

Sintering, high temperature strength and oxidation resistance of liquid-phase-pressureless-sintered SiC–AlN ceramics with addition of rare-earth oxides

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Abstract

SiC–AlN composites can be pressureless sintered with the addition of rare-earth oxides (RE = Y, Yb, Er, Lu, Ho, Sm, Ce) and without protective powder bed. Sintered bodies showed high density ($\geq 97\%$ T.D.) and were mainly composed by 2H SiC–AlN solid solution with additive containing grain boundary phases. The high temperature strength and oxidation resistance of the composites were also investigated and correlated with the cationic radii of the oxide additives. The flexural strength at 1500 °C increased from 269 MPa to 587 MPa as direct consequence of the rare-earth cationic radius decrease. The specific weight gain during oxidation at 1500 °C for 200 h showed the same behaviour, decreasing from 12.60 mg/cm² to 3.13 mg/cm².

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1. Introduction

Liquid-phase sintered (LPS) silicon carbide is a candidate material for high temperature structural components. Sintering additives such as Al₂O₃–Y₂O₃ and AlN–Y₂O₃ were extensively used to obtain fracture resistant LPS–SiC ceramics.^{1–4} In the case of SiC–AlN–Y₂O₃ composites, previous studies reported that 2H SiC–AlN solid solution with improved high temperature properties could be achieved with SiC/AlN weight ratio greater than 80/20 and above 1850 °C.^{1,2} With SiC/AlN weight ratio of 50/50, densification is achieved by liquid-phase sintering using metal oxides such as yttria (Y₂O₃) as the sintering additives.³ This oxide reacts with SiO₂ and Al₂O₃ which are always present at the surface, respectively, of SiC and AlN particles, to form an oxynitride melt, Y₁₀Al₂Si₃O₁₈N₄.⁴

A problem associated with the use of additives is the resultant degradation of high temperature properties due to residual

grain boundary glassy phase.⁵ Several attempts to prevent the observed degradation in the high temperature properties have been reported.^{6–10} These include the use of different types of sintering additives in order to change the crystalline nature of the grain boundary phases in which these additives reside. For example silicon carbide ceramics containing AlN and Er₂O₃,^{7,9} Lu₂O₃,^{7,8,10} Sc₂O₃,^{7,10,11} Sm₂O₃,⁶ Yb₂O₃^{6,12} as sintering-aids were reported to show favourable high temperature properties and oxidation resistance, with SiC–AlN–Er₂O₃, SiC–AlN–Lu₂O₃ and SiC–AlN–Sc₂O₃ systems resulting in the best performance at high temperature due to the higher eutectic temperature of the grain boundary phases, RE₂Si₂O₇ (RE = Er, Lu, Sc) compared to oxynitride phases. All these studies are focused on SiC–AlN–RE₂O₃ ceramics with high SiC content (79–99 wt%) and sintered by hot-pressing or gas-pressure sintering, while studies on the effects of the nature of the grain boundary phases on the high temperature behaviour of pressureless-sintered 2H SiC–AlN composites have not been published yet. Our purpose was to examine the effectiveness of seven rare-earth oxides (Y₂O₃, Er₂O₃, Ho₂O₃, Lu₂O₃, Sm₂O₃, Yb₂O₃, CeO₂) as densification aids for SiC (50 wt%)–AlN

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Table 1
Characteristics of the starting powders.

Powder	Purity (wt%)	Specific surface area (m ² /g)	Particle size (μm)
α-SiC	>97.0	15.6	0.48
AlN	>98.0	3.3	0.1–0.5
Y ₂ O ₃	99.9		<5
Lu ₂ O ₃	99.9		<5
Er ₂ O ₃	99.9		<5
Yb ₂ O ₃	99.9		<5
CeO ₂	99.0		<5
Ho ₂ O ₃	99.9		<5
Sm ₂ O ₃	99.9		<5

(50 wt%) ceramics sintered by an innovative pressureless process without protective powder bed. Another important purpose was to investigate how the sintering additives and, hence, the intergranular phase chemistry influence the mechanical properties and oxidation resistance of the SiC–AlN composites.

