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STRENGTH OF A CERAMIC AT HIGH TEMPERATURES UNDER DIAMETRAL COMPRESSION

J. E. O. Ovri[1]and T.J. Davies[2]

[1]Department of Materials and Metallurgical Engineering,
Federal University of Technology, Owerri,
NIGERIA.
jimvri@yahoo.com
[2]Manchester Materials Science Centre,
University of Manchester|UMIST
Grosvener Street, Manchester M1 7HS,
UK.

ABSTRACT

A novel design is presented for the high temperature compression testing of ceramic discs up to temperature of 1300°C. Two proprietary silicon nitrides containing magnesia (MgO) and a mixture of magnesia and yttria ($MgO + Y_2O_3$) were pressureless sintered and their tensile strengths measured at room temperature and elevated temperatures (900°C to 1270°C) using the diametral compression disc test.The results obtained showed that the material sintered with MgO maintained only 28% of its room temperature strength at 1270°C whilst the material sintered with a mixture of MgO and Y_2O_3 maintained 90% and 89% of its room temperature strength at 900°C and 1270°C respectively.Grain fracture/pull out were observed in some of the samples doped with Yttria at high temperature which gives room for speculation that both intergranular and transgranular fracture mechanisms were perhaps operative in this material.

Keywords:Disc test,Room temperature strength,Grain fracture/pull out,Doped,Intergranular/transgranular fracture

INTRODUCTION

Silicon nitride is used as a material for certain high temperature components of the gas turbine engine due to its high temperature strength retention and inertness under corrosive and degradativeenvironments[1-3].. Inherent problems, however, exist in consolidating this material to high density[4] whilst maintaining sufficiently high purity. Fabrication often presents difficulties which are thought[2] to be associated with the co-valent bonding and the lack of diffusive processes to provide material transport for densification; this has led to the use of various densification aids to remove porosity[1].

Nomenclatures	
^{o}C	Temperature in degree centigrade
MgO	Magnesia
Y_2O_3	Yttria
Si_3N_4	Silicon nitride
mm	Millimeters
//	Inches
Pt/Pt-13%Rh	Platinium and platinium plus 13%Rubium thermocouple
σ_c	Tensile strength
MPA	Megapascal
AV	Average
SD	Standard Deviation
CV	Coefficient of variation
SEM	Scanning electron microsope
µm	Micronmeters

These densification aids often give rise to mechanical strength degradation at elevated temperatures because they solidify as glasses on the grain boundaries after sintering.Magnesia (Mgo) is often used as a densifying aid but it is widely recognized that this material produces a nitrogen containing glassy grain boundary phase which limits the use(4) at temperatures greater than 1200^{oC}. Alternatively, yttria has been used to produce a more refractory grain boundary phase which promotes high temperature strengths. However, these refractory phases also appear to produce thermal instability in the material at intermediate temperatures (700 – 1100^{o}C) because the material exhibits an abnormal increase in thermal expension coefficient in this temperature range[4,5,6,7,8,9,10,11].

The work reported here is a part of a study on the fabrication and the mechanical strength of silicon nitride. In particular, the effect of sintering aid on the room and elevated temperatures of two proprietory silicon nitrides is investigated.

MATERIALS AND METHODS

Materials

Pressureless sintered silicon nitride (supplied by Turner and Newal Materials Research Limited, Trafford Park, Manchester) was used for the investigation. Two compositions were used: A, 95 wt.% Si_3N_4 + 5wt.% Mg0, B, 94wt.% Si_3N_4 + 1wt.% Mgo + 5wt.% Y_20_3.The chemical composition of the silicon nitride is given in [1,3]. The dimensions of samples were nominally 20.454mm x 4.009mm.

Mechanical Test Procedures

The room and elevated temperature compression disc tests were carried out in air using an instron testing machine (model TT-DML – A06 – 75) at a constant displacement rate of 0.2mm/min. Friction was assumed to be negligible in all cases[3].

High temperature test rig

The high-temperature test rig, designed to operate at temperatures up to 1300^{o}C in air, was fabricated from a commercial alumina. The test fixture consisted of an alumina loading ram and a refractory brick base support (78.2 x 76.2mm). The loading ram was held in position with 50.8mm (2") mild steel to the Instron testing machine.

