



# Microstructure and Thermomechanical Behaviour of a SiC-based Refractory

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## Abstract

Refractory materials are commonly used in waste-to-energy plants. They compose part of the wall covering the combustion chamber and protect metallic cooling tubes. Refractories are subjected to severe thermal gradients, to corrosion and to high temperature levels. They also collect part of the thermal energy generated by the waste combustion in order to allow electrical energy production. Such materials must couple a high thermal conductivity and high thermomechanical properties to cope with running solicitations.

Nitride-bonded silicon carbides (NBSC) are porous materials with high thermal and mechanical properties. Microstructures and thermomechanical properties are investigated in order to understand their behaviour in waste-to-energy plants. Specific properties come from their processing route. The resulting microstructure deals with SiC grains bonded with silicon nitride phases. Such bonding phases are characterised by a high complexity degree. Microstructural properties of as-received NBSC refractory are studied. They deal with the crystalline phase identification (by X-ray diffraction) and phase morphology (by SEM).

As the material is used at high temperature in oxidizing atmospheres, the oxidation occurs. Long-term oxidation effects are investigated. Phase and morphological changes are considered. Kinetic aspects of oxidation are taken into account, too. Thermomechanical properties are also studied. In the 20-1200°C temperature range, NBSC refractory exhibits a linear elastic behaviour. The effect of long-term oxidation on the thermomechanical behaviour is also investigated.

**Keywords:** Nitride-bonded silicon carbide, Refractory, Microstructure, Oxidation, Thermomechanical behaviour

## MIKROSTRUKTURA I ZACHOWANIE TERMOMECHANICZNE MATERIAŁU OGNIOTRWAŁEGO OPARTEGO NA SiC

Materiały ogniotrwałe są powszechnie wykorzystywane w instalacjach pozyskujących energię ze śmieci. Składają się one na część ściany pokrywającej komorę spalania i chronią metalowe rury chłodzące. Materiały ogniotrwałe poddawane są ostrym gradientom termicznym, działaniu korozyjnemu i wysokim temperaturom. Gromadzą również część energii cieplnej wytworzonej przy spalaniu śmieci w celu wyprodukowania energii elektrycznej. Materiały takie muszą łączyć w sobie wysoką przewodność cieplną z wysokimi właściwościami termomechanicznymi, aby poradzić sobie z wymaganiami eksploatacyjnymi.

Węglik krzemu wiązane azotkiem (NBSC) to materiały porowate o wysokich właściwościach termicznych i mechanicznych. Mikrostruktury i właściwości termomechaniczne badane są po to, aby poznać ich zachowanie w instalacjach do pozyskiwania energii ze spalania śmieci. Właściwości specyficzne pochodzą ze sposobu otrzymywania. Powstałe mikrostruktury złożone są z ziaren SiC powiązanych za pomocą faz azotku krzemu. Takie fazy wiążące charakteryzują się wysokim stopniem złożoności. Badane są właściwości mikrostrukturalne świeżo otrzymanego materiału ogniotrwałego NBSC. Dotyczy one identyfikacji faz krystalicznych (za pomocą dyfrakcji promieniowania X) i morfologii fazy (za pomocą SEM).

Utlennianie występuje wtedy, gdy materiał stosowany jest przy wysokiej temperaturze w atmosferach utleniających. Badane są skutki utleniania występującego w długim okresie czasu. Rozważane są zmiany fazowe i morfologiczne. Uwzględnia się również aspekty kinetyczne. Badane są właściwości termomechaniczne. W zakresie temperatury 20-1200°C, materiał NBSC wykazuje liniowe zachowanie sprężyste. Badany jest również wpływ długotrwałego utleniania na zachowanie termomechaniczne.

**Słowa kluczowe:** węgiel krzemu wiązany azotkiem, materiał ogniotrwały, mikrostruktura, utlenianie, zachowanie termomechaniczne

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## 1. Introduction

Due to the running conditions and with the goal of producing energy, the materials constituting the tiles of waste-to-energy plants should conceal a high thermal conductivity, high thermomechanical properties but also oxidation resistance. Thus the tiles are made of SiC-based materials, as SiC offers high properties in this context. The material of this study is a nitride-bonded silicon carbide material

(NBSC). Such NBSC-tiles are elaborated following a specific processing route [1, 2]. During this process, SiC aggregates are bonded together with nitride-rich phases. The bonding phase microstructure is very close to the reaction bonded silicon nitride one (RBSN) [3-6].