2. Experimental procedure

Commercially available α-SiC (UF10, H.C. Starck, Germany), AlN (Grade F, Tokuyama Soda, Japan), Y₂O₃ (H.C. Starck, Germany), Er₂O₃, Ho₂O₃, Lu₂O₃, Sm₂O₃, Yb₂O₃ (Cerac Inc., USA) and CeO₂ (Sigma–Aldrich, Switzerland) were used as starting powders. Characteristics of these powders are reported in Table 1.

A batch of each powder composed by 48 wt% SiC–48 wt% AlN–4 wt% RE₂O₃ was wet-mixed in ethanol for 12 h using SiC grinding balls. After drying and sieving, the powder was compacted by die pressing at 67 MPa and subsequently was pressed at 150 MPa by CIP.

Sintering was always performed in a graphite element furnace in flowing nitrogen at 1 atm with green bodies put inside a graphite crucible without protective powder bed. Sintering was performed at 1950 °C, while an annealing step of 4–6 h was conducted at 2050 °C. Thermal cycle was characterised by heating and cooling rate of 20–30 °C/min and by dwell time of 0.5 h at the sintering temperature.

The bulk densities of the sintered samples were determined using the Archimede method. The microstructures were characterized using scanning electron microscopy (SEM), while X-ray patterns (XRD), were collected with a Philips powder diffractometer, with a Bragg–Brentano geometry, equipped with a Copper anode operated at 40 kV and 30 mA, step 0.02°, time

6 s. The phase analysis was carried out with the PC X'pertHigh Score software Version 2.2a (PANalytical B.V., Almelo, The Netherlands).

Oxidation experiments were carried out at 1500 °C over a period of 200 h in air. Rectangular pellets (18 mm × 3 mm × 3 mm) were prepared from the bulk specimens with a diamond saw. After grinding to reduce superficial roughness, the specimens were cleaned in an ultrasonic bath and degreased with acetone and ethanol. Dried samples were then weighed and the exact dimensions were measured in order to calculate the surface area. The experiments were conducted in a furnace having molybdenum disilicide heating elements.

Finally, flexural strength was determined by four-point bend tests at 1500 °C (ten samples for each composition). Samples as bars of 2 mm × 2.5 mm × 25 mm were prepared and tested in accordance with the standard ENV 843-1 (crosshead speed 0.5 mm/min).

3. Results and discussion

3.1. Sintering mechanism and microstructure

Bulk density and crystalline phases of the sintered samples are reported in Table 2. The sintered densities of SiC–AlN ceramics were found to be ≥97% of the theoretical density as calculated by the rule of mixtures. Hence, the proposed oxide sintering-aids are suitable for the densification of SiC–AlN composites. In fact, thermodynamic calculations confirmed the stability of SiC in the presence of the studied RE₂O₃ during thermal treatment.¹³ At the same time decomposition of AlN at the sintering temperature was efficiently suppressed by a N₂ atmosphere.¹⁴

Rare-earth oxides participate to the formation of the liquid phase that assists densification during pressureless sintering³ and precipitates at the grain boundary during cooling. A core-rim microstructure was put in evidence by plasma-etching as a consequence of a solution and reprecipitation mechanism with larger grains of free-SiC growing at the expense of smaller ones (Fig. 1).

Grain boundary phases such as Y₁₀Al₂Si₃O₁₈N₄ with Y₂O₃ addition⁴ and RE₄Si₂N₇O₂/RE₂Si₃N₄O₃ with RE = Er, Lu, Ho, Sm, Yb^{9,12,15,16} as sintering-aids were formed after sintering. Cerium oxide (CeO₂) promotes densification following a more complex mechanism. On the basis of previous study based on the hot-pressing of Si₃N₄–CeO₂ system,¹⁷ it can be hypothesised that during the pressureless sintering pro-

Table 2
Density (%T.D.) and crystalline phases of the SiC–AlN–RE₂O₃ composites (mp = main phase, sp = secondary phase, gbp = grain boundary phase, tr = trace).

