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Investigations of SiC aggregates oxidation: Influence on SiC castables refractories life time at high temperature

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Abstract

This paper reports results concerning the oxidation of silicon carbide of pure SiC powder (98% of SiC) and the oxidation of two silicon carbide castables, used in waste-to-energy plants (WTE), and containing 60% and 85% of SiC, respectively. The investigated temperature range (800–1200 °C) corresponds to typical service conditions in WTE. Thanks to thermogravimetric and thermal expansion tests, kinetics of oxidation of the powder of SiC and of the castables has been investigated. According to these tests, several important points have been underlined. Firstly, the oxidation of SiC aggregates has a high influence on the thermal expansion and on the weight gain of SiC castables. Secondly, the grain size distribution of SiC aggregates within castables plays a dominating role (especially enhanced for the fine particles) in castable expansion behaviour induced by oxidation. This is a key point according to the evolution of thermal expansion of such materials.

Keywords: A. Grain growth; B. Grain size; C. Thermal expansion; D. SiC; E. Refractories

1. Introduction

Silicon carbide (SiC) is generally known for its relatively good behaviour against oxidation under high partial pressure of oxygen since oxidation generates a dense layer of silica which limits the diffusion of the oxygen[1] (passive oxidation). Many previous works on the kinetics of oxidation of SiC powders and bulk SiC materials are reported in the literature[2–4] and deal with strong open porosity materials. In this paper, SiC castables are studied. They contain lower porosity (about 15%) and the oxidation kinetics is different compared with the traditional case.[5,6]

Although there are many results published on the oxidation behaviours of SiC powders and bulk materials, This paper contains a lot of useful information on the application of SiC based refractories because the oxidation process of SiC based refractories which usually has a strong open porosity is completely different with these SiC ceramics with lower porosity.

In waste-to-energy plants (WTE), SiC refractories are essentially used in the combustion chamber. Thanks to their high thermal conductivity, these materials permit the thermal transfer to the boiler tubes which produce vapour, frequently used for energy production. Moreover, they also proceed as thermal protection and anti-corrosion barrier of the metal parts constituting the unit framework.[1,7,8] Among this type of materials, hydraulically bonded castables are interesting because they can be casted into structures of complex shapes. Anyway, several inspections of refractory structures carried out when the WTE units stop, showed that walls, made up of SiC products, could be strongly degraded after several years of use. Even if this aspect is not often mentioned in the literature, the oxidation of SiC is strongly expansive. Therefore a significant expansion of the products is suspected to be the main cause of damage in refractory structures of WTE. This paper is a contribution to quantify this mechanism.

Starting from the study of the oxidation kinetic of the SiC powder constituting the aggregate skeleton of the material, an attempt is made in this paper to quantify the volume expansion due to the oxidation of SiC, within the microstructure of two SiC castables. This work is a part of a more important national French

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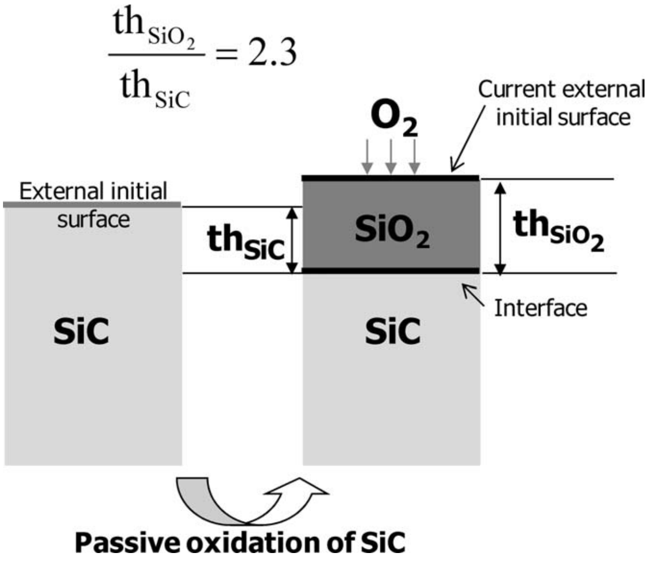

