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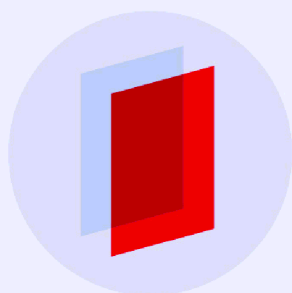
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Thermal shock resistance of pressureless sintered SiC/AlN ceramic composites

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Abstract

This paper conducts for the first time, a study of thermal shock resistance properties of different pressureless sintered SiC/AlN ceramic composites. SiC/AlN composites with different AlN content (0–40 wt%) were obtained by pressureless sintering of SiC and AlN powders at temperature of 2080 °C/2 h in Ar/vacuum atmosphere with using 2.5% yttria and alumina as sintering aids. The different ceramic composites are subjected to 20 thermal shock/water quenching cycles at temperature of 700 °C. Mechanical strength and microstructure investigation before and after thermal shock exposure were used to evaluate thermal shock properties and durability of the different composites. It was found that, the investigated carbide/nitride ceramics gave high thermal shock resistance without any cracking or spalling with retention of about 70% of its original strength. Results suggest that, SiC/AlN composites are strong candidate in severe thermal shock resistance environments.

1. Introduction

Thermal shock resistance (TSR) of a material is the ability of this material to withstand sharp changes in temperature. It is the name given to the resistance to cracking as a result of rapid temperature change.

When the ceramic material is rapidly cooled after heating, its surface reaches the temperature of cooling environment and tends to contract (thermal contraction). Since the interior regions of the material are still hot, thermal contraction of the skin surface is impossible. This leads to formation of tensile stress (thermal stress) in the skin. Such thermal stress leads to initiation of cracks formation. If nothing stops these cracks from propagating through the material, it will cause the material's structure to fail. Glass and ceramics are particularly vulnerable to this form of failure, due to their low toughness and low thermal conductivity [1].

TSR is considered one of the most important and everlasting issue that facing ceramic materials especially when they used in structural and mechanical applications under severe thermal environments. The continuous exposure of rapid heating/cooling cycles can generate thermal stresses large enough to cause failure and spalling of the monolithic materials. So that, searching for advanced ceramic materials with high TSR and thermal properties become an inevitable exigency.

Many efforts and attempts have been exerted in order to enhance the thermal shock behavior of the monolithic ceramic materials by the addition of other phases, reduction of their thermal expansion coefficient, improvement of their toughness mechanism and strength [2, 3].

It was found that, TSR can be improved by forming a variety of different composite architectures. Some of the methods that have been used to improve the TSR compared with monolithic ceramic materials include engineered porosity [4] that results in crack tip blunting [5], fibrous/platelet/whisker inclusions [6–8] that

Table 1. Designation (wt%) of different SiC/AlN composites.

Sample	SiC (wt%)	AlN (wt)
0 YA	100%	0%
1 YA	90%	10%
2 YA	80%	20%
3 YA	70%	30%
4 YA	60%	40%

produce crack bridging and crack deflection, metal additions that give functionally graded materials [9] or a randomly distributed second phase [10] that produces a high-low modulus composite [11].

On the other side, SiC/AlN ceramic composites are expected to be beneficial and important for consideration in new severe applications with predictable high thermal shock properties. This can be attributed to the excellent appealing properties of SiC and AlN monolithic ceramics. SiC ceramics have high hardness, toughness, Young's modulus, excellent absorption, high temperature strength, high seebeck coefficient, good electronic properties, high creep resistance, thermal shock and oxidation resistance at high temperatures [12]. However, AlN ceramics have high thermal conductivity, low coefficient of thermal expansion, high TSR, high corrosion and oxidation resistance, electrical resistivity and high mechanical strength [13–15].

Due to the great importance of SiC/AlN ceramics system, their properties, applications and synthesis, they have attracted material scientists' attention during the last decade. SiC and AlN composites can give new materials that combine the excellent properties of the two materials in one structure.