Material	Bulk density (%T.D.)	Crystalline phases
SiC–AlN–Y ₂ O ₃	97	2H SiC–AlN (mp), α-SiC (sp), Y ₁₀ Al ₂ Si ₃ O ₁₈ N ₄ (gbp), α-Cristobalite (tr)
SiC–AlN–Lu ₂ O ₃	98	2H SiC–AlN (mp), α-SiC (sp), Lu ₂ O ₃ (gbp), Lu ₂ Si ₂ O ₇ (gbp), α-Cristobalite (tr)
SiC–AlN–Er ₂ O ₃	99	2H SiC–AlN (mp), α-SiC (sp) Er ₂ O ₃ (gbp), Er ₂ Si ₂ O ₇ (gbp)
SiC–AlN–Ho ₂ O ₃	99	2H SiC–AlN (mp), α-SiC (sp), Ho ₂ O ₃ (gbp), Ho ₂ Si ₂ O ₇ (gbp), Ho ₄ Si ₂ N ₇ O ₂ (tr)
SiC–AlN–Yb ₂ O ₃	98	2H SiC–AlN (mp), α-SiC (sp), Yb ₂ O ₃ (gbp), Yb ₂ Si ₂ O ₇ (gbp), α-Cristobalite (tr)
SiC–AlN–Sm ₂ O ₃	98	2H SiC–AlN (mp), α-SiC (sp), Sm ₂ O ₃ (gbp), Sm ₂ Si ₂ O ₇ (gbp)
SiC–AlN–CeO ₂	97	2H SiC–AlN (mp), α-SiC (sp), Ce ₂ SiO ₅ (gbp), Ce ₂ Si ₂ O ₇ (gbp), α-Cristobalite (tr), Ce _{4,67} (SiO ₄) ₃ O(tr)

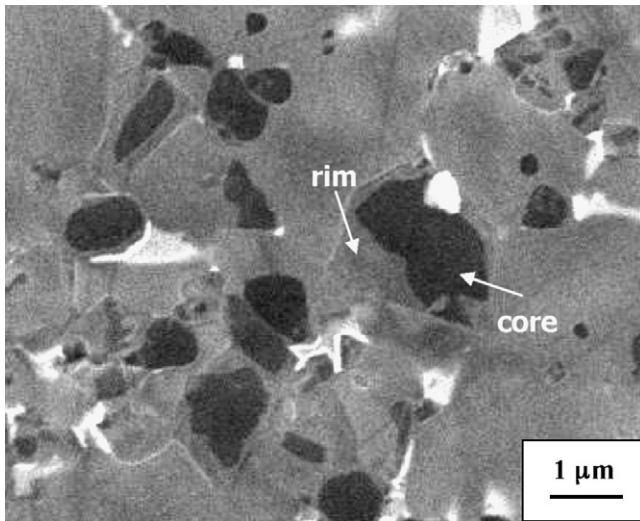


Fig. 1. Microstructure of SiC–AlN–Y₂O₃ showing core-rim structure of the SiC grains.

cess, CeO₂ is mostly reduced to Ce₂O₃ by reaction with SiO₂ located on the surface of SiC particles.¹⁸ XRD analysis of pressureless-sintered SiC–AlN–CeO₂ confirmed that Ce(III)-crystalline phases (Ce_{4.67}(SiO₄)₃O and Ce₂Si₆N₈O₃) were formed as grain boundary phases (Fig. 2). In addition, residual Ce_{4.67}(SiO₄)₃O was found in the annealed SiC–AlN–CeO₂ ceramics where Ce₂SiO₅ and Ce₂Si₂O₇ were also detected as consequence of the decomposition of Ce_{4.67}(SiO₄)₃O and Ce₂Si₆N₈O₃ (Fig. 2 and Table 2). According to the XRD pattern reported in Fig. 2, the majority of intergranular phases were