Fig. 1. Mechanism of passive oxidation at the border of a SiC particle.

research program (DRuIDe) which aims to improve the quality and the durability of refractories used in combustion chambers of WTE.[9]

2. Method for the evaluation of the thermal expansion due to SiC oxidation

When the SiC castables are used, at high temperature, in an oxygen rich atmosphere, the passive oxidation of SiC occurs.[3] As illustrated in Fig. 1, the oxidation involves the consumption of SiC and the formation of a silica layer,[10] according to the reaction (1):

$$SiC(s) + \frac{3}{2}O_2(g) \rightarrow SiO_2(s) + CO(g) \quad (1)$$

The measured relative mass variation ($DM_{measured}$) resulting from the weight gain of the formed silica (DM_{SiO_2}) due to the consumption of SiC ($DM_{SiC\text{-}consumed}$) could be written as:

$$DM_{measured} = DM_{SiO_2} - DM_{SiC\text{-}consumed} \quad (2)$$

Considering the molar mass of each product ($M_{SiC} = 40\ g\ mol^{-1}$ and $M_{SiO_2} = 60\ g\ mol^{-1}$) and according to reaction (1), a weight gain of $20\ g\ mol^{-1}$ is expected to be observed by TGA.[3] The relative mass variation of formed silica (DM_{SiO_2}) could be obtained thanks to the following Eq. (3):

$$DM_{SiO_2} = 3 \times DM_{measured} \quad (3)$$

If a fine layer of silica is supposed uniformly distributed on the surface of the aggregates, the thickness (th_{SiO_2}) of formed SiO_2 can be found thanks to the relative mass variation of formed silica (DM_{SiO_2}), to the density of SiO_2 ($\rho SiO_2 = 2.10\ g\ cm^{-3}$) and to the surface of the oxidation reaction (by the specific surface area of the grains of SiC, $S_{SiC} = 0.0548\ m^2\ g^{-1}$) with Eq. (4):

$$th_{SiO_2} = \frac{DM_{SiO_2}}{\rho_{SiO_2} \times S_{SiC}} = 3 \times \frac{DM_{measured}}{\rho_{SiO_2} \times S_{SiC}} \quad (4)$$

As illustrated in Fig. 2, for a spherical grain, the diameter L of the grain after a time τ of oxidation can be determined by Eq. (4):

$$L = (L_0 - 2 \times th_{SiC}) + 2 \times th_{SiO_2} \quad (5)$$

where L_0 represents the initial average diameter of the SiC grain. With the ratio defined in Fig. 1, Eq. (4) can be simplified:

$$L = L_0 + 1.13 \times th_{SiO_2} \quad (6)$$

From Eq. (5) combined with the Eqs. (2) and (4), the relative linear thermal expansion ($DL = \Delta L/L_0$) after a time τ of oxidation can be written as:

$$DL(\tau) = \frac{3.39}{L_0 \times \rho_{SiO_2} \times S_{SiC}} \times DM_{measured}(\tau) \quad (7)$$

As previously reported in the literature,[11,12] the oxidation kinetics for a single-crystal of SiC can generally be described using the linear-parabolic model of Deal and Grove.[13,14] Consequently, the evolution of DL for a spherical grain of the SiC powder should be parabolic and, according to Eq. (6), the maximum possible for DL is 130% which corresponds to the entire transformation of SiC grain into SiO_2 particles.

