COMPRESSIVE CREEP BEHAVIOR OF HOT-PRESSED Si_3N_4 CERAMICS

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Abstract. Silicon nitride ceramics (Si_3N_4) is a ceramic widely used in structural applications due to its physical and mechanical properties, such as high wear, hardness and creep resistance. These materials possess a great application field in engines and turbines components. In this work, a natural yttrium and rare earth oxide solid solution, CRE_2O_3, produced at FAENQUIL-DEMAR, at a cost of only 20% of pure commercial Y_2O_3, was used as sintering additive of hot-pressed Si_3N_4 ceramics. The objective of this work was to investigate the compressive creep behavior of these ceramics. Compressive creep tests were carried out in air, between 1250 and 1300^0C, for 60 hours, under stresses of 200 to 300 MPa. The stress exponent under all conditions was determined to be about unity. The apparent-activation-energy obtained, was on around 500 $kJmol^{-1}$, corresponding to the heat of solution of the Si_3N_4 in the glassy phase. Both the stress exponent n and the apparent activation energy, Q, are within the range of values obtained in other studies of the compressive creep of Y_2O_3-Al_2O_3-doped-Si_3N_4 ceramics. These results indicated that grain-boundary sliding controlled by viscous flow can be ruled out in the present study. The creep behavior indicates that the additive can substitute pure Y_2O_3 in the fabrication of Si_3N_4 ceramic materials, resulting in equivalent properties at room and high temperatures.

Key Words: Silicon nitride, hot pressing, creep mechanisms, CRE_2O_3,, rare earth oxides, compressive creep.

1. Introduction

Silicon nitride (Si_3N_4) is a ceramic widely used for structural applications due to its physical and mechanical properties, such as high wear, hardness and creep resistance (Bellosi, 1999, Komeya, 1988, Riley, 2000). The liquid phase sintering of Si_3N_4 is governed by a solution-precipitation ($\alpha-\beta-Si_3N_4$ transformation) mechanism, that occurs in temperatures between 1400 and 1800 ^0C and is related, among other factors, to the initial $\alpha-Si_3N_4$ phase content of the starting powder, amount of additives used and sintering parameters such as temperature, time and atmosphere (German, 1985, Riley, 2000, Lai, 1993, Komeya, 1988). The microstructural control is the preponderant factor for successful structural applications. In special the grain morphology of β-Si_3N_4 grains is partially responsible for the high creep resistance of these materials (Cannon, 1983, Whiederhorn, 1999, Shibuya, 1998).

Due to their characteristcs previously mentioned, the technological applications of silicon nitride ceramics (Si_3N_4) are numerous, and in this context, it is of primary interest the study and understanding of the mechanical properties of these materials at high temperatures. It has been also demonstrated that the secondary glassy phases wich appear during the sintering have a strong influence in the creep behavior.

Hot-pressed silicon nitride is generally fabricated using the oxide additives to enhance the densification. However, the remaining glassy phase softens at high temperature and gives a deleterious effect on the high temperature strength. The creep deformation of materials containing intergranular glassy phase is generally through to occur in combination of viscous flow, solution-precipitation and cavitation mechanisms (Evans, 1993, Silva, 1989, Silva, 1997). Creep deformation of Si_3N_4 could not be directly attributed to any single factor of content and composition of additive, grain size , etc, since the grain size is also dependent on the composition and amount of sintering additives. Thus, the creep behavior of silicon nitride ceramics must consider many factors, such as the amount and composition of glassy phase, the size and distributions of the matrix silicon nitride grains, the partial recrystallization of grain boundary glassy phase and the oxidation (Yoon, 1997, Barsoum, 1997).