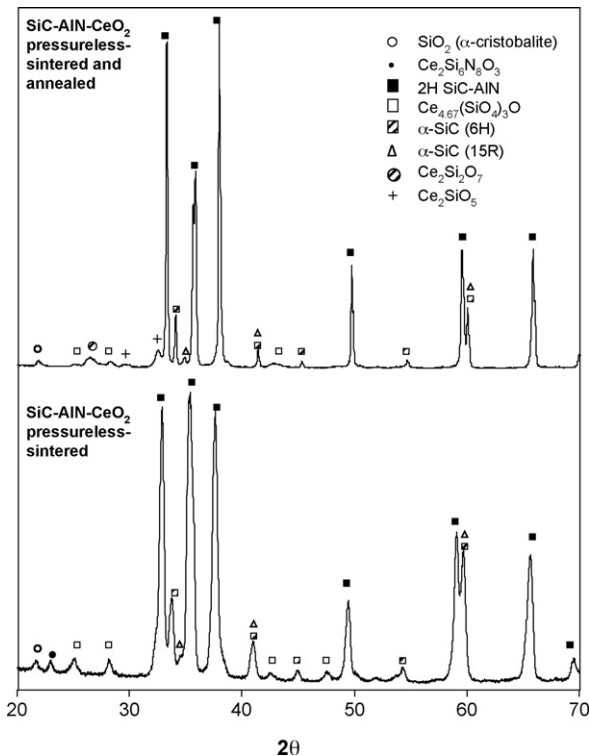


Fig. 2. XRD pattern of SiC–AlN–CeO₂ composites before and after annealing (2050 °C × 4 h).

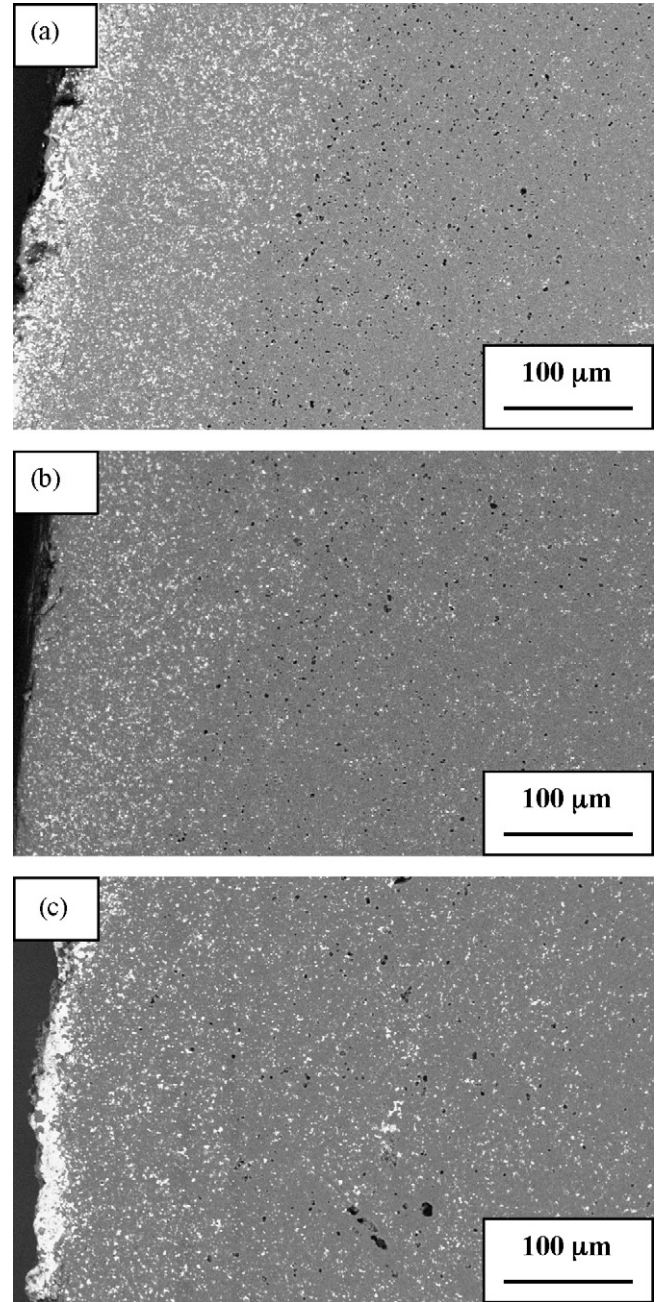


Fig. 3. SEM images of SiC–AlN–Y₂O₃ after an annealing time of (a) 1 h, (b) 2 h and (c) 4 h.

Table 3

Flexural strength at 1500 °C and RE³⁺ radius values of the SiC–AlN–RE₂O₃ composites.

Material	Flexural strength at 1500 °C (MPa)	Cationic radius of RE ⁿ⁺ (10 ⁻¹ nm)
SiC–AlN–Y ₂ O ₃	479	1.019
SiC–AlN–Lu ₂ O ₃	521	0.977
SiC–AlN–Er ₂ O ₃	587	1.004
SiC–AlN–Yb ₂ O ₃	430	0.985
SiC–AlN–CeO ₂	286	1.140
SiC–AlN–Ho ₂ O ₃	400	1.015
SiC–AlN–Sm ₂ O ₃	423	1.079

crystalline in as-sintered and annealed materials showing sharp reflections in the XRD pattern, clear indication of the low amount of residual amorphous phases. In any case, additional work will be needed in order to evaluate the amount of amorphous phases as a function of the cationic radius of the oxide additives.

