as follows: oxycarbide/titanium suboxides $\text{Ti}_x\text{O}_{1-x}$ ($0 < 1-x < 0.5$) \rightarrow TiO_2 (anatase) \rightarrow TiO_2 (rutile). When the temperature was below 693 K, the rate-determining step was the oxygen diffusion through the layer of the titanium suboxides and when above 693 K, the titanium suboxide and anatase layers tended to expand and cracks formed, therefore, the diffusion of oxygen through the oxycarbide phase should be the controlling step with lower activation energy. Similarly, the oxidation mechanism would also change with time at the same temperature because of the morphological development of the oxidation products. Figures 2 and 3 showed the isothermal parabolic oxidation behavior of TiC powder with oxygen partial pressure of 3.9 and 7.9 KPa, respectively.

Shimada and Kozeki [8, 9] also researched the effect of oxygen pressure on oxidation both under isothermal and non-isothermal conditions. The experimental data showed that the higher oxygen pressure, the larger the oxidation rate. In terms of the isothermal oxidation at 652 K with oxygen partial pressure of 3.9 and 7.9 KPa, the oxidation behavior

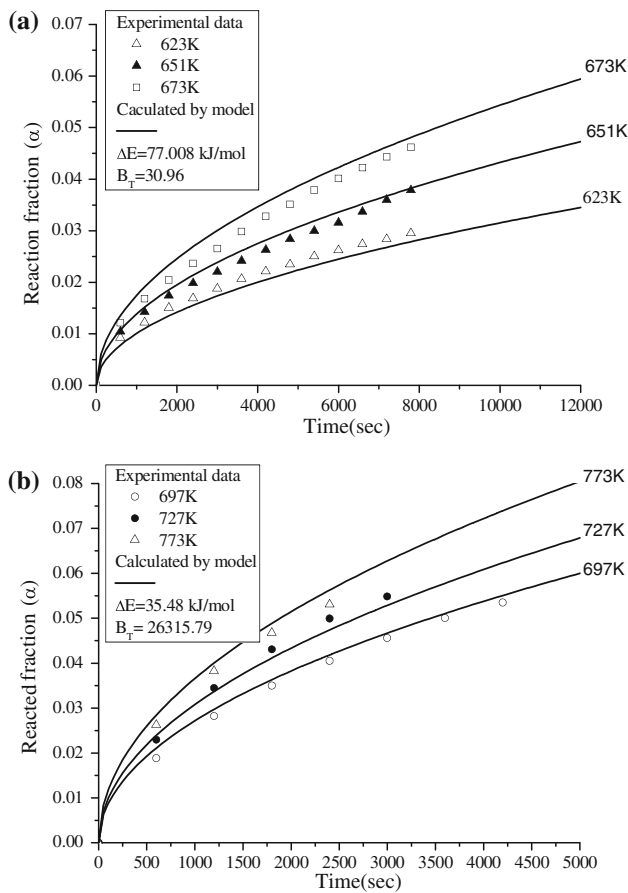


Fig. 2 Isothermal oxidation behavior of TiC powder in oxygen partial pressure of 3.9 kPa: (a) 623–673 K, (b) 697–773 K [8]

followed parabolic rate law for first 8,000 s and after that, the mechanism changed because of the change of morphology. In view of the non-isothermal condition, the mechanism changed above 773 K or so. In our present work, we only treat the oxidation behavior with the same oxidation mechanism in a plot as shown in Figs. 4 and 5, respectively.

It is the goal pursuing in this article to explain all these phenomena not only qualitatively but also quantitatively, and based on which to obtain some idea for future application and development of TiC material.

Theoretical aspects

It is well known that the oxidation of TiC powder consists of a series of sequential steps and its oxidation mechanism is affected by its oxidation products. During the oxidation process, diffusion occupied a considerable proportion. Therefore, in our article we mainly treat the diffusion-controlled process. Further study will be followed to describe the oxidation behavior when the oxidation reaction is controlled by other steps.