The microstructure highly depends on nitridation conditions, such as the atmosphere composition and the additives used. The literature is well documented on this aspect

because the conditions affect the nitride formation and also the resulting morphology [6-9].

NBSC materials are separated in to two classes, which depend on SiC grain size. NBSC materials are called refractory in the case of a large size of SiC aggregates (3 mm), whereas they are called composites for a maximal SiC grade close to 500  $\mu\text{m}$  [10].

Such a refractory material has been studied previously due to its use in the siderurgical industry (blast furnace lining) [11], in waste-to-energy plants [8] but also in kiln furniture [12]. NBSC oxidation studies are also present in the case of aluminium cell applications[1].

Oxidation in air of NBSC refractories is very similar to RBSN oxidation [10]. It occurs in two stages [2, 10, 11] but the reaction kinetic differs with the material heterogeneity. Indeed the material structure is complex because NBSC oxidation corresponds to the oxidation of two non-oxide materials (SiC and  $\text{Si}_3\text{N}_4$ ) and each component gives silica as the oxidation product [13, 14]. The presence of high porosity level with pore channels also affects NBSC oxidation [2, 11]. Furthermore, oxidation kinetic of NBSC material highly depends on the presence of steam water in the oxidising atmosphere [2] but also on a sample shape [11, 15].

The microstructure evolution of the bonding phase with oxidation were characterised [11]. Oxidation affects the morphology and progressively transforms the bonding phase (especially the  $\alpha\text{-Si}_3\text{N}_4$ ) to a dense silica layer, which leads to a decrease of the pore volume [2, 11, 15]. The two-stages oxidation process of NBSC refractory can be explained by pore oxidation directed by the Knudsen diffusion [15]. Pores are coated with a silica layer at high temperature (1200°C). Most of the species reacts quickly in the pore mouth instead of diffusing inside the pore. Thus the pores are closed, so that porosity content decreases and the weight gain has stopped. This phenomenon concerns a given pore diameter range [2, 15].

NBSC thermomechanical properties were studied with the increasing test temperature [5, 8, 11, 12, 14, 16, 17]. Strength increases with temperature. Its comes from the bonding phase oxidation, which implies morphological changes [13]. Porosity is an important parameter in the material thermomechanical behaviour [10, 18].

This study plays a role in a research project concerning the behaviour of NBSC-tiles used in waste-to-energy plants. The tiles permit to cover the combustion chamber. They also bring protection to metallic tubes, which are used as energy collectors. Tiles are under high running solicitations. This article deals with the study of a NBSC material at as-received state. The material is then characterised after a long-term oxidation exposure. These steps will permit to understand the tile behaviour during running. The relationship between microstructure and thermomechanical behaviour will be done.

## 2. Material and experimental

### 2.1. Chemical composition

The material used in this study is mainly composed of SiC (80 %) and of nitrated phases (17 %). The presence of silica and additives has also been observed.

Table 1. NBSC phase composition [wt%].

SiC	$\text{Si}_3\text{N}_4 + \text{Si}_2\text{ON}_2$	$\text{SiO}_2 + \text{Si}$	Other
80	17	2t	1

A characteristic feature of this material is the high porosity content of 16 vol.%. Its density is equal to 2.65  $\text{g}/\text{cm}^3$ . These properties result from the processing route of NBSC materials.

### 2.2. Material presentation

Fig. 1 represents an optical observation of the material. Dense SiC aggregates are dispersed heterogeneously through the material. They result from the Acheson process and from a milling step. The aggregates aspect is dense and cleaved. They are surrounded by a nitrated bonding phase. At this magnification, it is not possible to observe bonding phase details. Fig. 1 also depicts the presence of a porosity network, which is here filled with an organic resin. A large amount of porosity is open.