Upon annealing, the oxynitride phases ($\text{RE}_4\text{Si}_2\text{N}_7\text{O}_2/\text{RE}_2\text{Si}_3\text{N}_4\text{O}_3$) slowly decompose to oxide/silicate ($\text{RE}_2\text{O}_3/\text{RE}_2\text{Si}_2\text{O}_7$) phases¹⁵ (Table 2). Furthermore, secondary phases

are expelled from the grain boundary region upon annealing, and very small amounts of residual secondary phases remain at the grain boundary. The reduction of residual secondary phases occurs due to the volatilisation of these phases via carbothermal reductions (graphite-heated furnace).¹⁹ Movement of liquid phase from the grain boundary to the surface has been already shown by Lee et al. in SiC–YAG composites²⁰ which presented a porous reaction-layer of 90 μm on the outside region

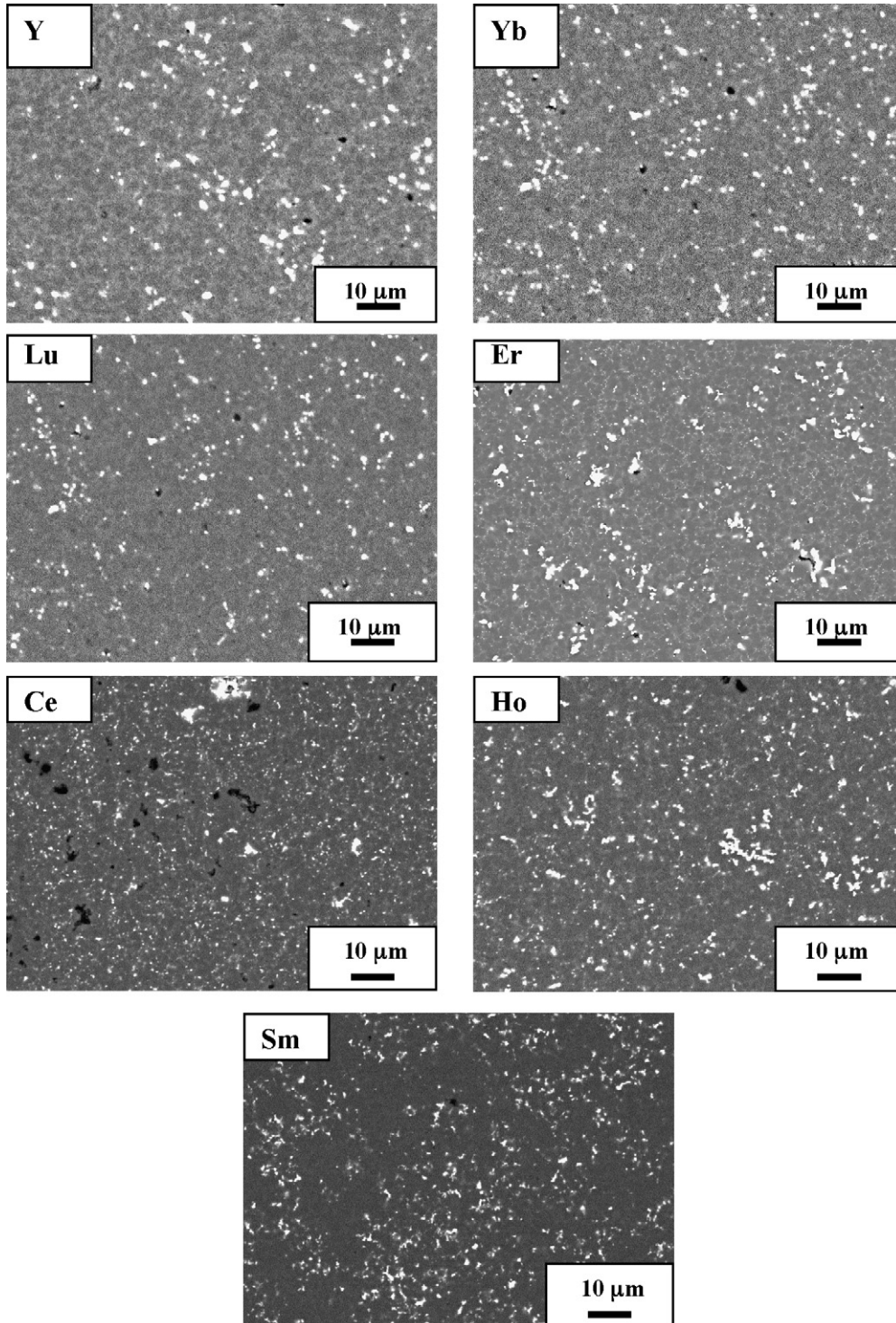


Fig. 4. SEM images of the microstructure of the SiC–AlN– RE_2O_3 (RE = Y, Yb, Er, Lu, Ho, Sm, Ce) ceramics after pressureless sintering and annealing.

of the specimens. Fig. 3 shows typical behaviour of the grain boundary phases during thermal annealing of sintered SiC–AlN ceramics. SEM micrographs reported here are relative to the SiC–AlN–Y₂O₃ system and clearly demonstrate that the grain boundary phase (Y₁₀Al₂Si₃O₁₈N₄) melts during annealing and moves in the direction of low Gibbs free energy, i.e. the surface, where it volatilises as SiO or Al₂O.²¹

After annealing, residual grain boundary phase is homogeneously distributed in the samples. Images reported in Fig. 4 and obtained with back-scattered electrons put in evidence the bright spots of the liquid phase in the SiC–AlN–RE₂O₃ composites.

3.2. Mechanical properties

The flexural strength values measured at 1500 °C on sintered specimens are presented in Table 3. The highest strength of 587 MPa was obtained with the SiC–AlN–Er₂O₃ ceramics, while SiC–AlN–Ho₂O₃ ceramics showed lower strength values as compared to the others. The variation of the high temperature strength with the radius of the cation in the oxide additive is shown in Fig. 5. The cationic radii used in this study are listed in Table 3.