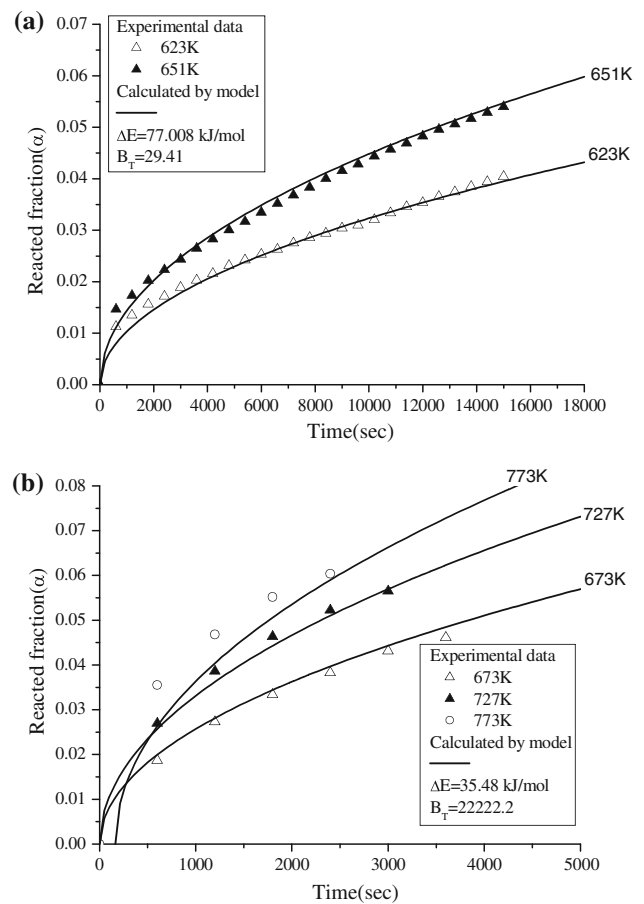


Fig. 3 Isothermal oxidation behavior of TiC powder in oxygen partial pressure of 7.9 kPa: (a) 623–651 K, (b) 673–773 K [8]

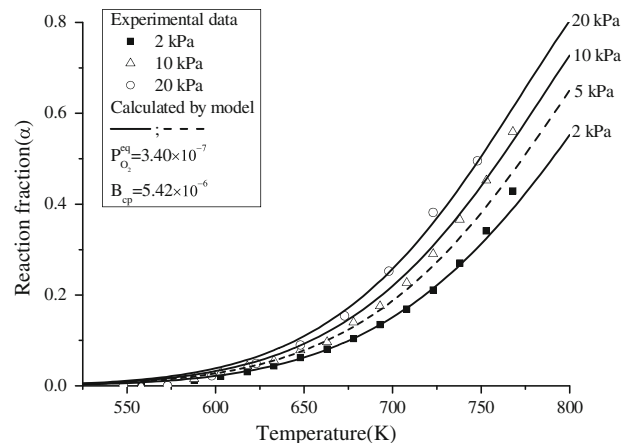


Fig. 4 Non-isothermal oxidation behavior of TiC powder under different oxygen partial pressure [9]

Recently Chou [15] has developed a simpler and physical meaningful explicit analytic formula in order to describe the reacted fraction of oxidation of ceramics with time, which is derived under a simplified but still reasonable assumption, therefore that might not sacrifice too

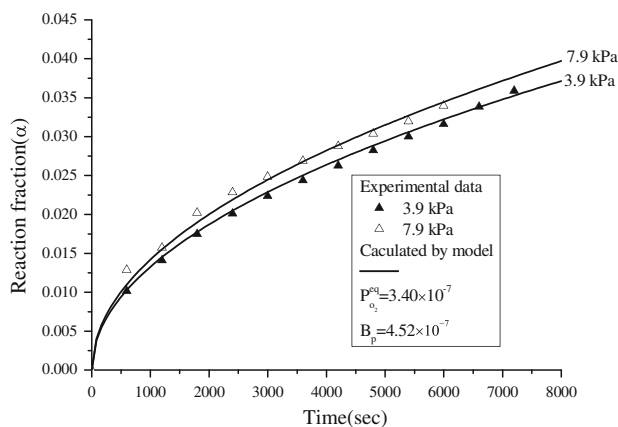


Fig. 5 Isothermal oxidation behavior of TiC powder at 651 K under different oxygen partial pressures [8]

much calculation accuracy. The new model has already been successfully employed to treat the oxidation of Si_3N_4 , AlN, SiC, and SiAlON materials [16–20]. These equations express the reacted fraction ζ of oxidation as a function of temperature T , time t as well as the oxygen partial pressure P_{O_2} as follows:

(1) The effect of temperature T on the reacted fraction ζ at a constant temperature

$$\zeta = 1 - \left(1 - \sqrt{\frac{\exp(-\frac{\Delta E}{RT}) t}{B_T}} \right)^3, \quad (1)$$

where

$$B_T = \frac{1}{\frac{2K_0^{\alpha\beta} D_0^{\alpha\beta}}{v_m} \left(\sqrt{P_{\text{O}_2}} - \sqrt{P_{\text{O}_2}^{\text{eq}}} \right) R_0^2} \quad (2)$$

and ΔE represents the apparent activation energy; B_T is a function of P_{O_2} , $P_{\text{O}_2}^{\text{eq}}$ and R_0 , in which $P_{\text{O}_2}^{\text{eq}}$ is the oxygen partial pressure in equilibrium with oxide and should be related to temperature T . $K_0^{\alpha\beta}$ and $D_0^{\alpha\beta}$ are constants independent of temperature specific to the material; V_m is a coefficient that depends on the properties of substance and reaction; R_0 is the radius of the particle. If the value of $P_{\text{O}_2}^{\text{eq}}$ is very small or the temperature coefficient of $P_{\text{O}_2}^{\text{eq}}$ can be neglected, thus B_T will be constant as the oxygen partial pressure and the particle radius are fixed.

(2) The effect of oxygen partial pressure P_{O_2} on the reacted fraction of oxidation ζ . Define

$$B_P = \frac{1}{\frac{2K_0^{\alpha\beta} D_0^{\alpha\beta}}{v_m} \exp(-\frac{\Delta E}{RT}) R_0^2} \quad (3)$$

where B_P is a function of the particle size, R_0 and temperature, T . The value of B_P will be constant as the

particle size and temperature are fixed. Thus, Eq. 1 will become

$$\zeta = 1 - \left(1 - \sqrt{\frac{(\sqrt{P_{\text{O}_2}} - \sqrt{P_{\text{O}_2}^{\text{eq}}}) t}{B_P}} \right)^3. \quad (4)$$

(3) The effect of oxygen partial pressure P_{O_2} on the reacted fraction of oxidation ζ in variable temperature process. If the system was heated from room temperature T_0 to the experimental temperature T at a certain temperature-increasing rate “ η ” where $\eta = dT/dt$, the relationship between temperatures T with time t should be

$$T = T_0 + \eta t. \quad (5)$$

Define

$$B_{CP} = \frac{1}{\frac{2K_0^{\alpha\beta} D_0^{\alpha\beta}}{v_m} R_0^2 \eta}, \quad (6)$$

where B_{CP} is a function of the particle size, R_0 and the temperature heating rate, η , the value of which will be constant as the particle size and the temperature heating rate are fixed. Thus Eq. 4 will become

$$\zeta = 1 - \left(1 - \sqrt{\frac{(\sqrt{P_{\text{O}_2}} - \sqrt{P_{\text{O}_2}^{\text{eq}}}) \exp(-\frac{\Delta E}{RT}) (T - T_0)}{B_{CP}}} \right)^3. \quad (7)$$

The above equations describe the relation of the reacted fraction ζ with temperature T , oxygen partial pressure P_{O_2} and other variables. In the following section, the application of our equations to the oxidation of TiC powder will be illustrated.