Previous works have shown that the rare earth oxide mixture, CRE_2O_3, consisting mainly of Y_2O_3 (44%), Yb_2O_3 (17%), Er_2O_3 (14%) and Dy_2O_3 (10%) produced at DEMAR-FAENQUIL (Ribeiro, 1997), is an effective and cheap substitute for pure Y_2O_3 as sintering additive for Si_3N_4 ceramics, presenting an obtaining cost 80% lower that Y_2O_3 (Ribeiro, 1997), a similar sinterability (Strecker, 2000, Ribeiro, 1998, Ribeiro–2, 1997) and similar mechanical properties at room temperature (Santos, 2003, Strecker, 2000, Ribeiro, 1997, Ribeiro, 1998, Ribeiro–2, 1997).

The objective of the present work has been to characterize the creep behavior of the hot-pressed Si_3N_4 ceramic produced with Al_2O_3 and CRE_2O_3 as sintering additives, demonstrating the potential of this sintering additive in the high performance ceramics production.

2. Experimental Procedure

The powder batches prepared consisted of a mixture of 95 vol.% of commercial $\alpha\text{–}Si_3N_4$ (E10, UBE Industries–Japan, containing also 5wt.% $\beta\text{–}Si_3N_4$), and 5 vol.% of additives, Al_2O_3 (CR6, Baikalox-Germany) and CRE_2O_3 (FAENQUIL-Brazil), at a molar ratio of 0.6:0.4, respectively, in order to form $Y_3Al_5O_{12}$ as intergranular phase after sintering. The powders were mixed by ball milling during 2 h using ethanol as milling media. After mixing, the powder batches were dried first in a rotary evaporator and subsequently in an oven at 120 ^{0}C during 12 h. The powder mixtures were then compacted isostatically under a pressure of 300 MPa and sintered by uniaxial hot-pressing under 20 MPa pressure in 0.1 MPa N_2 atmosphere at 1750 ^{0}C for 30 minutes, with a heating rate of 15 ^{0}C/minute. From the as received materials, parallelepipedical samples of aproximate dimensions 6 x 3 x 3 mm^3 were cutted and gruonded for tests.

The samples were characterized by relative density using the immersion method. Phase analysis was done by X-ray diffraction (XRD), in both samples before and after creep tests comparing the diffraction patterns with the JCPDS files. XRD was conducted on a plane parallel and perpendicular to the hot pressing direction in a bulk specimen. In the XRD, the ratio of β-phase (101)/(210) peak area was used as an indication of preferred orientation degree of elongated grains (Lee, 1992, Yoon, 1997).

For microstructural analysis, the samples were grinded, polished and chemically etched by a 1:1 mixture of NaOH and KOH at 500 ^{0}C for 4 minutos. The grains size distributions and aspect ratios were measured using the method proposed by Wötting et *al* (Wötting, 1986); around 1200 grains were analyzed per sample and the aspect ratio was determined as the average of the 10% of largest grains found in the sample. A software was used for image analysis, in order to obtain the average width, length and aspect ratio of the grains.

The hardness was determined by Vicker´s indentations for 30 s under an applied load of 2 kg. For statistical reasons, 20 indentations have been made per sample. The fracture toughness has been determined by the measurement of the crack length created by the Vicker´s indentations. The calculation of the fracture toughness was done by the relationship proposed by Evans *et al.* (Evans, 1976), valid for Palmqvist type cracks.

Compressive creep tests were carried in air, at temperatures ranging between 1250 and 1300^{0}C and under nominal stresses of 200, 250 and 300 MPa. The details of the experimental device can be found elsewhere (Santos-2, 2003). *Strain x time* curves were obtained and the results are analysed in terms of the creep equation as shown in Eq. (1):

$$\varepsilon_s = A\sigma^n \exp(-Q/RT) \qquad (1)$$

Where ε_s is the true steady-state strain rate, A is a constant, σ is the applied stress, T the absolute temperature (in Kelvin) and R the gas constant, n and Q are, respectively, the stress exponent and the apparent activation energy for creep. The results were performed: (1) at constant temperature and different stress, in order to evaluated the stress exponent *n;* (2) at constant load and different temperatures, to evaluate the apparent activation energy *Q*. All tests were finished after 60 hours, without evidence of macroscopic failure.