²² As shown, the high temperature strength of SiC–AlN–RE₂O₃ ceramics is strongly dependent on the cationic radius of the oxide additive. A similar correlation between the ionic radius of rare-earth oxides additives and mechanical properties was also observed in the case of liquid-phase sintered SiC–Al₂O₃–RE₂O₃,²³ hot pressed SiC–AlN–RE₂O₃⁶ and hot pressed Si₃N₄–RE₂Si₂O₇²⁴ ceramics. In these studies the difference in the mechanical properties was attributed to the chemistry of the intergranular phases. Shelby et al.²⁵ firstly reported that the physical properties of rare-earth aluminosilicate glasses, such as glass transformation temperature and thermal expansion coefficient, varied linearly with the ionic radius of the rare-earth ions. Similarly, the rare-earth ions might play a major structural role in intergranular phases found in the SiC–AlN ceramics. Since the RE³⁺ ions are all trivalent, their field strength (the effective force to attract neighbouring anions) varies with the reciprocal of the ionic radius. So, the smaller RE³⁺ ions made the network more compact and tight and, consequently, produced stronger intergranular phases. Under such conditions, the stronger grain boundary would result in higher flexural strength of ceramics.

3.3. Oxidation behaviour

Fig. 6 is a plot of weight gain per unit surface area as a function of time at 1500 °C. All materials had specific weight gains on the order of 2–7 mg/cm² after 200 h with the exception of SiC–AlN–CeO₂ which presented a weight gains of 12 mg/cm² after the same time at temperature. The oxidation of pressureless-sintered SiC–AlN–RE₂O₃ has been shown to obey a parabolic rate law of the type:

$$\Delta W^2 = kt \quad (1)$$

where ΔW is the specific weight gain, k is the kinetic constant of parabolic oxidation and t is the oxidation time.