Producing SiC/AlN composites with high TSR is expected to be beneficial and important for consideration in new advanced applications, such as microwave absorption in high-power amplifiers and microwave components, sensor materials, thermoelectric conversion elements, high temperatures and as volumetric receiver in solar energy technology [16–19].

Several works have extensively studied the processing of SiC/AlN ceramics by different techniques such as chemical vapor deposition, spark plasma sintering, self propagating combustion synthesis and pressureless sintering [18, 20–26]. However, very limited works have hardly studied their thermal and mechanical properties specifically at high temperatures. Besides, no works have studied or investigated their TSR before.

The purpose and the main idea of this work is optimizing new carbide/nitride composites with high thermal shock properties that capable of working under severe thermal conditions. Additionally, from the industrial point of view, this target should be achieved by a simple and low cost method. So that, in this study we report on for the first time the evaluation of TSR properties of different pressureless sintered SiC/AlN ceramic composites. SiC/AlN composites with different AlN content (0–40 wt%) were obtained by pressureless sintering of SiC and AlN powders at temperature of 2080 °C/2 h in Ar/vacuum atmosphere with using 2.5% yttria and alumina as sintering additives. Mechanical strength and microstructure investigation before and after thermal shock exposure were used to evaluate thermal shock properties and durability of the different investigated composites. Moreover, the influence of different AlN wt% on the TSR, densification, microstructure and strength of the different composites will be inspected and analyzed.

2. Experimental procedure

Different SiC/AlN composites were produced with different AlN content (0%–40%) as indicated in table 1. The powder mixture of each composite containing high purity 99.99% AlN and SiC powders was ball planetary milled in 100% ethanol solution for 5 h using a zirconia ball to produce a homogenous mixture and subsequently dried at 60 °C for 24 h. 2.5% yttria and alumina powders were used as sintering aid to enhance the composites sinterability. Green compacts in cylindrical form with dimensions of ≈ 20 mm in diameter and ≈ 5 mm in height were produced by uniaxial cold pressing (KPD-30 A, Spain) at 200 MPa. Finally, pressureless sintering of the green compacts was performed at a temperature of 2080 °C/2 h with heating rate of 10 °C in a high temperature graphite furnace in argon/vacuum atmosphere. Densification parameters in terms of apparent porosity and bulk density of the produced composites were measured by Archimedes immersion method.

For the TSR test, the different SiC/AlN composites were heated in air inside a muffle furnace to a temperature of 700 °C. The soaking time for each sample was 15 min, followed by cold water quenching to provide a sudden temperature change. The treated samples were subjected to microscopic and macroscopic examination to determine damage/distortions if any developed on surface or sub surface regions. In all cases, the thermal shock treatment was repeated for 20 cycles on the same sample to see the behavior under repeated

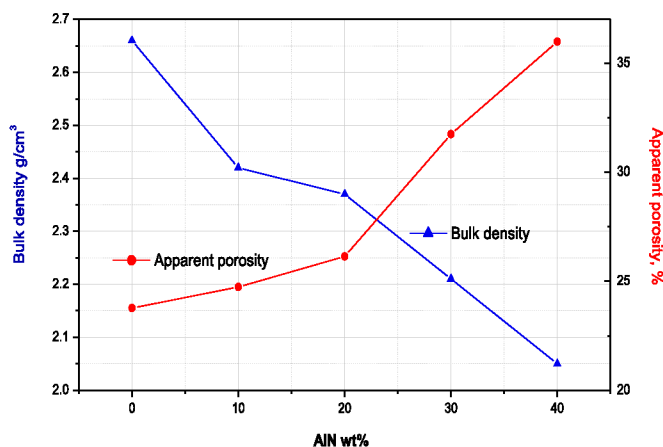


Figure 1. Densification parameters of different SiC/AlN composites sintered at 2080 °C/2 h in Ar/vacuum.

thermal shock. Mechanical strength cold crushing strength (CCS) and microstructure were studied after thermal shock treatment and compared with the results from untested sample data. This test was conducted according to (PRE/R5 standard) [27].