In order to better understand the consequence of such oxidation kinetics in the case of refractory castables containing SiC aggregate, the work reported in this paper followed three steps:

i. Determination of the parameters of the oxidation kinetics in air from TGA experiments performed in a selected size range of the SiC powder used for the elaboration of castables.
ii. Investigation of the consequence of SiC oxidation in materials with the same composition as the studied castables but

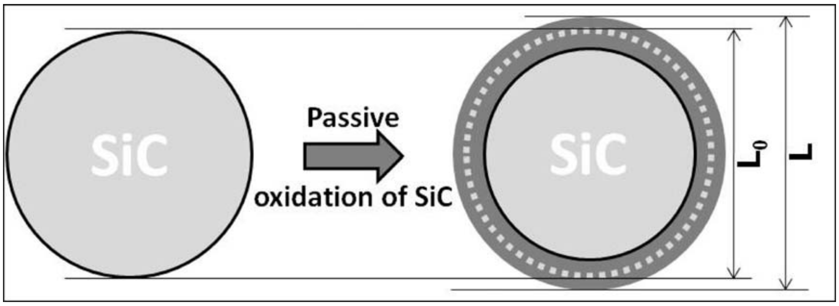

Fig. 2. Evolution of diameter, for an ideal spherical grain, due to passive oxidation of the SiC.

Table 1
Characteristics of SiC castables.

Formulation	C-SF60	C-CV85
SiC (wt%)	60	85
Cement (wt%)	5	7
CaO (wt%)	1.4	2.1
Silica and alumina phase (wt%)	35	8
Water use for the elaboration (wt%)	6.9	6.5
Density ($g\,cm^{-3}$)	2.55	2.51

with SiC grain size inferior to 200 μm, which are expected to be more sensitive to oxidation. These materials are referred as matrices.

iii. Transposition to the case of industrial castables which can be considered as composites with matrices reinforced by coarse SiC grains (superior to 200 μm up to 3 mm).

3. Materials and techniques

3.1. Materials

Two low cement castables (LCC) were studied. They are manufactured by CALDERYS (France), one referred as C-SF60 containing 60% of SiC aggregates and one referred as C-CV85 with 85% of SiC aggregates. Table 1 gives the main characteristics of these castables. They are composed of aluminous cement and of different aggregates mainly SiC, with a wide particle size distribution from 0.0002 to 3 mm.

In order to understand the consequence of SiC oxidation in castables, the study is first carried on simplified materials called matrices. These materials were proceeded from a mix of the finest particles (cement, silica–alumina phases, SiC small particles), with diameter typically lower than 200 μm (Fig. 3). These fine particles were obtained by sieving the powder used for the fabrication of castables. After water addition corresponding to that required for the castable, the obtained matrices are referred here as M-SF60 and M-CV85 according to the castable references.

In addition to castables and matrices, a preliminary study was also carried out on the SiC aggregates themselves. From a commercial SiC powder (Carsilon from TIMCAL, Switzerland) used for castable, grains from 80 to 100 μm were selected by sieving.

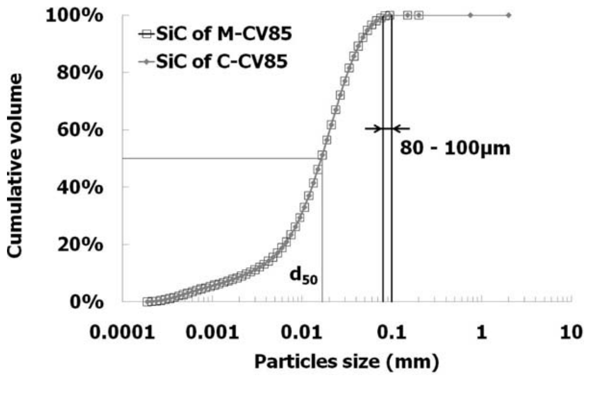

Fig. 3. Particle size distribution of SiC grains within M-CV85 and C-CV85.