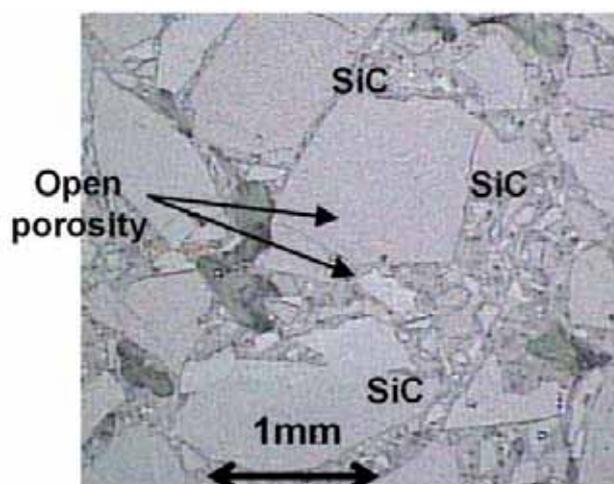


Fig. 1. Optical micrograph of the NBSC material.

### 2.3. Experimental methods

Because of the high complexity of NBSC microstructure, the use of complementary investigation methods will permit to better understand the material properties. XRD diffractograms are obtained with a spinner holder and a Cu-anticathode (Philips X-PERT). All measurements were made on powders in order to avoid material heterogeneities and then to permit to compare the results. Powders have been obtained by milling NBSC samples.

NBSC morphology is observed by optical and SEM microscopy.

The mass gain due to NBSC oxidation was measured on samples of 5 x 25 x 25  $\text{mm}^3$ , placed in a furnace at high temperature (800, 1000 and 1200°C) during 200 h in air. Samples were weighted at regular time. For each temperature, two samples were oxidised at the same time.

Four-point bending tests are carried out on a MTS 810 universal testing machine supplied with a high tem-

perature furnace (1600°C). The bending samples size is 150x25x25 mm<sup>3</sup>. The two lower rollers are 125 mm spaced and the two upper ones are 45mm spaced. Samples are loaded at a constant displacement rate of 0.2 mm/min. During the test, applied load and beam deflection are recorded.

### 3. Results and discussion

#### 3.1. As-received material

##### 3.1.1. Crystalline phase identification (XRD)

Complementary studies are made on NBSC in order to obtain more information on the material. Fig. 2 shows the NBSC diffractogram. High intensity peaks (close to  $2\theta = 35^\circ$ ) represent the silicon carbide phase. The intensity is high because the SiC content is closed to 80 wt%. Peaks with lower intensity, in the  $2\theta = 12^\circ$  to  $33^\circ$  range, represent the bonding phase, which is the material minor phase. The bonding phase is mainly made of silicon nitride phases, in a form  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> and  $\beta$ -Si<sub>3</sub>N<sub>4</sub>. Oxynitride is also present in the NBSC-bonding phase. The XRD analysis shows a  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> intensity lower than the  $\beta$ -Si<sub>3</sub>N<sub>4</sub> one. This may come from the finer grain size of the  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> phase.

As it was explained before, evolutions of the material mainly deal with the bonding phase. This diffractogram segment ( $2\theta = 12$ - $33^\circ$ ) will be further analysed in order to have specific indications on the phase evolutions with environmental conditions.

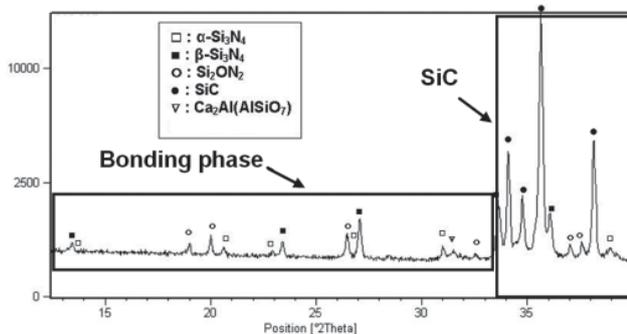


Fig. 2. As-received NBSC: XRD-diffractogram.

##### 3.1.2. NBSC microstructure at different scales

As it was explained before, a macroscopical observation at low magnification of the material reveals the presence of SiC aggregates linked together with a nitrated bonding phase and some open porosity channels. Fig. 3 represents the microstructure of the as-received material at a higher magnification. SiC aggregates are always dense and surrounded by the bonding phase. The presence of open porosity is emphasized in Fig. 3, compared to lower magnification. Porosity is present in the bonding phase. Fig. 3 shows different pore diameter sizes. Thus the NBSC bonding phase is porous and it can be described as a multimodal pore size distribution.