The fixture was designed in such a way that it ensured that the ram and base support would be axially aligned during loading.

The whole setup was assembled into a square section furnace (460mm x 460mm) at room temperature. The furnace was electrically heated using a silicon carbide element. Silicon carbide plates were cemented onto the surface of both the ram and the support base. The silicon carbide was preferred to alumina for base facing because it was not susceptible to failure by thermal shock. The test rig is shown in Figure 1.

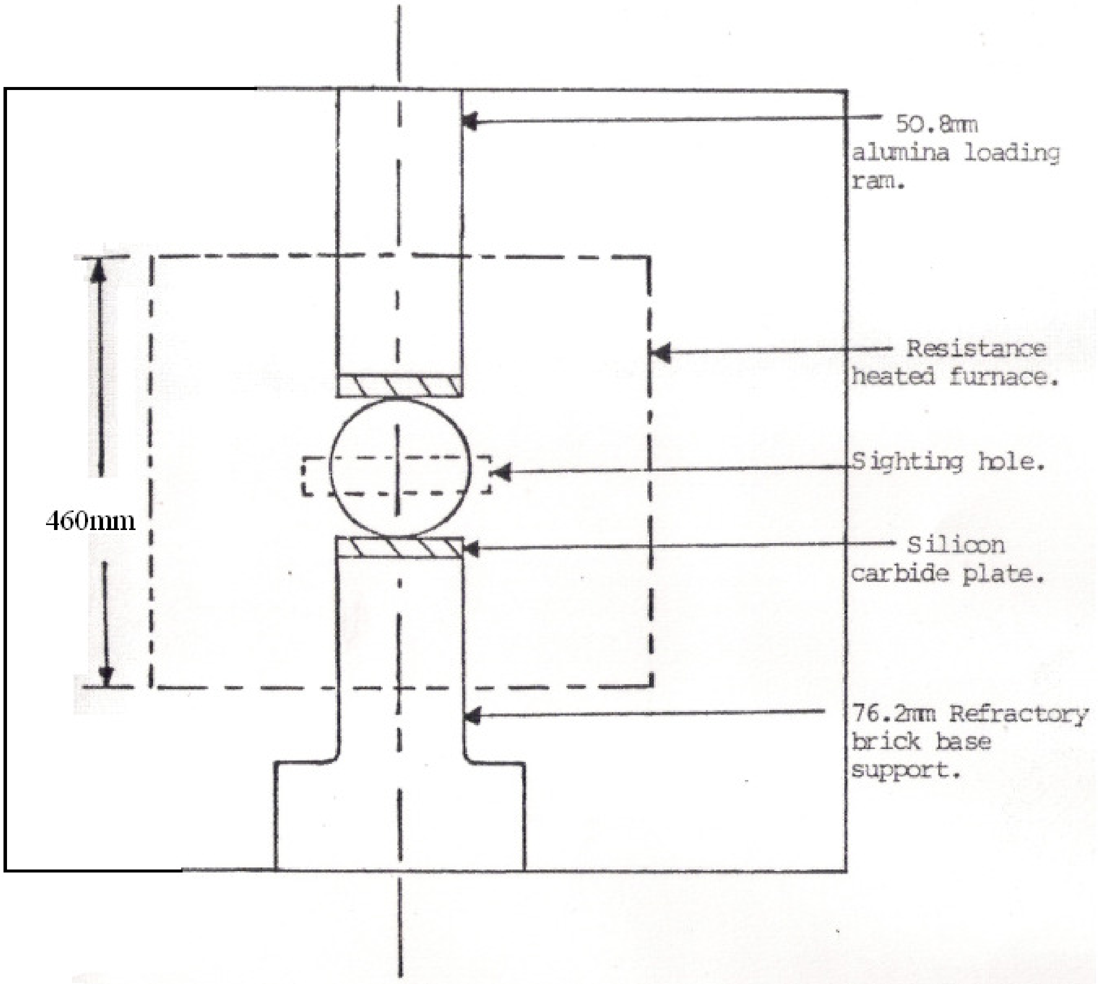

Figure 1. High Temperature Test Rig

High temperature test

The specimens were placed on the test fixture in the furnace at room temperature and the furnace was switched on. The tests were conducted at 900 ± 10°C and 1270 ± 10°C after holding at the operating test temperatures for about 30 minutes. The test temperature was determined using a Pt/Pt – 13%Rh thermocouple placed just beneath and within 3mm of the centre of the specimen. Five samples were tested for each material.