3. Results and Discussion

3.1. Specimens Characterization

In Tab. (1) the relative density and crystalline phases, as well as the results of the quantitative Si_3N_4 phase analysis, hardness and fracture toughness are listed.

Table 1. Sintering results of the $\beta\text{-}Si_3N_4$ specimens before creep tests.

Sintering Conditions	**Relative density** (%)	**Crystalline phases**	[β/β+α] (%)	**Hardness** (HV)-GPa	**Fracture Toughness** K_{IC} – MPa.m$^{-1/2}$
Hot-pressing, 1750 ^{0}C/0.5h 20 MPa	98.6 ± 0.2	$\beta\text{-}Si_3N_4$	100	16.4 ± 0.3	5.9 ± 0.2

The relative densities of the specimens after hot-pressing presented values higher than 98% for this composition, demostrating that dense ceramics were obtained. Phase analysis by X-ray diffraction (XRD) revealed only the presence of $\beta-Si_3N_4$ indicating that the $\alpha-$ to $\beta-Si_3N_4$ transformation has been completed and, furthermore, that the additives formed an amorphous intergranular phase.

The specimens investigated yielded ceramic materials of high hardness, more than 16 GPa. This result is directly related to factors such as grain size, grain morphology, $\beta-Si_3N_4$ content, reduced additive content, low porosity, among others (Peillon, 2002, Mukhopadhyay, 1999, Perera, 2000, Herrmann, 2001). The fracture toughness values are near to 6 $MPa.m^{1/2}$. This results of fracture toughness can be attributed to several factors such as the high relative density, the complete α to $\beta-Si_3N_4$ transformation, but mainly due to microstructural aspects. The reduction of porosity influenced the fracture toughness due to the reduction of the amount and size of defects.

3.1.1. Microstructure

During liquid-phase sintering of Si_3N_4 ceramics, $\alpha-Si_3N_4$ particles dissolve in the liquid phase and precipitate in the form of elongated, hexagonal $\beta-Si_3N_4$ grains. The morphology (aspect ratio) depends to a large degree on the additives used and their quantity and sintering conditions. The results of the microstructural analysis of the hot-pressed samples are resumed in Tab. (2); listing the average grain sizes, the planar grain density (defined as number of grains per area), and the aspect ratio of the $\beta-Si_3N_4$ grains. Figure 1 shows the SEM micrographs of the sintered specimens.

Table 2. Microstructural characteristics of hot-pressed specimens.

Sintering Conditions	Average grain size (µm)	Planar grains density [N^0 grain/ μm^2]	Aspect Ratio
Hot-pressing, 1750 ^{0}C – 0.5h 20 MPa	2.5 ± 0.8	1.28	6.5 ± 1.1