Application of new model to oxidation of TiC powder

Effect of temperature on oxidation behavior

In this section, Eq. 1 will be verified by using the data offered by Stewart and Cutler [6] (as shown in Fig. 1). Since the experimental data are the relationships between the dimensionless mass changes, $\frac{\Delta m}{m_0}$ versus time, the conversions are needed before using Eq. 1. The relation between ζ and $\frac{\Delta m}{m_0}$ is

$$\zeta = \frac{\Delta m / m_0}{\Delta m_{\text{max}} / m_0}, \quad (8)$$

where m_0 represents the original sample mass and Δm_{max} is the theoretical maximum increment after a complete

oxidation. $\frac{\Delta m_{max}}{m_0}$ for oxidation of TiC powder is 33.3 according to the following reaction [6, 8, 9].



The activation energy ΔE calculated by Eq. 1 through the experimental data regression fitting is 178.54 kJ/mol, which is close to the value of 193.2 kJ/mol, offered by Stewart and Cutler [6]. Substituting the parameters into Eq. 1, the isothermal oxidation behavior of TiC can be described as follows:

$$\frac{\Delta m}{m_0} = 33.33 \left(1 - \left(1 - 21.4\sqrt{t} \exp\left(-\frac{10737.5}{T}\right) \right)^3 \right). \tag{10}$$

The theoretical curves of $\frac{\Delta m}{m_0}$ versus t are now plotted in Fig. 1. It can be seen that there is an excellent agreement between the theoretical results and experimental data.

Next, let us use our model to study the data offered by Shimada and Kozeki [8]. They have investigated the oxidation of TiC powder at the temperature range of 623 to 773 K with oxygen partial pressures of 3.9 and 7.9 kPa, respectively and the results were shown in Figs. 2 and 3, respectively. Please note that, the oxidation mechanism changed with temperature and time that can be explained from the change of morphological development of oxidation product as mentioned above. For example, the oxidation behavior with oxygen partial pressure of 3.9 kPa followed parabolic rate law for first 8,000 s at 623–673 K (Fig. 2a) however from 697 to 773 K, the parabolic oxidation law only met for 4,000 s (Fig. 2b). Figure 3 shows the plots of reacted fraction versus time at higher oxygen partial pressure (7.9 kPa), it can be seen that the results are similar to that at lower oxygen partial pressure (3.9 kPa), i.e., at lower temperature range (from 623 to 652, Fig. 3a), the parabolic relation will extend to 8,000 s, while at higher temperature range (from 673 to 773 K) the parabolic relation only extends to 4,000 s. According to our theoretical treatment, there are two parameters in Eq. 1, i.e., B_T and ΔE need to be evaluated through fitting the experimental data and the formula for the oxidation with time should be as follows:

Oxidation behavior with oxygen partial pressure of 3.9 kPa:

(1) temperature range (623–673 K)

$$\xi = 1 - \left(1 - 0.1797 \exp\left(-\frac{4631.25}{T}\right) \sqrt{t} \right)^3 \tag{11}$$

(2) temperature range (697–773 K)

$$\xi = 1 - \left(1 - 0.006164 \exp\left(-\frac{2133.7}{T}\right) \sqrt{t} \right)^3. \tag{12}$$

Oxidation behavior with oxygen partial pressure of 7.9 kPa:

(1) temperature range (623–651 K)

$$\xi = 1 - \left(1 - 0.18 \exp\left(-\frac{4631.25}{T}\right) \sqrt{t} \right)^3 \tag{13}$$

(2) temperature range (673–773 K)

$$\xi = 1 - \left(1 - 0.006519 \exp\left(-\frac{2133.7}{T}\right) \sqrt{t} \right)^3 \tag{14}$$

At present the above theoretical curves of ξ versus t are also shown in Figs. 2 and 3, respectively. It can be seen that the accordance between the experimental data and theoretical prediction should be fine. In addition, the activating energies calculated from the new model were to be 77 kJ/mol for low temperature and 35.48 kJ/mol for high temperature, which fall in the calculation result range offered by Shimada and Kozeki [8], i.e., 65–150 kJ/mol below 693 K or so and 32–71 kJ/mol above that temperature.