RESULTS AND DISCUSSION

Effect of Temperature On The Strength Of Silicon Nitride

The results on the effect of temperature on the strength of pressureless sintered silicon nitride are shown in Table 1 and displayed in Figure 2.

Table 1.Room and High Temperatures Strengths for Pressureless Sintered Silicon Nitride Using the Diametral Disc Test

Material	**Temperature of Test (°C± 20°C)**		
	Room (20°C) **σ_c (MPa)**	**900** **σ_c(Mpa)**	**1270** **σ_c(Mpa)**
	111.25		29.22
	115.95		30.70
A	119.28		
	128.25	-	31.93
	131.12		38.68
	121.17		40.38
Average (AV)	±7.46		34.18
Standard deviation(SD) Coefficient of variation(CV) (%)	6.2		4.43
			13.0
	121.11	107.23	106.54
	122.35	107.15	108.98
B	125.35	112.38	110.18
	126.65	113.63	111.83
	127.68	118.58	118.95
Average(AV)	124.63	111.79	111.30
Standard Deviation(SD) Coefficient of Variation(CV) (%)	±2.51	±4.28	±4.76
	2.0	3.8	4.3

Leena and Luna International,**Oyama, Japan.**

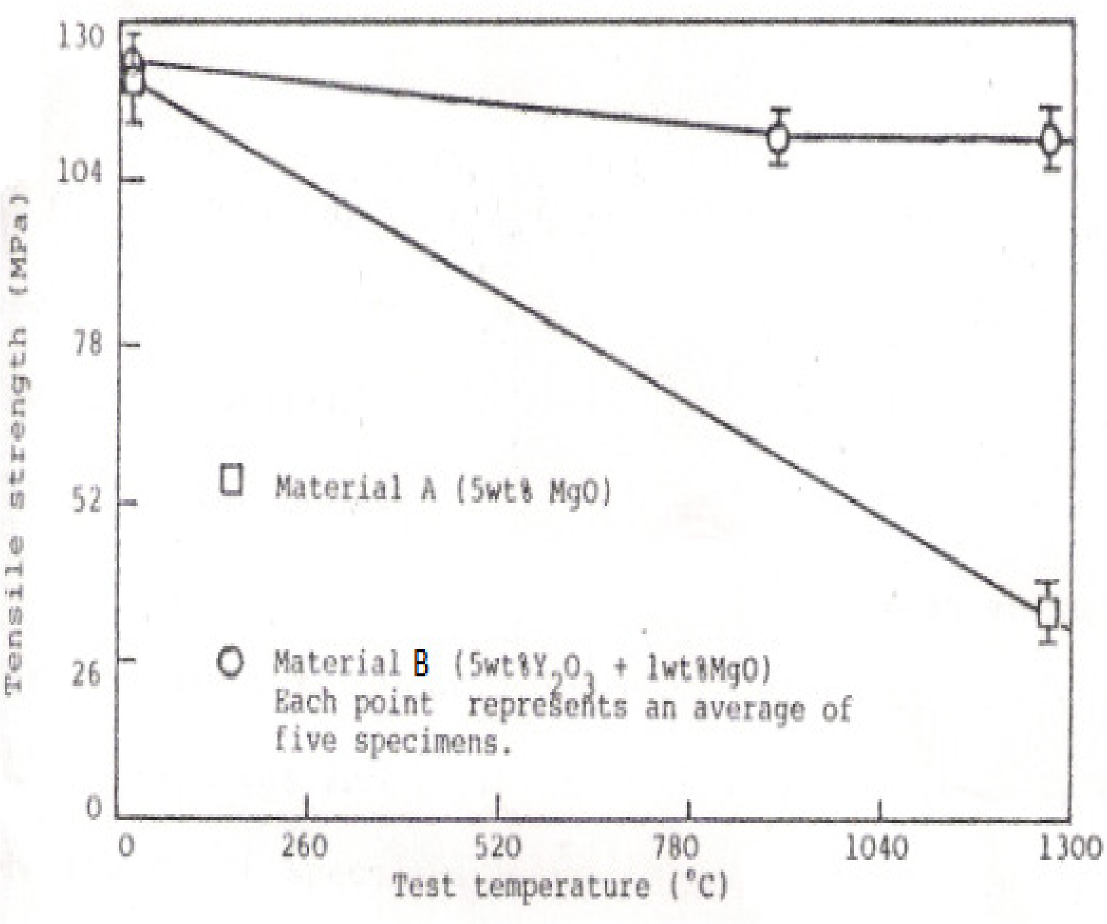

Figure 2.Effect of Temperature on the Tensile Strength of Pressureless Sintered Silicon Nitride Using the Diametral Disc Test

The arithmetic mean of the room and elevated temperature strengths listed in Table 1, showed that material A has a mean room temperature strength of 121.17MPa and a standard deviation of ± 7.46MPa and co-efficient of variation of 6.3% whilst the mean elevated temperature strength at 1270°C is 34.18 MPa, standard deviation of 4.43 MPa and a coefficient of variation of 13%. Material B, the mean room temperature strength is 124.63 MPa, standard deviation of ± 2.51 MPa and coefficient of variation of 2%. The elevated temperatures strengths were as follows: Mean strength is 111.79 MPa, standard deviation is ± 4.28 and coefficient of variation is 3.8% for 900°C and for 1270°C, the mean strength is 111.30 MPa, standard deviation is ± 4.76 MPa and coefficient of variation is 4.3%.

It is reported in the literature[12,13,14] that magnesia doped silicon nitride suffers from high temperature strength degradation at about 1200°C partly due to the formation of a nitrogen silicate glass during densification. This grain boundary phase is believed to soften at about 1200°C and hence the use of these materials is limited to 1200°C. Yttria doped materials are reported to possess higher temperature strengths partly due to the more refractory grain boundary phase formed during densification, [5,15] these materials are however, believed to suffer from intermediate temperature strength degradation due to chemical instability (700 – 1100°C) [16,17].