To detail the microstructure of the bonding phase, higher magnifications are required. Thus Fig. 4 represents the bonding phase at high magnification. Different morphologies,

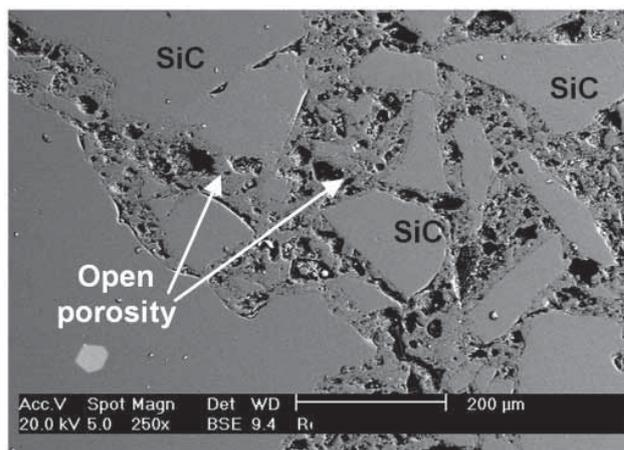


Fig. 3. SEM micrograph of the NBSC material.

which compose the nitride phase, are present in this picture. First of all the mean-rich grain size of the bonding phase is close to 1 micron and even below. Similarities are observed with the microstructure of a NBSC refractory previously studied by Zymla *et al.*. In that case the bonding phase is obtained by nitridation of silicium grains.

So the authors explain the Si grain nitridation as a transformation in 3 types of well-known Si<sub>3</sub>N<sub>4</sub> morphologies [9, 11]. According to Zymla, the core of Si grain is transformed in  $\beta$ -Si<sub>3</sub>N<sub>4</sub> rod-like crystals. Then silicon nitride is present in the  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> form firstly as a dense layer composed by small isotropic crystals. This is the  $\alpha$ -matte phase, which is followed by another  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> phase with needle shape crystals ( $\alpha$ -needle like). Thus the initial Si grain is transformed in a silicon nitride-based structural element, which is schematically represented by Zymla [9, 11]. This element is well organised but presents a high porosity level. Zymla claims that the specific area of the structural elements constitutes more than 95 % of the whole material specific area [9]. Fig. 4 can be compared to the schematic representation of an individual silicon nitride grain proposed by Zymla [11].

The bonding phase has a major role in the NBSC material, as it links the SiC aggregates together. The mechanical resistance of NBSC highly depends on the bonding phase properties. Due to the microstructure, the bonding phase plays the main role, as the nitride microstructure obtained with this process provides good mechanical properties.

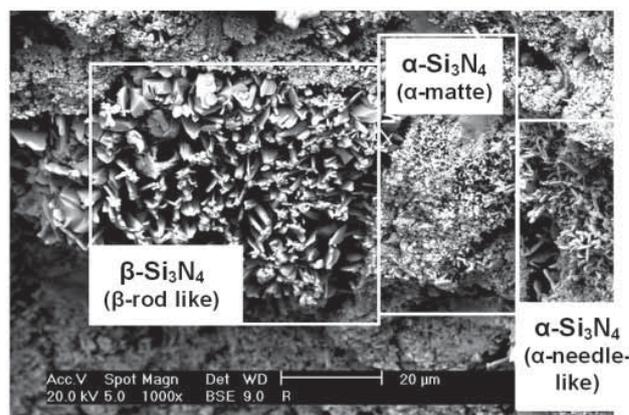


Fig. 4. Structural silicon nitride element (SEM micrograph).

The composition of NBSC bonding phase is characterised by a complex and highly porous microstructure with different porosity levels, such as the porosity in a structural element and the porosity channels with different pore diameters. Thus, the specific area of the bonding phase is much higher than the SiC specific area. It seems that during running solicitations of waste-to-energy plants, nitride phases will change with temperature. So the oxidation at high temperature should be studied in order to understand the NBSC behaviour with oxidation.