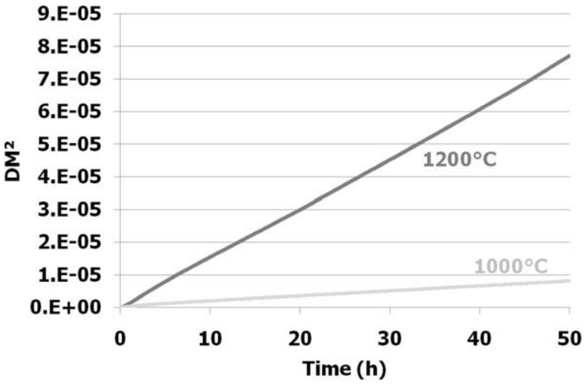

Fig. 4. Oxidation kinetics of SiC at different temperatures.

As shown in Fig. 3, this corresponds to an intermediate particle size of the concretes (between 0.2 μm and 3 mm). The specific surface area of these selected grains, referred as SiC 80-100 powder in this paper, has been estimated to $0.0548\,m^2\,g^{-1}$, using a BET device with krypton (suitable gas for low specific surface area). Such intermediate particle size leads to a significant weight gain in TGA during oxidation experiments, and allows reducing the percentage of free carbon regularly associated to silicon carbide production.[15]

3.2. Techniques

Thermogravimetric (TGA) and thermal expansion (TE) experiments were realized at 1000 and 1200 °C in air flow of 4 ml/min.

TGA were performed with powder samples in case of SiC powder and with bulk samples in case of matrix and castable. Sample of about 1 g were introduced in a PtRh 10% crucible for TGA and a heating rate of $5\,°C\,min^{-1}$ has been used up to the oxidation temperature for 50 h. In this study, 1 g is enough to give a statistically valid sample of the powder in view of the dominance of the small particles in the oxidation: only small grains have a real impact on the oxidation.

For TE, a vertical dilatometer SETARAM, Setsys evolution, was used with a load of 5 g. Samples of 9 mm × 9 mm × 15 mm have been subjected to the same temperature treatments.

4. Results and discussion

4.1. Study of the SiC 80-100 powder

The evolution of the square of the relative weight ($DM^2 = (\Delta m/m_0)^2$) versus oxidation time of SiC 80-100 powder is reported in Fig. 4. The obtained straight lines show that the oxidation kinetics follows a linear-parabolic model and according to Deal and Grove[3,13] this law is characteristic of the passive oxidation of SiC. It means that a silica layer is formed around the SiC particles.

The evolution of DM vs. oxidation time of SiC 80-100 powder and the subsequent evolution of the thickness of SiO_2 (th_{SiO_2}) obtained from Eq. (3), for 50 h are plotted in Fig. 5(a). The results of DM are coherent with the literature[5,16,17]: an increase

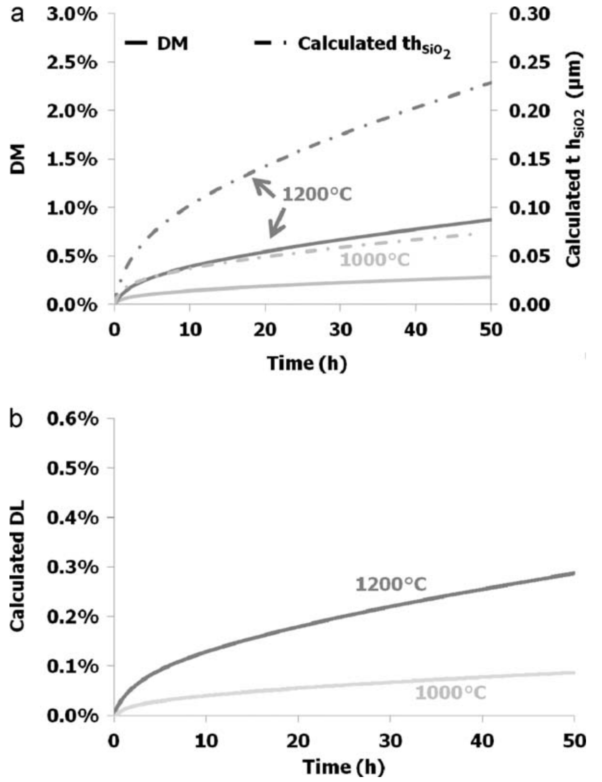

Fig. 5. SiC 80-100 powder oxidation characteristics vs. time: (a) DM and calculated thSiO2; (b) calculated relative thermal expansion (DL).