To investigate the synergistic action of yttria and magnesia as densification aids, two materials were chosen, material A (5wt% MgO) and (1wt.% MgO + 5wt.% Y_2O_3) material B. The MgO in material B is expected to provide its good sinterability whilst the yttria is expected to provide a refractory phase. The results shown in Table I and displayed in Figure 2 show that the material sintered with 5wt.%MgO suffered a drastic strength degradation at 1270°C (only maintained about 28% of its room temperature strength). The implication of this experimental observation is that the use of this material from mechanical strength view point is limited to about 1200°C which gives some experimental backings to previous reports [12,13,14,16,17] on the high temperature mechanical strength capability of magnesia doped silicon nitride.

The yttria and magnesia doped material maintained its mechanical relative strengths throughout the range of the temperature of the test. It maintained 90% of its room temperature strength at 900°C and only dropped to 89% at 1270°C. No drastic strength degradation was observed at 900°C contrary to previously held views[12,13,17].The synergy of the mixture of yttria and magnesia as densification aids in the enhancement of the high temperature strength of pressureless sintered silicon nitide in this study is of great interest and a paper on the high temperature strength of yttria doped pressureless sintered silicon nitride will soon be submitted for possible publication.It is only then that the synergistic action of the mixture of yttria and magnesia can be evaluated qualitatively. However, the results are in qualitative agreement with delayed fracture strengths for hot-pressed yttria doped silicon nitride for the same temperature range reported by Weiderhorn and Tighe[18].It is suggested however,that further work especially longer exposure time before strength testing, before a firm conclusion can be drawn on the behaviour of this material at intermediate temperatures.

The spread of the strength data which is indicated by the standard deviation and coefficient of variation are larger for the materials tested at the elevated temperatures. For the two materials, the coefficient at the elevated temperatures is almost twice of those at room temperatures. This experimental observation implies that more severe flaws were encountered at elevated temperatures.

Failures at elevated temperatures are affected by various processes[19,20] such as:

a. nucleation and growth of cavities and crack,
b. linking up of cavities to form bigger cracks and the growth of these cracks (or pre-existing flaws), to form critical-size flaws for spontaneous failures, and,
c. crack healing and blunting.