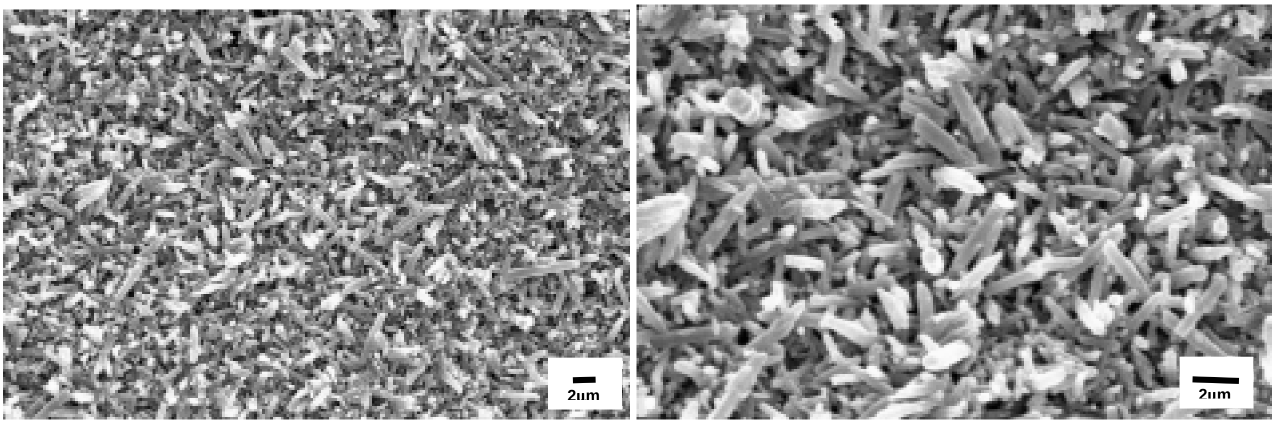

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Figure 1. Microstructure of the hot-pressed creep specimens.

The wettable liquid phase, in association with the isothermal holding time and sintering pressure used, influenced in obtaining of dense ceramics with elongated $\beta-Si_3N_4$ grains (2.5µm) with high aspect ratio (higher 6), in an complete $\alpha-\beta$ transformation and a rigid and intergranular phase resistant to crack propagation (Peillon, 2002, Mukhopadhyay, 1999, Perera, 2000). The high fracture toughness of the sintered samples with longer isothermal sinter times is explained by the microstruture consisting of elongated grains with high aspect ratio, which act as crack propagation barriers, increasing the energy consumed during crack growth.

It is well known that the anisotropic growth of $\beta-$ Si_3N_4 grains is related to the different growth velocities of the basal plane and the lateral planes of the hexagonal $\beta-$ Si_3N_4 prism, due to the different specific surface energies of each plane (Björklund, 1997, Perera, 2000, Herrmann, 2001). During hot-pressing, this effect of preferential grain growth is further enhanced by the applied pressure (Coble, 1970, Pyzik, 1993, Yoon, 1997, Lee, 1992), resulting in grains of even higher aspect ratios, as shown in Tab. (2). It can be observed that the aspect ratio of the $\beta-Si_3N_4$ was 6.5. This result is explained by the higher grain growth velocity of the $\beta-Si_3N_4$ grains along their c-axis, when compared to the a and b axis, consistent with the work of Lee et al (Lee, 1992).

The alignment of the $\beta-Si_3N_4$ grains during hot-pressing is known to be the result of pressure gradients in planes parallel and perpendicular to the hot-pressing axis. Therefore, the grains grow preferably perpendicular to the hot-pressing direction. This information is very important for the subsequent creep behavior discussion.

3.2. Creep Behavior

Previous works show that in Si_3N_4 ceramics, creep occurs by viscous flow, with grain-boundary sliding, solution precipitation and/or cavitation mechanisms (Yoon, 1996, Yoon-2, 1997, Crampon, 1997, Wiederhorn, 1999, Arellano-Lopéz, 2002, Silva, 1989). Despite of the form of the creep test, by tensile or compresive stress, the different mechanisms act. For much of the range of applied stress, creep occurs faster in tension than compression tests, and cavitation in compression is much less than in tension (Wiederhorn, 1999).

Compressive creep curves at 1250^0C (1523 K), 1275^0C (1548 K) and 1300^0C (1623 K) under stresses of 200 ~300 MPa are shown in Fig. (2). The curves exhibits the secondary creep after around 15 h depends on applied stress. No tertiary stage was detected in this tests after 60 hours.