CCS of the different tested and non tested SiC/AlN composites was determined according to (ASTM C 1424-04) [28].

To measure the CCS, test was carried out using the universal testing machine (ТОЧПРИБОР Corporation made in Russia-model 5057-50 YXJI 4.2—Capacity 50 kN). The specimen was carefully placed into two load blocks and alignment of the specimen in the load blocks was ensured. The load was slowly applied with crosshead rate 1 mm min^{-1} . CCS was calculated according to the following formula [29]:

$$\text{CCS} = W/A,$$

where

CCS = Cold crushing strength (N mm^{-2}),

W = Maximum load (N),

A = the cross section area of the specimen (mm^2).

The obtained microstructures of the different SiC/AlN composites examined by using backscattered electron in the field emission scanning electron microscopy (FESEM; QUANTA FEG250, Holland).

3. Results and discussion

Densification parameters in terms of apparent porosity and bulk density of different sintered SiC/AlN composites are depicted in figure 1. It was inferred that, composites with high SiC content gave the best densification behavior. They gave the highest density of 2.66 g cm^{-3} for composite 0YA and the lowest porosity of 23.7% for the same composite. However, increasing AlN wt%, deteriorated their densification behavior. Composite 4YA gave the lowest density 2 g cm^{-3} of and highest porosity of 36%.

TSR of the different pressureless sintered SiC/AlN composites was evaluated by measuring the difference in the CCS between the thermal shock tested and untested sample.

The CCS distribution across the different SiC/AlN composites before and after thermal shock treatments is presented in figure 2. No spalling or cracking is observed through the 20 cycles for the different composites. The comparison between tested and untested composites revealed that: the strength is slightly lower in thermally exposed samples than in the unexposed one. This reduction is due to the softening of the matrix caused by the thermal exposure (thermal cycling and shock treatments).

CCS of thermal shocked samples shows the same behavior of unshocked samples; it is gradually decreased with addition of AlN wt% due to increasing the porosity and decreasing the density as explained in figure 1. However, the reduction % in TSR values between tested and untested composites reduced gradually by the increase in SiC and the decrease of AlN contents as given in table 2. Composite 0YA gave the highest TSR value. It gave 20 cycles with retention of about ≈ 83.5 of its original strength. While by increasing AlN wt%, this percentage decreased gradually until reach to composite 4YA with retention of 67% of its original value.

According to Buchheit *et al* [30], this thermal shock behavior belongs to (regime I) where strength of thermally shocked samples are about 70% of the unshocked samples strength. In this type, quenched samples show no strength degradation compared to unquenched samples because the stresses generated during