of temperature enhances the oxidation kinetics of SiC with a parabolic law (0.26% and 0.86% for oxidation at 1000 °C and 1200 °C, respectively). Moreover, the importance of the SiC particle size must be underlined. According to the Fig. 6, the evolutions of DM for nano and micro-sized SiC particles at 1200 °C are more important (multiplied by 10 or 20) than for the SiC 80-100 powder.

The silica growth is found to be parabolic, as referred in literature,[13,14] and the thicknesses of silica after 50 h are 0.07 µm and 0.23 µm at 1000 °C and 1200 °C, respectively.

The evolution of relative thermal expansion (DL) calculated by Eq. (6) for a spherical grain of the SiC powder ($L_0 = 90$ µm, corresponding of the average size of SiC 80-100 powder) is plotted in Fig. 5(b). For an oxidation time of 50 h, DL is 0.08% and 0.28% at 1000 °C and 1200 °C, respectively. These values are low but they are obtained for a time of oxidation with several orders of magnitude lower than those corresponding to an industrial context (several years).

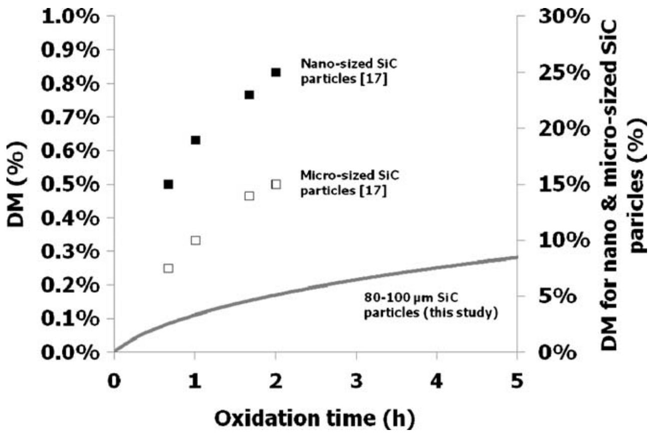

Fig. 6. Comparison of DM of SiC 80-100 powder with nano and macro-sized SiC particles during oxidation at 1200 °C.

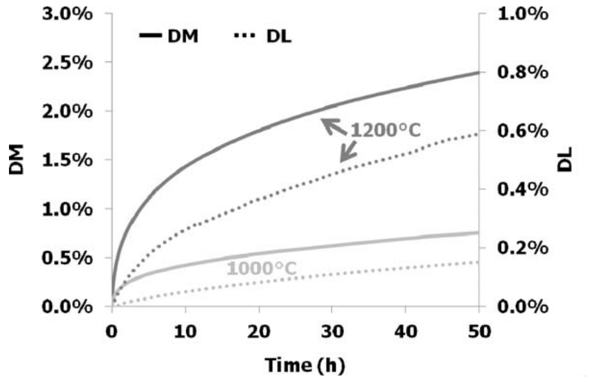

Fig. 7. Relative weight gain and relative thermal expansion of M-CV85 vs. oxidation time.

4.2. Oxidation of SiC within the matrix

The influence of the SiC oxidation within the matrix could here be compared to the results obtained on pure SiC powder. Then, the influence of the SiC particle size distribution could be clarified. Indeed, the matrix is the part of the castable containing fine particles (lower than 200 µm). In fact, the results of M-CV85 are presented here in more details because this matrix contains the highest proportion of SiC particles (64 wt%) and those of M-SF60 are just used as a comparison to observe the influence of the quantity of SiC (41 wt%).

The evolutions of DM and of DL versus oxidation time, shown for M-CV85 in Fig. 7, are consistent with the results obtained for SiC powder: a parabolic progression is observed, which can reach 2.4% for DM and 0.58% for DL after 50 h at 1200 °C.