### 3.2. High temperature results: long-term oxidation

#### 3.2.1. Mass gain

Fig. 5 represents the mass gain evolutions versus time of NBSC oxidised at 800, 1000 and 1200°C for 200h in air. At 800 and 1000°C the mass gain curves of 2 samples are well overlaid for a given temperature. At 1200°C, a small deviation is observed. In Fig. 5 kinetics increases with oxidation temperature. The curve aspect at 800 and 1000°C is very similar. This is not the case at 1200°C. At this temperature, the weight gain fast increase initially and slows down after 8 hours of oxidation. Furthermore, the 1200°C curve is crossed by the 1000°C one. The oxidation mechanism seems to change between 1000°C and 1200°C.

The different oxidation kinetics measured during 200 h are comparable to NBSC kinetics previously studied [2, 10, 11]. Indeed, the oxidation occurs in two steps. At the beginning of oxidation, the surface exposed to oxidation is the sum of inner and external surfaces. This first step is rapid and the bonding phase is oxidised. In a second step, the silica layer has a significant thickness, which closes a given porosity range. So, the exposed surface is mainly reduced to the external surface, which slows down the oxidation kinetics.

Kinetics in Fig. 5 cannot be simulated by a parabolic model, so the kinetic do not result from the superposition of the oxidation of SiC aggregates and the oxidation of the nitrated bonding phase. The kinetic evolution of NBSC material shows that the oxidation of this material is complex. Not only one phenomenon plays a role in the oxidation mechanism.

The oxidation mechanism will be further explained. In order to better understand the oxidation effect on NBSC

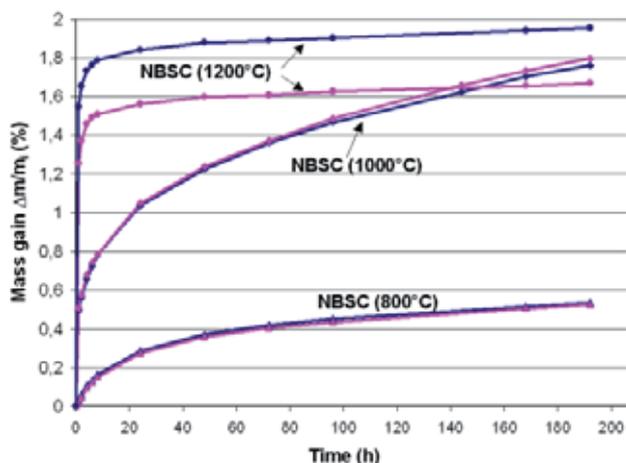


Fig. 5. Weight gain of NBSC material at 800°C, 1000°C and 1200°C for 200h in air.

material, further analysis should be done, such as the phase composition after high temperature exposure.

#### 3.2.2. Crystalline phase identification (XRD)

Oxidised samples were milled and then analysed by XRD. As it was explained before, the analysis in the bonding phase shows the evolution of the phases with oxidation. Fig. 6 represents the results obtained for different oxidation conditions. For high temperature (1000°C and 1200°C) a peak at  $2\theta = 22^\circ$  appears. It corresponds to  $\text{SiO}_2$  in different crystallised forms: cristobalite and tridymite. At 1000°C, this peak appears for a long-time exposure of 200 h, whereas it is present for the characterisation of a material exposed one hour at 1200°C. Except for a long exposure at 1200°C, the  $\text{SiO}_2$  peak is large and not very well defined. This may come from the crystal size of  $\text{SiO}_2$ . At 1000°C for 200 h, silica is present in the bonding phase as cristobalite and tridymite. These phases are present in the material when oxidation appears but the two peaks in the diffractogram overlay (spectrums 4, 5, 6 on Fig. 6). With oxidation, crystallites of the two silica phases grow. So the silica peaks are separated and the two phases appear clearly on the diffractogram (spectrum 7 on Fig. 6).

Other changes in the bonding phase are not significant.