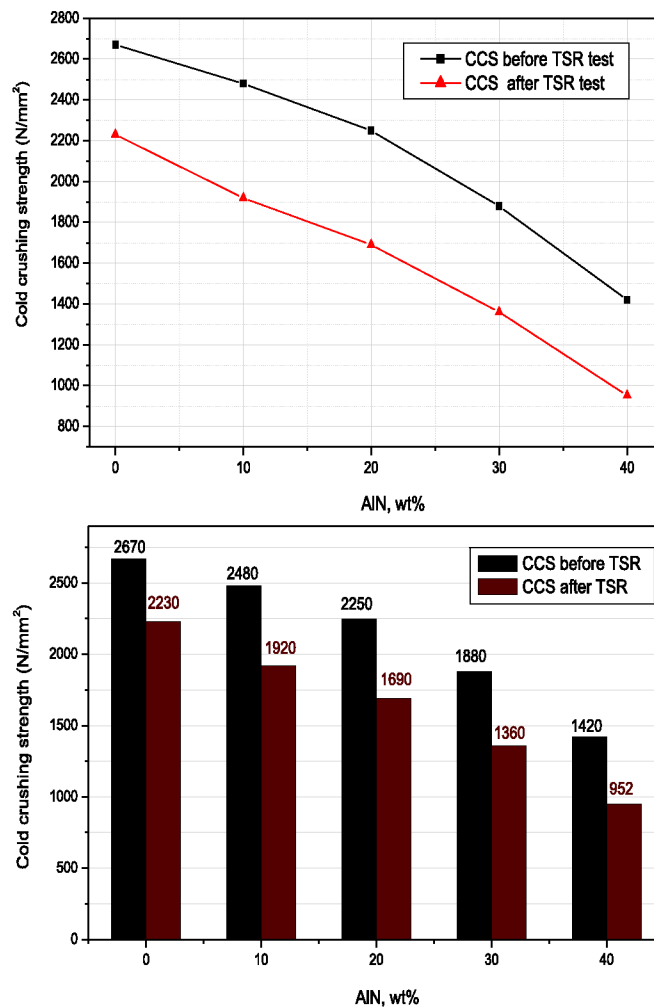


Figure 2. CCS distribution of the different SiC/AlN composites before and after thermal shock treatment.

Table 2. CCS reduction, % of the different SiC/AlN composites before and after TSR.

Composite designation	CCS reduction, %
0 YA	16.47
1 YA	22.58
2 YA	24.88
3 YA	27.65
4 YA	32.95

quenching are lower than those required for crack initiation or for growth of cracks already present in the specimen. This TSR demeanor of SiC/AlN composites can be attributed to presence of porosity that results in cracks tip blunting and elongated grains behavior which in turn lead to crack bridging and crack deflection as stated and confirmed by other works [3–5].

On the other side, TSR trend of the different composites is attributed to several factors. Firstly, TSR increases if the phases constitute the structure of the object has low thermal expansion coefficient [31]. Composite 0 YA has the lowest thermal expansion and addition of AlN lead to increasing the thermal expansion values as explained previously. This can be realized from the higher CTE of AlN ($\approx 4.5 \times 10^{-6} \text{ K}^{-1}$) when compared to SiC ($\approx 4 \times 10^{-6} \text{ K}^{-1}$) [32]. Therefore, presence of SiC and increasing its content contributed to the enhancement of the resistance to thermal shock. Secondly, the generation of fine intergranular cracks caused by anisotropic thermal expansion properties of the structure phases during the subsequent thermal shock cycles absorbs the strain energy during the cycles, which, consequently, enhances TSR. In addition, SiC ceramic itself has higher TSR than AlN. So that, increasing its content led to enhancing their resistance to thermal shock [33, 34].