It is seen from the fractographs of some of the specimen at elevated temperatures that nucleation and growth of cavities was probably one of the fracture processes that occurred; the pores were generally larger and less regular in shape and disposition at high temperatures (plate 2). The change in the coefficient of variation is believed to be due partly to the change in size and shape of the pores at elevated temperatures.

Fracture Modes/Fractographs

Fracture modes

Specimen were visually examined to try and identify the types of fracture. In general, in specimen where valid tensile fractures occurred, the triple-cleft fracture pattern occurred more frequently in all materials both at the room and elevated temperatures. Typical room and elevated temperatures fracture modes are shown in plate 1.

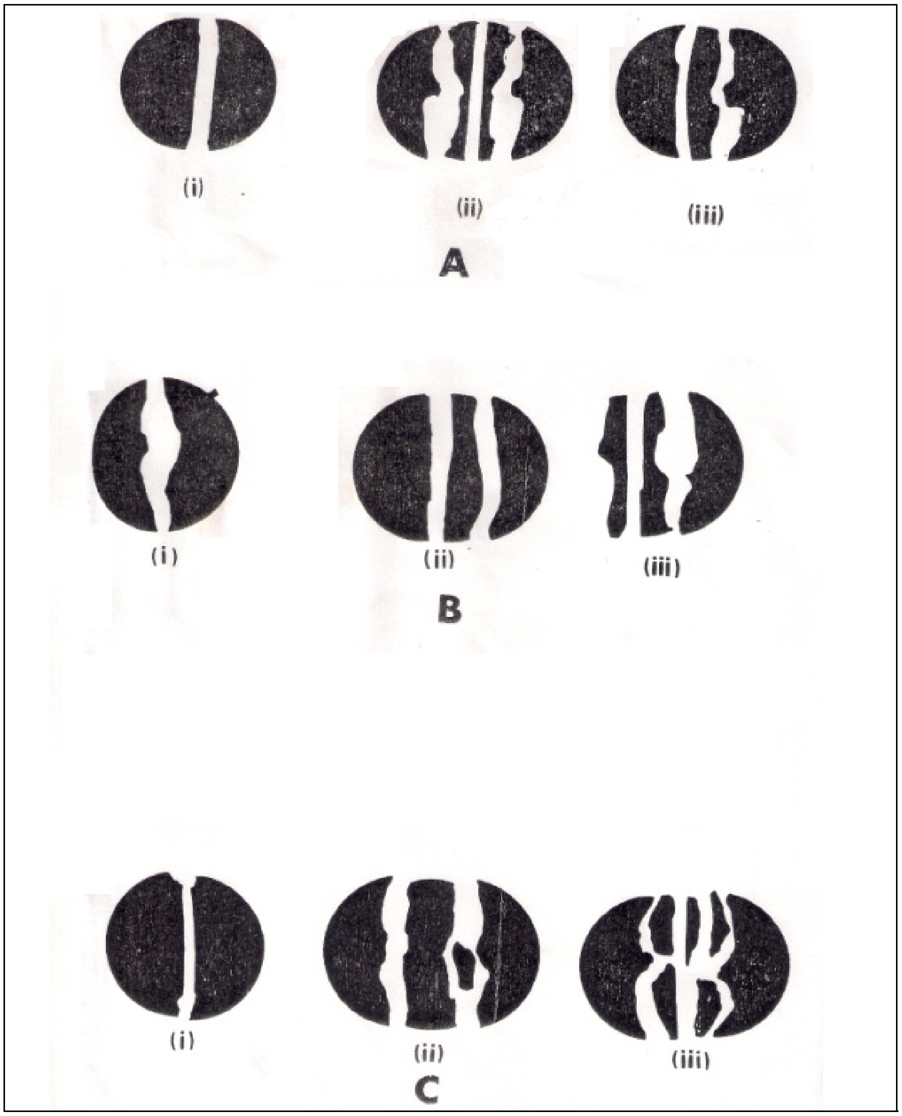

Plate 1: Typical Room and Elevated Temperatures Fracture Modes for Pressure lessSintered Silicon Nitride

Plate 1A gives the room temperature fracture modes for the silicon nitride. A(i) shows a normal tensile fracture behaviour with a single fracture plane parallel to the loading direction whilst a triple-cleft fracture is shown in 1A(ii), the specimen fractures into four pieces with fracture consisting of a central normal plane fracture (as in A(i) above) and two nominally co-linear fracture on either side of the central fracture; these two fractures have a tongue-and-groove profile. 1A(iii) shows a specimen that fractured in a triple-cleft mode without a central fracture plane but containing two smaller fractures displaced nominally equidistant from the central axis.