Similar results obtained with M-SF60 are shown in Table 2. Weight gain values are consistent with the quantity of SiC in matrices since values of DM for M-CV85 and are always superior to the results of M-SF60. Nevertheless, the impact of the oxidation on DL is less clear due to the other mechanisms which could also affect the thermal expansion: for example, sintering could involve shrinkage or CA_2 formation could induce expansion.

Table 2
Comparison of M-SF60 and M-CV85 results after 50 h of oxidation.

	After 50 h at 1000 °C		After 50 h at 1200 °C	
	M-SF60	M-CV85	M-SF60	M-CV85
DM	0.58%	0.75%	1.51%	2.40%
DL	0.10%	0.10%	0.64%	0.58%

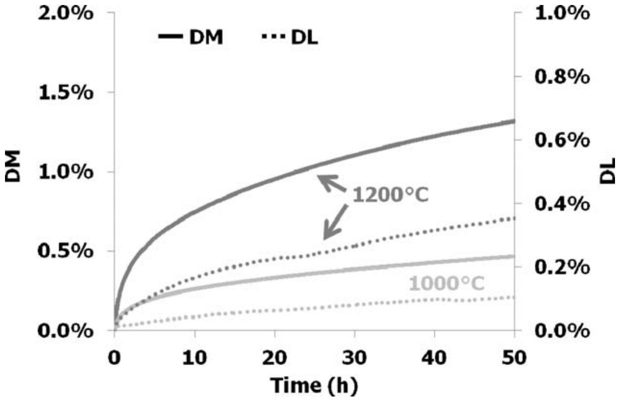

Fig. 8. . Relative weight gain and relative thermal expansion of C-CV85 vs. oxidation time.

4.3. *Oxidation of SiC within the castable*

The evolutions of DM and of DL versus oxidation time, shown in Fig. 8, are similar with the curves previously obtained. A parabolic evolution is observed with lower values than those previously reported for the matrix: only 1.31% for DM and 0.35% for DL after 50 h at 1200 °C.

In a similar way than for the matrices, the weight gain and the thermal expansion reported in Table 3, are amplified by the temperature and by the SiC content. Indeed, the values of DM and of DL for C-SF60 are lower than the C-CV85 ones.

4.4. *Discussion*

4.4.1. *Comparison between castable, matrix and pure SiC powder*

To resume the impact of oxidation on a SiC castable, the results obtained for the SiC 80-100 powder, for the matrix and for the castables, at 1200 °C are compared in Fig. 9. Considering these results and taking into account the amount of SiC in each case (100 wt% for the powder, 85 wt% for the castable and 65 wt% for the matrix), it becomes evident that the SiC quantity is not pertinent here to explain the difference of behaviour regarding the oxidation effects. In a first approach, values of thermal expansion for C-CV85 after 50 h of oxidation at 1200 °C seem to be rather low (less than 0.4%). However, considering the time in use in a WTE (typically several years), the extrapolated expansion for such a long time could induce important troubles. In order to have a better control on this expansion, the influence of the SiC aggregates must be clarified.

Table 3
Comparison of C-SF60 and C-CV85 results after 50 h of oxidation.

	After 50 h at 1000 °C		After 50 h at 1200 °C	
	C-SF60	C-CV85	C-SF60	C-CV85
DM	0.37%	0.47%	0.72%	1.32%
DL	0.03%	0.10%	0.25%	0.35%