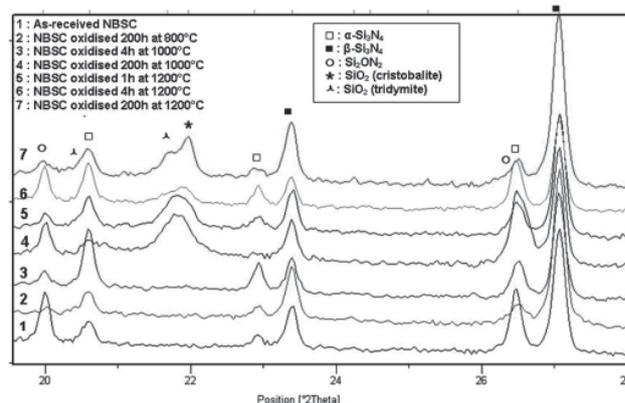


Fig. 6. Crystalline phases formed with temperature in the bonding phase (XRD diffractograms).

#### 3.2.3. Morphological evolutions

As the XRD analysis of oxidised sample shows changes in the bonding phase was also observed by SEM. Fig. 7 is based on the description of an individual silicon nitride grain described by Zymla [11]. Fig. 7 allows to observe the oxidation of each phase composing the structural element of the bonding phase.

In the case of  $\beta\text{-Si}_3\text{N}_4$ , rods of as-received material (A1) grow with temperature. At 1000°C the morphology is kept but rods are thicker (A2). After an exposure of 200h at 1200°C,  $\beta\text{-Si}_3\text{N}_4$  has lost the rod morphology and the phase is less porous than at 1000°C. So long-time exposure at high temperature modifies the morphology of  $\beta\text{-Si}_3\text{N}_4$  rods (A3).

The  $\alpha\text{-Si}_3\text{N}_4$  phase is present in two morphologies: a dense phase ( $\alpha$ -matte) and a porous phase ( $\alpha$ -needle like). Their evolution with the long-term exposure at high temperature differs, as their morphology is different. In the

case of the dense phase (B1), each grain is oxidised, thus at 1000°C the phase remains dense but the morphology seems to be more compact and the grains are not distinguishable (B2). At 1200°C oxidation follows, causing the loss of the initial morphology. The surface aspect is smoother (B3) and the surface porosity present in as-received NBSC (B1) is present no more.

The third nitride phase present in the bonding phase is  $\alpha$ -needle like  $\text{Si}_3\text{N}_4$ . It is present in the as-received material as small whiskers, thin and numerous (C1). At 1000°C, the temperature effect implies the oxidation of each whisker, so the diameter of whiskers increases (C2). At 1200°C, the oxidation effect is noticeable (C3), as few needles are present and their aspect seems melted.

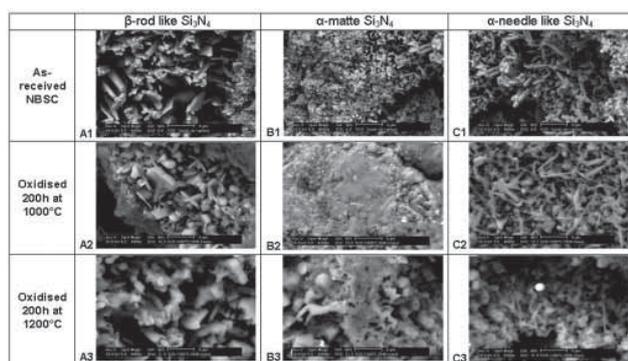


Fig. 7. Morphological evolutions with oxidation in air : comparison between as received material and NBSC oxidised 200h at 1000°C and 1200°C (SEM micrographs).

With oxidation, each grain of the different nitride-phases is covered by a silica layer, which grows with temperature and time exposure. The oxidation of each phase changes the surface aspect of its morphology. So with the long-term exposure at high temperature the surface porosity of the bonding phase due to the structural element decreases.