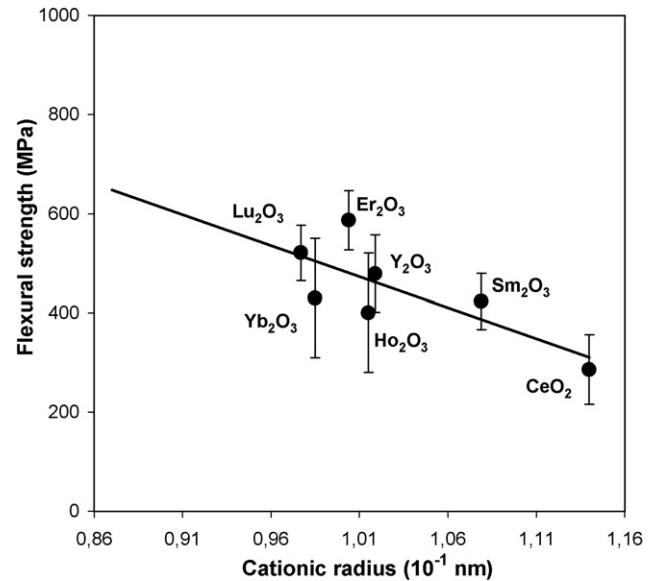


Fig. 5. High temperature strength as function of RE³⁺ radius.

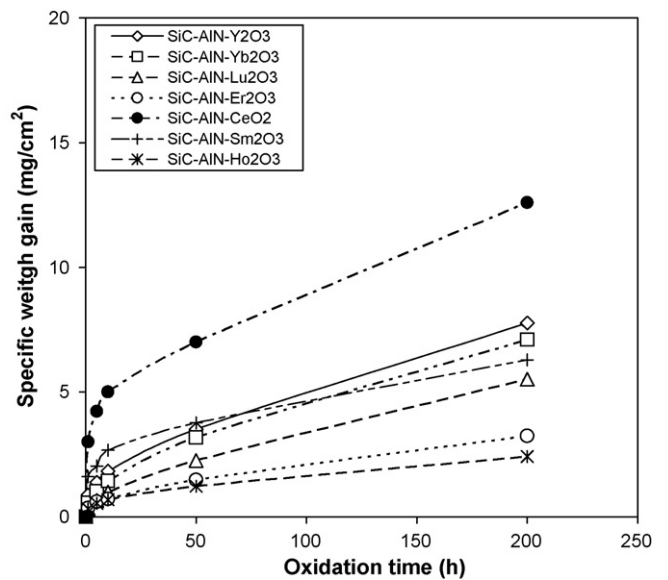


Fig. 6. Specific weight gain as function of the oxidation time ($T = 1500$ °C).

Table 4

Specific weight gains and constants rate of the different SiC–AlN–RE₂O₃ ceramics after oxidation at 1500 °C for 200 h.

Material	Specific mass gain (mg/cm ²)	Rate constant (mg ² /(cm ⁴ h))
SiC–AlN–Y ₂ O ₃	7.77	2.99×10^{-1}
SiC–AlN–Lu ₂ O ₃	5.52	1.49×10^{-1}
SiC–AlN–Er ₂ O ₃	3.25	5.22×10^{-2}
SiC–AlN–Yb ₂ O ₃	7.09	2.49×10^{-1}
SiC–AlN–CeO ₂	12.6	7.47×10^{-1}
SiC–AlN–Ho ₂ O ₃	3.13	4.92×10^{-2}
SiC–AlN–Sm ₂ O ₃	6.29	1.86×10^{-1}

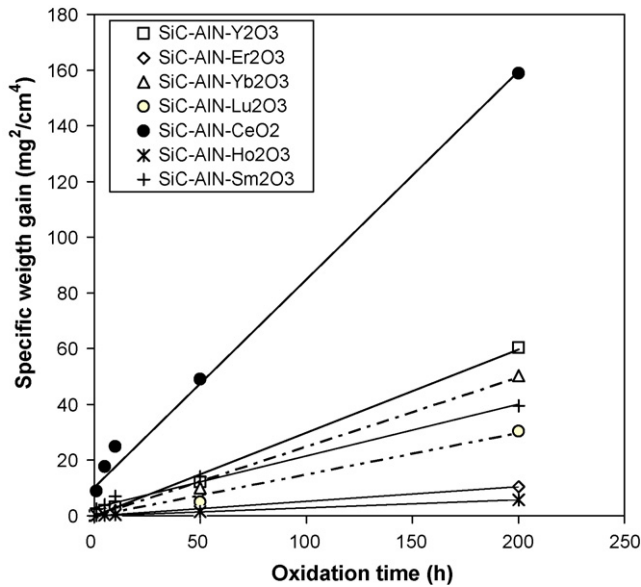


Fig. 7. Square of the specific weight gain as function of the oxidation time ($T=1500\text{ }^{\circ}\text{C}$).