It is evident that the left side fracture is of a central crack type A(i) and the right side fracture a tongue-and-groove type A(ii).

1B and C gives the elevated temperatures fracture modes for the two materials. B(i) is a type A(i) fracture with small deflection from the vertical plane similar to A(iii). B(ii) fracture with the two fractured portions displaced symmetrically about the central vertical plane.

C(i) is a type A(i) fracture. C(ii) is a type A(ii) fracture but where the central crack has not propagated to fracture. C(iii) is a type A(ii) fracture with the two centrally displaced pieces also fractured in the horizontal plane at mid-section. It is seen from the foregoing that the fracture modes for silicon nitride is independent of temperature.

Fractography

Fractographic examinations of some of the test specimens were made using the Scanning Electron Microscope (SEM). Fracture origins were usually at some interior positions, nearly always at the centre of the discs and never at the loading arcs. Examinations of some of the fractured specimens indicated that fractures were caused by sub-surface and internal defects. Plate 2A shows a room temperature fracture surface with pores of comparable size with the grain. 2B shows a nominally planar fracture surface for material B with some mode of fracture occurring over the fracture surface; no obvious effect of grain pull out, porosity or inclusion on the mode of fracture.

Failures at elevated temperatures were associated with pores. Plate 2C shows some fracture surfaces of some of the high temperature specimens. It shows general fracture surfaces with pores larger than the grains of the specimens, these pores were generally larger than those observed at room temperature for the same material (compare plate 2A). Plate 2D shows a high temperature fracture specimen (material B), there is evidence of grain pull out/fracture on this specimen.

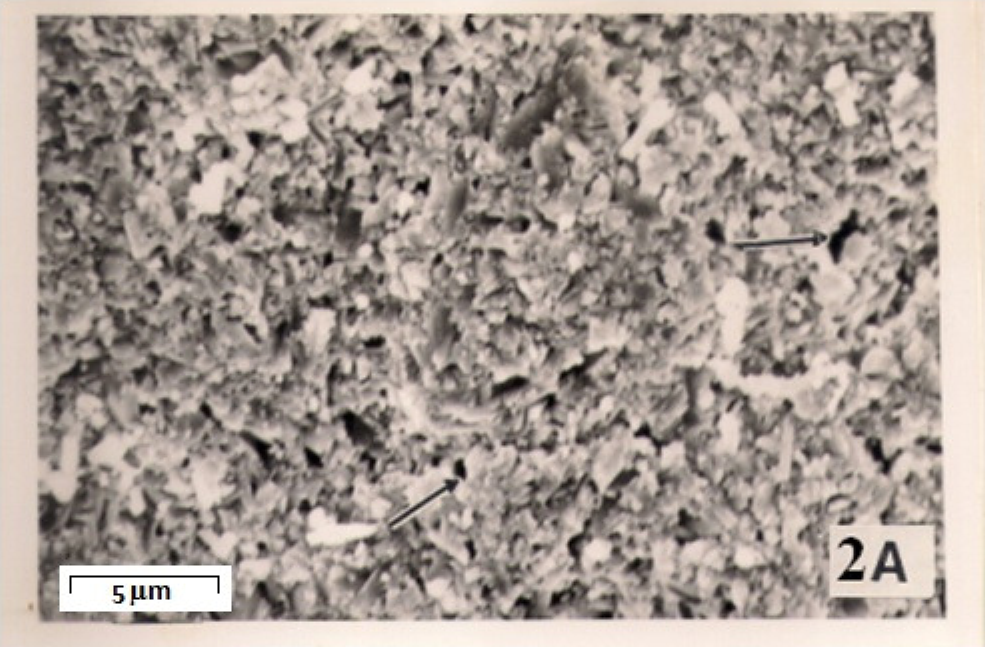

Plate 2A. SEM Room Temperature Fractograph

Showing pores on fracture surface (arrowed) of size comparable with the grain size. Pores were generally less than 1µm in average diameter.