### 3.2.4. Oxidation mechanism

Oxidation of both components of the NBSC-refractory leads to the formation of a silica layer and to a volume expansion. Furthermore, thermodynamically SiC is oxidised before  $\text{Si}_3\text{N}_4$  but due to the bonding phase characteristics, such as its high porosity, and also due to the high specific area of  $\text{Si}_3\text{N}_4$ , in NBSC material, the bonding phase is mainly oxidised. In agreement with Zymła [11], the bonding phase is composed by an interlocked structure of nitride structural elements. During oxidation, the  $\alpha$ - $\text{Si}_3\text{N}_4$  phase is mainly affected by oxidation compared to the  $\beta$ - $\text{Si}_3\text{N}_4$  one. The  $\alpha$ -needle like crystals are particularly affected by oxidation and after a long-time exposure at high temperature, they form a porous silica layer around  $\beta$ -rod like core and  $\alpha$ -matte crystals, which are less affected by oxidation. In the area around the pore mouth, oxidation occurs with a different mechanism. Indeed due to porosity channels, oxidising atmosphere diffuses freely inside the NBSC material. So pore walls are totally oxidised and the initial  $\alpha$ -needle-like crystals, which are sintered by VLS mechanism, are oxidised. A dense silica layer then covers pore walls. This dense layer acts as a barrier to oxidation and protects the bonding phase surrounding the pore mouth

to oxidation. Thus around pores, there is a formation of a dense and passive silica layer, as oxidising atmosphere cannot diffuse inside the bonding phase. Furthermore, the dense silica layer grows with time exposure increasing the layer thickness. After a given time, the silica layer will reach a critical thickness and will lead to pore closure. So oxidation implies a decrease of material porosity volume. This case corresponds to the oxidation mechanism at 1200°C. Indeed the weight gain increases rapidly during the first hours (Fig. 5), corresponding to the oxidation of needle-like crystals and to the growth of dense silica layer inside pore channels. Then the weight gain slows down due to the closure of a given range of pore diameters. Oxidising atmosphere still circulates inside the material through the larger pore channels, which cannot be closed.

In agreement with NBSC kinetics (Fig. 5), the oxidation mechanism at 1000°C and 1200°C differs. Indeed at 1000°C, the weight gain still increases with time exposure, so that after 200 hours the weight gain at 1200°C and 1000°C are similar. Thus the whole bonding phase is still oxidised after 200 hours, as the kinetic does not seem to slow down. So oxidation at high temperature should be studied in order to understand the NBSC behaviour with oxidation.

## 3.3. Thermomechanical behaviour of NBSC

### 3.3.1. Thermomechanical behaviour of as received NBSC

NBSC material behaviour is tested with a four-points bending test, firstly in the as-received state and then after oxidation. Fig. 8 depicts the behaviour of the as-received NBSC refractory. At the test early beginning, a non-linear behaviour is observed. This phenomenon must be related to the strain levels, which take place at the contacts between the specimen and the loading rollers [19]. Then, the behaviour of NBSC becomes linear and elastic up to the material fracture. The material exhibits a brittle behaviour in all the considered

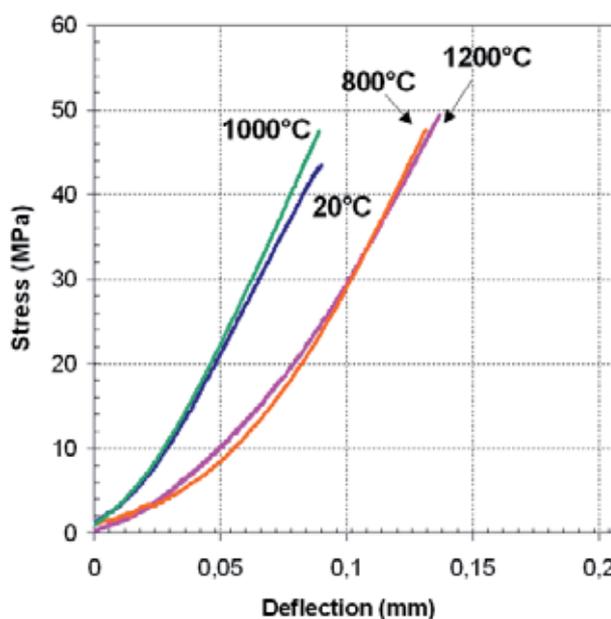


Fig. 8. Four-points bending behaviour of as-received NBSC refractory: evolution with testing temperature.

temperature. The rupture stress increases a little bit with testing temperature, whereas the flexural rigidity is slightly affected when the temperature increases. Such behaviour was already observed for similar materials [1, 5, 8].