Plotting these data, results in straight lines for all curves are shown in Fig. 7. Table 4 contains the parabolic oxidation rate constants (slope of the straight lines) for the seven compositions investigated, as well as the total specific weight gains after 200 h. The parabolic oxidation behaviour of these ceramics indicates a diffusive process as the rate-limiting step associated with the migration of additive cations (RE^{3+}) towards the interface between ceramic and the surface oxide.²⁶

The high oxidation resistance of these materials, demonstrated by the values of specific weight gains of Table 4, can be attributed both to a minimal amount of amorphous grain boundary phases, with high viscosity due to the presence of refractory RE^{3+} , and to thermodynamically stable crystalline secondary

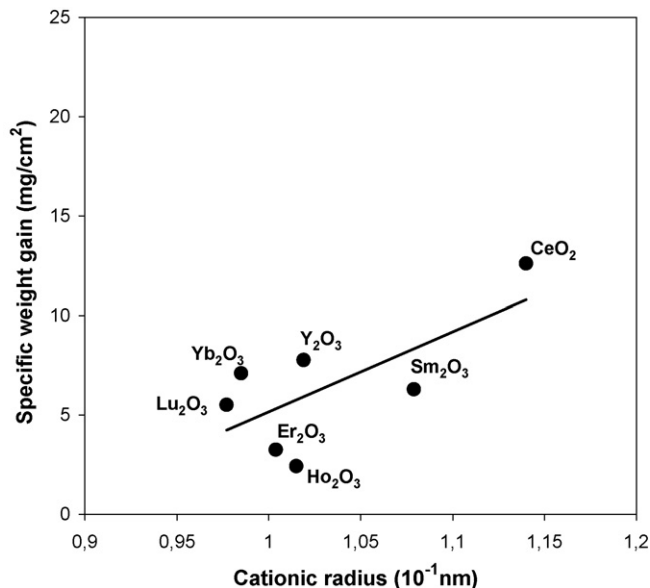


Fig. 8. Specific weight gain as function of RE^{3+} radius.

phases, such that the driving force for additive cation diffusion to the surface is minimized.

In addition, a greater refractory nature of the amorphous grain boundary phase is expected for those materials sintered with the oxide additives having a smaller cationic radius.²⁴ As a consequence it was observed that the specific weight gains of the specimens during oxidation were dependent on the cationic radius of the oxide additives (Fig. 8).

4. Conclusion

SiC–AlN composites can be obtained by pressureless-sintering process with different RE_2O_3 ($\text{RE} = \text{Y}, \text{Yb}, \text{Er}, \text{Lu}, \text{Ho}, \text{Sm}, \text{Ce}$) as densification-aids and without protective powder bed. The main characteristics of the sintered samples are:

1. The microstructure presents SiC grains, showing a core-rim structure, dispersed into a matrix composed by solid solution 2H SiC–AlN. Rare-earth oxides form oxynitride species, which precipitate at the grain boundary and decompose during annealing at high temperature to produce silicate phases ($\text{RE}_2\text{Si}_2\text{O}_7$).
2. High temperature strength reaches the highest value (587 MPa) with SiC–AlN– Er_2O_3 composites. A strong dependence between flexural strength and cation radius of the rare-earth oxides has been also showed. Smaller RE^{3+} ions produce stronger intergranular phases, which leads to a higher flexural strength of SiC–AlN ceramics.
3. Pressureless-sintered SiC–AlN– RE_2O_3 composites show a high oxidation resistance at $1500\text{ }^{\circ}\text{C}$. In particular, SiC–AlN– Ho_2O_3 and SiC–AlN– Er_2O_3 exhibit the lowest values of specific weight gain after oxidation at $1500\text{ }^{\circ}\text{C}$ for 200 h. Oxidation always follows a parabolic rate law with migration of RE^{3+} ions as rate-limiting step. In addition, cationic radius of the oxide additives greatly influence the oxidation resistance of the different SiC–AlN– RE_2O_3 ceramics due to the different level of refractoriness of the residual amorphous grain boundary phase.

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