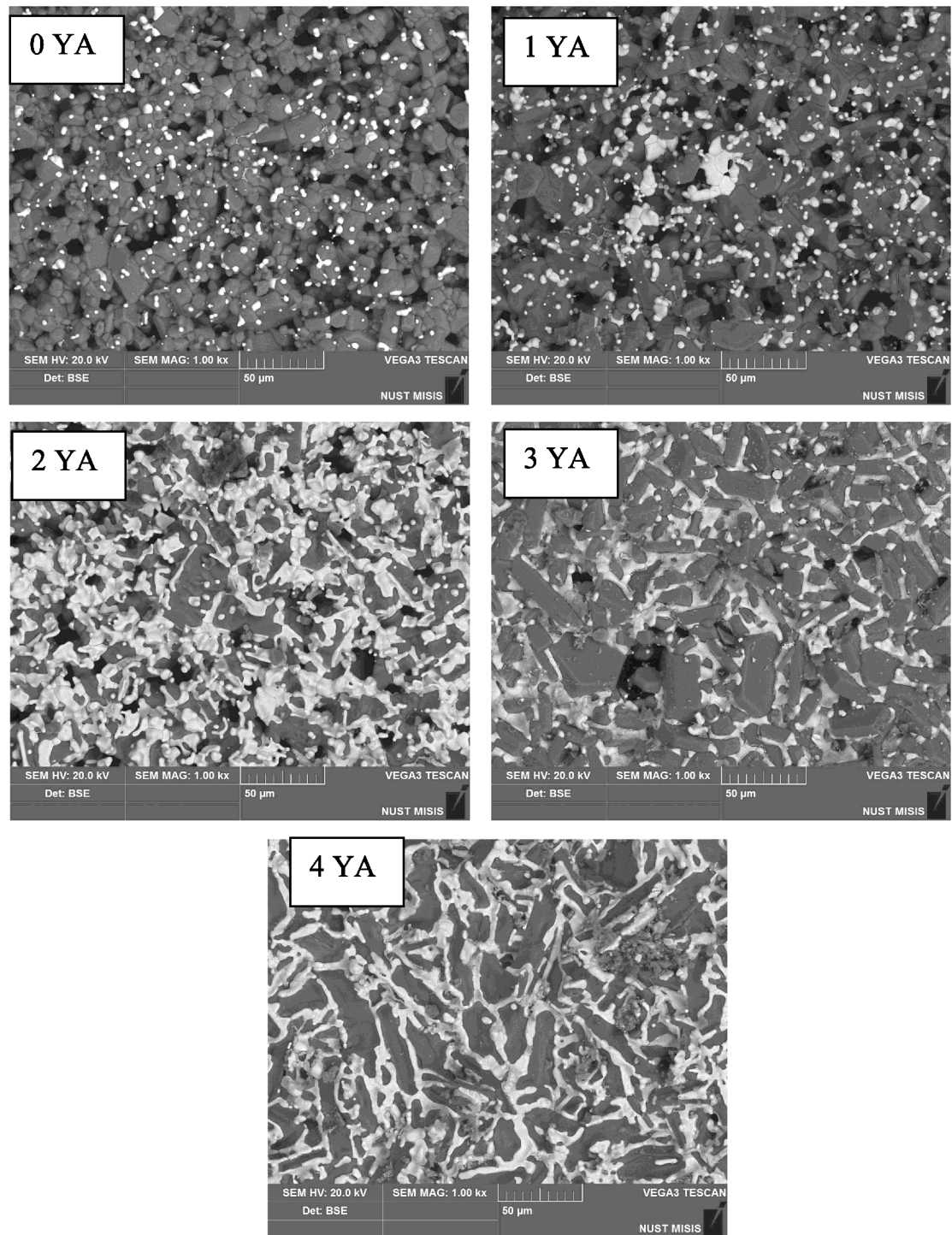


Figure 3. FE-SEM images of the different SiC/AlN composites subjected to thermal shock cycling.

Studying the microstructure of the different SiC/AlN composites before and after the thermal shock cycles and comparing their FE-SEM micrographs have revealed that: thermal shock treatment of different composites resulted in no change in the microstructure. Moreover, there was no any noticeable crack or fracture in any composite as illustrated in figure 3. This behavior can be explained by that, the stresses resulted from the thermal shock quenching are lower than those required for crack initiation and propagation in the sample. In addition, the high porosity percentage distributed in the different composites may play a role in inhibiting the crack formation. Finally, it can be deduced that, different properties of the pressureless sintered SiC/AlN composites can be controlled by controlling SiC and AlN contents. Besides, the investigated ceramic composites gave superior TSR accompanied with high mechanical strength and stable microstructure that strongly nominate them to be used in high temperature applications under severe conditions.

4. Conclusion

TSR of different pressureless sintered SiC/AlN composites has been investigated and evaluated. The different ceramic composites were capable of withstanding exposure to 20 thermal shock/water quenching cycles at temperature of 700 °C without destruction or failure. Investigation of compression strength of the inspected composites before and after shock treatment revealed that, their thermal shock behavior were belonged to regime I with retention of about 70% of their original strength. Composites with higher SiC and lower AlN contents gave the best thermal shock properties. Microstructure characterization of the tested composites did not show any change in the grains morphology or any fracture appearance. This behavior can be attributed to presence of porosity that results in cracks tip blunting besides the elongated grains morphology which lead to crack bridging and crack deflection. Accordingly, pressureless sintered SiC/AlN composites are strongly nominated to be used in applications under severe thermal properties environments.

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