Effect of oxygen partial pressure

Figure 4 shows the results of non-isothermal oxidation of TiC powder with a heating rate of 5 K/min under the oxygen partial pressure of 2, 10, and 20 kPa, respectively [9]. The experimental data showed that the oxidation began at about 573 K and the reaction mechanism changed at 783 K or so because of the transformation of oxidation product. Therefore, the new model has been checked only in the range from room temperature up to 783 K. According to Eq. 7, there are two parameters B_{CP} and ΔE that are required to be determined by fitting the experimental data. Based on the present experimental data, these two parameters could be easily extracted by regression method. ΔE and B_{CP} were determined to be 128.25 kJ/mol and 5.42×10^{-6} , respectively. Substituting the above values of ΔE and B_{CP} into Eq. 7, the description of the model of oxidation behavior of TiC can be expressed as a function of oxygen partial pressure and temperature as follows:

$$\xi = 1 - \left(1 - 429.67 \exp\left(\frac{-7712.89}{T}\right) \sqrt{(T - 298)(\sqrt{P_{O_2}} - 0.000583)} \right)^3. \tag{15}$$

The computed results are listed in the same figure for comparison with the corresponding experimental data under non-isothermal conditions. The excellent agreement between the theoretical treatment and the experiment data again confirms the validity of the present theoretical approach to TiC oxidation reactions. In addition, the oxidation behavior of TiC powder with other oxygen partial

pressure, such as 5 kPa, can be predicted by using Eq. 15 as shown in Fig. 4 (dash line).

Shimada and Kozeki [8] also studied the effect of oxygen partial pressure on oxidation of TiC powder under isothermal condition (as shown in Fig. 5). The oxygen partial pressures, achieved by mixing with argon, were 3.9 and 7.9 kPa. In an analogous way, the equations describing the effect of oxygen pressure on oxidation behavior at 652 K is as follows:

$$\xi = 1 - \left(1 - 0.000316 \sqrt{(\sqrt{P_{O_2}} - 0.000583)t} \right)^3. \quad (16)$$

For comparison the theoretical curves are also presented in Fig. 5. It may be seen that the experimental data and theoretical prediction are in good agreement again validating our present theoretical approach.

Discussion

As reported in literatures, most researchers studied the oxidation kinetics of TiC materials by using the following parabolic formula [6–12]:

$$(\Delta w)^2 = kt + C, \quad (17)$$

where Δw was the oxidation mass change, k the reaction rate constant, and C is a numerical constant. The major difference between the equations used in the present work and the parabolic rate law is the expression of the constant. We have revealed the physical meaning of the parameter k of the parabolic rate law that can be expressed as a function of temperature T , diffusion coefficient $D_0^{p\beta}$, particle size R_0 , oxygen pressure P_{O_2} , etc. All these parameters appearing in our model have clear physical meaning. In some particular cases, one can combine part of these parameters to construct an auxiliary function like B_T , B_p and then our model also have a simple expression.

As mentioned above, the oxidation reaction of TiC powder is a kind of complicated heterogeneous reaction. If one deals with it rigorously, the mathematic treatment would be very complicated to solve a group of differential equations and it is hard to obtain a simple solution that will prevent us from theoretical description and discussion. In our present work, we got a simple analytic solution with an explicit function form after making some approximate assumptions. As a result, they should be easy to use and can be used as a tool of theoretical analysis. The examples show that our treatment is feasible. From the view of mathematical treatment, although errors exist in both our new model and the parabolic rate law, which caused by the approximation treatment, our model is much better than the parabolic rate law in extracting the activation energy because it only needs to perform regression just one time.

Therefore, the calculated error from our model should be smaller. The calculated error from the two models can be obtained by the following equation:

$$\Delta = \frac{1}{N} \sum_{i=1}^N \frac{|(\chi_i)_{\text{mea}} - (\chi_i)_{\text{cal}}|}{|(\chi_i)_{\text{mea}}|} \times 100\%, \quad (18)$$

where Δ is the average relative error, $(\chi_i)_{\text{mea}}$ experimental data, $(\chi_i)_{\text{cal}}$ the value calculated from the model, and N is the sum of experiment points. According to Eq. 18, the error for the oxidation of TiC powder offered by Stewart and Cutler [6] calculated from the new model was to be 2.98%. It was much smaller than that calculated from the parabolic rate law, which was 10.5%. This further validates the performance of the new model. Since the error calculated from the new model is much smaller than that from parabolic model, it makes us feel more comfortable to add some predicted lines, i.e., 1,028 and 1,043 K (dash line), in Fig. 1 as well as the non-isothermal curve at oxygen partial pressure of 5 kPa in Fig. 4 that might be interested in application.