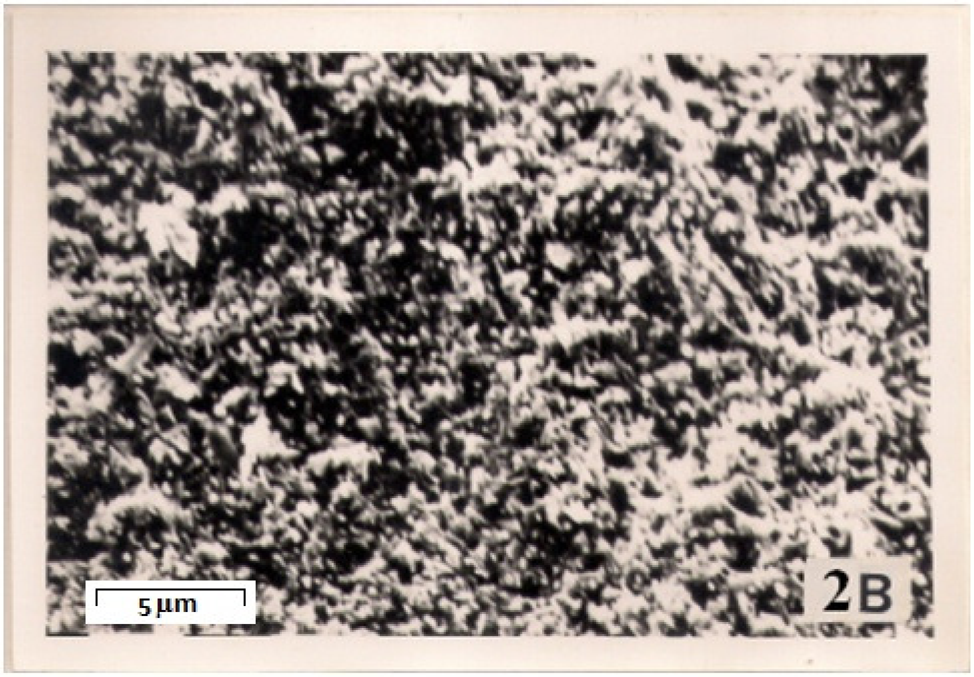

Plate 2B.SEM Room Temperature Fractograph

Nominally planar surface with a single mode of fracture occurring over the entire fracture surface: no obvious effect of grain pull out, porosity or inclusion on the mode of fracture.