### 3.3.2. Thermomechanical behaviour of oxidised NBSC

The thermomechanical behaviour is also characterised after an exposure of 200 h at high temperature in air, in order to determine the effect of long-term oxidation (Fig. 9). The material behaviour is the same compared to the as-received state. After long-term oxidation at 800°C and 1200°C, the thermomechanical behaviour of NBSC is slightly affected. At 1000°C, the rupture stress is higher than at other temperatures, whereas the rigidity is the same.

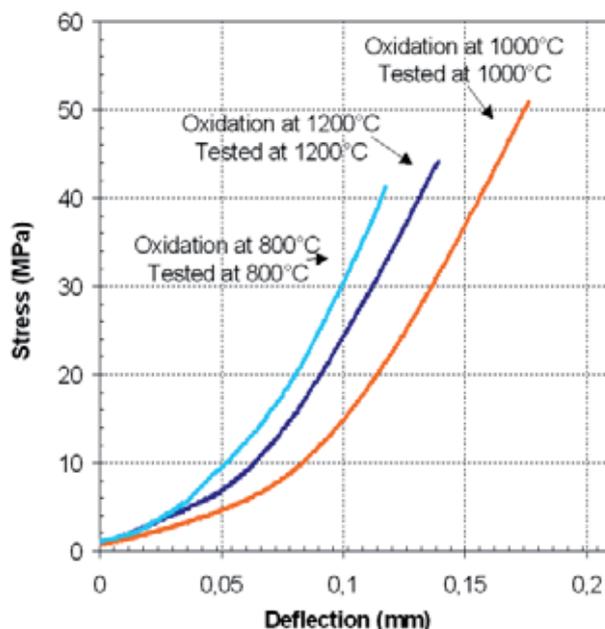


Fig. 9. Four-points bending behaviour of oxidised NBSC for 200h exposure in air: evolution with testing temperature.

### 3.4. Relationship between microstructure and thermomechanical behaviour after Oxidation

As it was shown in previous works [13], the thermomechanical behaviour and its evolution with oxidation temperature can be linked to the microstructure evolution. In the present study the thermomechanical behaviour of NBSC refractory can be linked with the oxidation mechanism occurring at each temperature. Indeed, it was explained that at 1200°C NBSC oxidation leads to a passive silica layer around pores, which prevents the bonding phase from oxidation. Thus, after long-term oxidation at 1200°C, the bonding phase does not seem to change a lot, except locally in surrounding pores. This fact is noticeable with bending test on NBSC refractory, as few changes appear between thermomechanical behaviours at the as-received state and at the 1200°C-200h oxidised state.

It was also explained that at 1000°C, the oxidation mechanism differs than at 1200°C. In this case, the oxidation seems to close a given range of pore diameters. This

mechanism hypothesis is verified with the thermomechanical behaviour of 200h-oxidised material at 1000°C. Indeed on Figure 9, the rupture stress is much higher than for the material oxidised at 1200°C.

This mechanism can also be considered at 800°C. But oxidation is very slow at this temperature, which implies few changes between the thermomechanical behaviour of the as-received NBSC refractory and after oxidation at 800°C for 200 h.

## 4. Conclusions

Refractories are heterogeneous materials. Tiles covering the combustion chamber of waste-to-energy plants are made of refractory materials. A SiC-based material is considered in this study: a nitride-bonded silicon carbide. This material is composed of two non-oxide components. SiC aggregates are bonded by a nitride phase. The SiC phase is dense and with grading size, whereas the nitride phase is highly porous and composed by nitride elements of micron as a grain size order. The complex microstructure of NBSC comes from its processing route. Thus the bonding phase is investigated in the as-received state. In waste-to-energy plants, tiles are subjected to high temperature and to oxidising atmosphere, which leads to the formation of a silica layer in the bonding phase. NBSC presents two oxidation mechanisms in air which depend on temperature. In this application NBSC-tiles are under high running solicitations. The as-received material offers high mechanical properties which evolve with oxidation temperature. These evolutions can be linked to the mechanical behaviour of the oxidised NBSC refractory in air.

Complementary works are now in progress concerning the influence of oxidation steam water on the material behaviour.

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