The experimental data offered by Stewart et al. and Shimada et al. showed that the effect of temperature and oxygen partial pressure on the oxidation behavior of TiC was evident. When our new explicit formulae expressed by the reacted fraction as a function of temperature and oxygen partial pressure have been applied to it, a good agreement was found between theoretical calculations and the experimental data, which further confirmed that this new model can be used as a tool for treating oxidation problem for this kind of inorganic non-metallic materials.

According to the experimental results reported in literatures, the oxidation mechanism of TiC powder seems not a simple one. It can be divided into two ranges: high and low temperature ranges. In the low temperature range, one can further divide it into two regions: higher and lower temperature regions. Therefore, in our treatment two regions have been considered. The Stewart et al.'s data are in the high temperature range, for this simple range our model is in good accordance with the experimental results. While for the Shimada et al.'s experiments two regions existed apparently that can be proved from the morphological development of the oxidation products either. Therefore, our model must be used separately for two different regions when the diffusion of oxygen is the controlling step.

Conclusion

There are two sets of experimental data used in this analysis: Stewart and Cutler's data [6] and Shimada and Kozeki's data [8, 9], the experimental condition of former one was set in high temperature and high oxygen partial

pressure range (temperature range from 873 to 1,058 K, oxygen partial pressure 85 kPa), while the Shimada’s experimental condition was in low temperature range and low oxygen partial pressure (temperature range from 623 to 773 K and the oxygen partial pressures were 3.9 and 7.9 kPa, respectively).

For Stewart’s data, our theoretical treatment has reached an excellent agreement with experimental data. The formulae of reacted fraction versus time can be expressed as the following form

$$\frac{\Delta m}{m_0} = 33.33 \left(1 - \left(1 - 21.4\sqrt{t} \exp\left(-\frac{10737.5}{T}\right) \right)^3 \right). \tag{10}$$

This formula not only can accurately describe Stewart’s data but also can predict some area data where Stewart’s data are absent.

For the low temperature and low oxygen partial pressure experiments performed by Shimada et al., the situation was a little bit complicated. It seems there existed two regions that were controlled by diffusion: one is in lower temperature region and other is in high temperature region. Since the final oxidation products were different they should be described by different equations, i.e., Eq. 11 ($P_{O_2} = 3.9$ kPa, in temperature range from 623 to 673 K), Eq. 13 ($P_{O_2} = 7.9$ kPa, in temperature range from 623 to 651 K) for low temperature region and Eq. 12 ($P_{O_2} = 3.9$ kPa, in temperature range from 697 to 773 K) and Eq. 14 ($P_{O_2} = 7.9$ kPa, in temperature range from 673 to 773 K) for high temperature region.

Shimada et al. further studied the effect of oxygen partial pressure on the reacted fraction versus time in both non-isothermal and isothermal conditions. Both results have been well described by our formulae

$$\xi = 1 - \left(1 - 429.67 \exp\left(\frac{-7712.89}{T}\right) \sqrt{(T - 298)(\sqrt{P_{O_2}} - 0.000583)} \right)^3, \tag{15}$$

$$\xi = 1 - \left(1 - 0.000316 \sqrt{(\sqrt{P_{O_2}} - 0.000583)t} \right)^3, \tag{16}$$

respectively.

Based on the error analysis, it is clear that our model can give more accurate result than that offered by the parabolic rate model in the treatment of oxidation of TiC.

Acknowledgement The authors would like to express their thanks to the Doctor Foundation of USTB for their kind financial support on Contract No: 11120010.

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