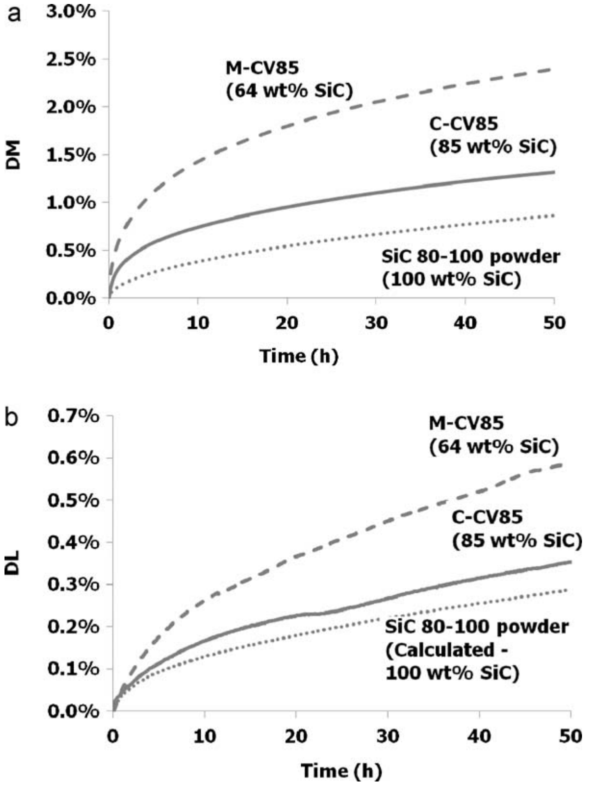

Fig. 9. Comparison of oxidation behaviour at 1200 °C between C-CV85, M-CV85 and SiC 80-100 powder: (a) weight gain; (b) linear expansion.

4.4.2. *Analyses of the influence of SiC aggregates*

Starting from experimental results obtained on SiC 80-100 powder and considering the expression (4), the thickness of silica created ($\mathrm{th}_{\mathrm{SiO}_2}$) could be determined for 1200 °C. The thickness of SiC consumed ($\mathrm{th}_{\mathrm{SiC}}$) could also be calculated and the diameter $\varphi(\tau)$ could be estimated from the initial diameter of the SiC particle φ_0, by the following expression:

$$\phi(\tau) = \phi_0 + 2 \times \mathrm{th}_{\mathrm{SiO}_2} - 2 \times \mathrm{th}_{\mathrm{SiC}} \quad (8)$$

Consequently, the expansion due to SiC oxidation could be determined for different particle diameters (from 0.2 μm to 2 mm) and for longer time (1 month, 1 year, 5 years); the results are shown in Table 4.

The expansion values gathered in this table underline the following points:

- The volume expansion could become very important (in the case of small size or long time).
- The big aggregates of SiC do not play a significant role in the expansion phenomenon due to the SiC oxidation.
- The small aggregates are a key-point for this expansion phenomenon.
- Higher is the temperature, more the oxidation phenomenon is amplified.
- For very long oxidation time, at high temperature, the SiC particles could be entirely consumed and become SiO_2 particles (expansion limited to 130% in such situation).

Table 4
Calculation of the expansion size of SiC aggregates due to oxidation (extrapolated from results obtained after 50 h on SiC 80-100 powder). The shaded portions correspond to the maximum possible expansion size of SiC aggregates.

Aggregates diameter (μm)	Oxidation conditions (dry air)				
	Temperature	50 h	744 h (1 month)	8760 h (1 year)	43,800 h (5 years)
2000 μm	1000 °C	0.004%	0.016%	0.052%	0.115%
	1200 °C	0.012%	0.049%	0.170%	0.381%
90 μm	1000 °C	0.087%	0.345%	1.147%	2.546%
	1200 °C	0.274%	1.099%	3.785%	8.468%
	1000 °C	0.156%	0.622%	2.065%	4.582%
	1200 °C	0.493%	1.978%	6.813%	15.242%
10 μm	1000 °C	0.781%	3.108%	10.326%	22.910%
	1200 °C	2.467%	9.888%	34.064%	76.209%
1 μm	1000 °C	7.809%	31.078%	103.258%	130%
	1200 °C	24.672%	98.881%	130%	130%
0.2 μm	1000 °C	39.047%	130%	130%	130%
	1200 °C	123.359%	130%	130%	130%

In comparison to the expansion values calculated for 50 h at 1200 °C (Table 4), C-CV85 (with an expansion of 0.35% in same conditions) seems to exhibit expansion behaviour close to an average SiC diameter of 50–60 μm.