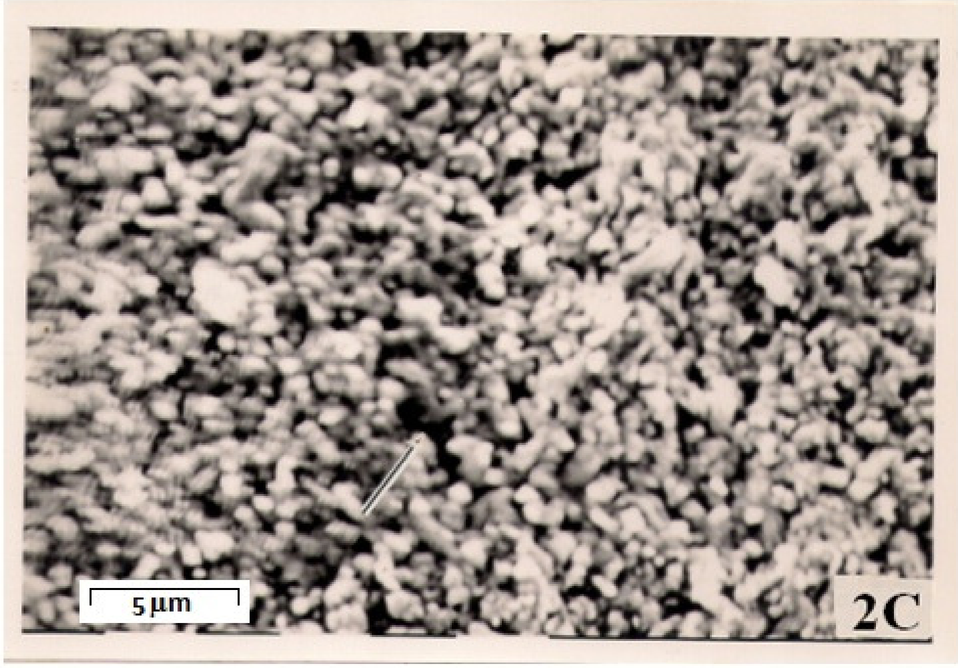

Plate 2C.SEM Elevated Temperature Fractograph

Showing general fracture surface with enlarged irregular pores (arrowed).These pores are generally larger than those observed at room temperature for the same material (compare plate 2A). Most of these pores were less than 1µm in length although a few were in the range 1µm to 5µm in length.

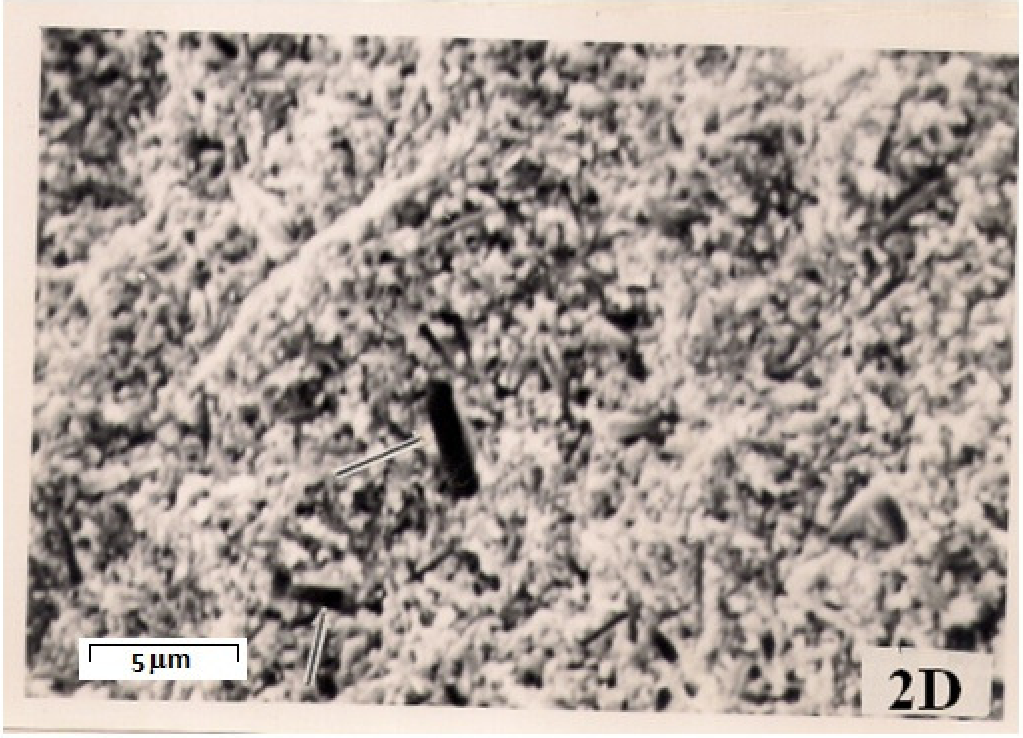

Plate 2D.SEM Elevated Temperature Fractograph

General fracture surface showing suspected grain fracture/pull out (arrowed); this was not observed with the room temperature specimens (compare plate 2B). grains were found to be in the range 1µm to 5µm in average diameter

CONCULSION

The following conclusions could be made from this study:

1. Yttria doped silicon nitride retained about 90% of its room temperature tensile strength at 900^{o}C and 89% at 1270^{o}C; the magnesia doped silicon nitride had only 28% of its room temperature tensile strength.
2. The fracture modes of room and elevated temperatures for silicon nitride doped with yttria and magnesia are similar.
3. Grain fracture/pull out were observed in some of the silicon nitride tested at elevated temperatures, this must have been responsible for the maintenance of the high temperature strength of these materials
4. Fracture of silicon nitride at elevated temperatures was found to be associated with pores, these pores were larger than those at room temperature.
5. Higher scatters in strength data were observed for silicon nitride tested at elevated temperatures (the scatter were almost twice the values of those obtained at room temperature).

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