To conclude, the key point of the oxidation process is not the chemical composition but it is the particle size distribution. The particle size distribution of castable is different of the SiC powder one (only 80–100 μm range is considered). In the castable, the proportion of fine particle are important (sizes of 20% of SiC aggregates are lower than 10 μm) and involves the faster oxidation of castable compared with the SiC powder one. This result is coherent with the literature[17] (Fig. 6).

4.4.3. Influence of SiC amount

To determine the impact of the quantity of SiC for a same particle size distribution, several ratios, after a 50 h oxidation at 1200 °C, are calculated in the Table 5. This could be done because the studied materials are castables. Consequently, they are not sintered. SiC aggregates are just link together via alumina and silica phase. So, the access of oxygen to the all grains is facilitated thanks to the skeleton of SiC aggregates. The diffusion process inside the pore network is not the limiting step of the oxidation.

The ratios of DM and DL for the matrix and for the castables are here very pertinent even if the quantities of SiC in these products are very different (64% for M-CV85, 41% for M-SF60, 85% for C-CV85 and 60% for C-SF60). Therefore, when two products are elaborated with a SiC powder having a similar particle size distribution, the ratio of SiC between these two products leads to a rather good estimation of the ratio of DM and of DL after a given oxidation time. Then, knowing expansion due to oxidation for one of these products allows estimating the DL value of the other in same oxidation conditions.

Table 5
Ratios of DM and of DL for castables and matrix after 50 h at 1200 °C.

	C-CV85/C-SF60	M-CV85/M-SF60
SiC ratio	1.42	1.56
Ratio of DM	1.81	1.58
Ratio of DL	1.41	0.91

5. Conclusion

Several inspections of refractory structures, carried out during the maintenance stop of WTE led by several partners of the DRuIDe program, showed that walls, made up of SiC tiles, could be strongly degraded after several years of use because of a significant expansion of the products (typically 5–7%). According to the present study, same kind of problems could occur with SiC castables. Even if this point is not often mentioned in the literature, the formation of a silica layer on the surface of silicon carbide aggregates, by oxidation at high temperature, can induce a very strong linear expansion, up to 130% at local scale.

Many studies dedicated to the oxidation of SiC have been reported in the literature during the last forty years. This oxidation is generally shown, rightly, as an advantage for SiC. The resulting silica forms a passive layer on the SiC surface, and thus limits, thereafter, the diffusion of the gas species for the continuation of the reaction. Indeed, this is interesting in the case of massive materials, but in the case of refractories, with a strong open porosity (generally from 15 to 20%), the oxidation of SiC must be reconsidered by taking account of the microstructure in the analysis of the phenomenon.

In particular, the present paper investigates the influence of SiC grain size on macroscopic expansion which can result from oxidation (sizes of 20% of SiC aggregates are lower than 10 μm). Indeed, the different results presented here, highlight that, although the layer of silica has a very low thickness (less than one micron) on each SiC grain, it can generate a very significant macroscopic expansion, within the framework of porous refractory products containing small grains of SiC. As an example, the thermal expansion values experimentally measured on samples after 50 h under air at 1200 °C, allows to extrapolate an expansion which can reach 6% in the case of the C-SF60, and

8% in the case of C-CV85, after 5 years in similar conditions of use.

In addition, it is advisable to recall that all the values mentioned in this article result from analyses carried out at laboratory scale under dried air. However, the kinetics of oxidation of silicon carbide are well known to be accelerated (by a factor which can go up to 2) in the presence of water vapour.[2,18–20] The presence of alkalis[21,22] could also accelerate the mechanisms of diffusion in the layer of silica. Since these two last points have not been considered in this present study, the estimations of thermal expansion after 5 years should probably be raised under the real conditions of use in WTE.

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