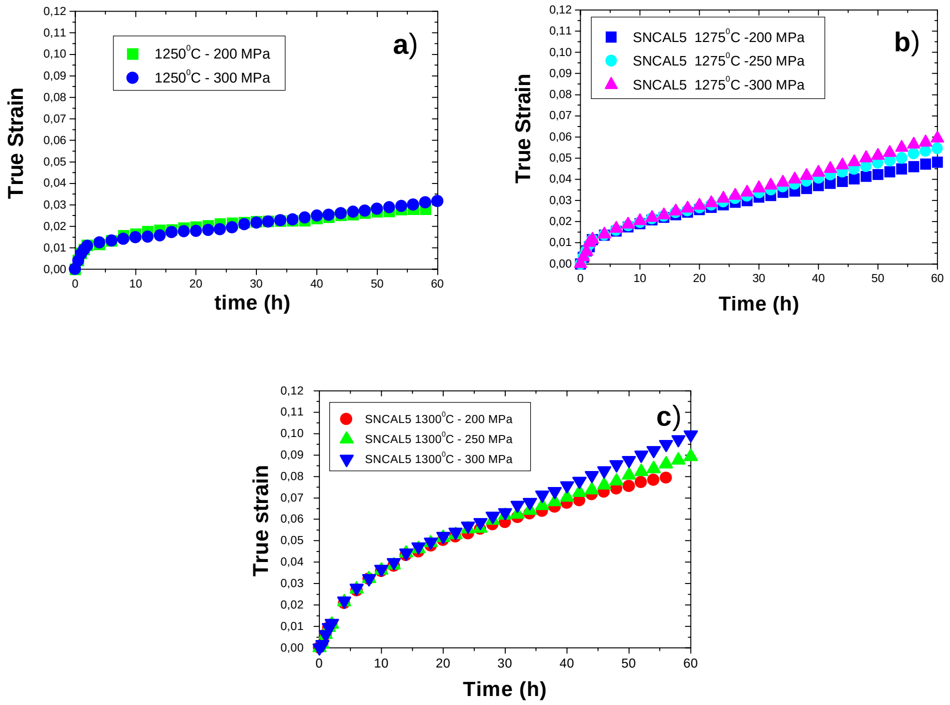


Figure 2. Creep curves of the hot-pressed silicon nitride specimens:a) at 1250^0C; b) at 1275^0 C; c) at 1300^0C (200 ~300MPa).

3.2.1. Specimens aspects

Figure 3 shows the specimens before and after creep tests. At the end of tests, the specimens showed an aspect of barreling. It is indicative that in central regions of the samples, the specimens were submitted to tensile stress in the perpendiculad plane to the load application axis during the creep tests. Besides, a reduction in the heigth is verified in the crept specimens. An uniform oxidized surface also was observed.

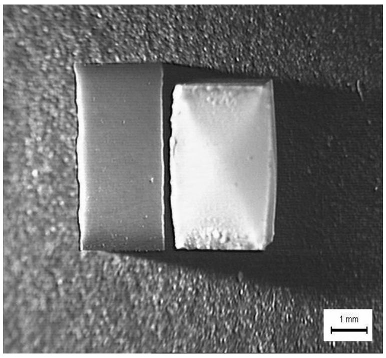


Figure 3. External surface morphology of specimens: (left) – before creep tests; and (right) a specimen crept under a stress of 300 MPa at 1300 ^{0}C (1573 K) characterized by a mixed tension and compression region.

3.2.2. Creep Mechanism

a) Stress Exponent (n)*-*

The dependency of the applied stress on the creep rate is summarized in Fig. (4). This Figure is a log-log plot of the compressive creep rates of Si_3N_4 ceramics as a function of applied stress.

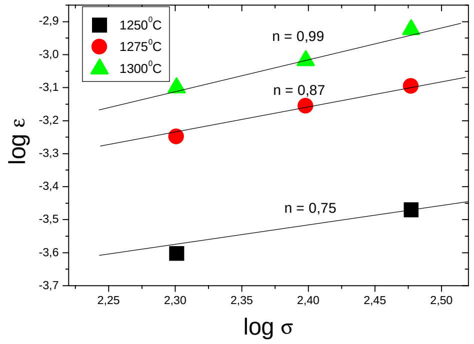


Figure 4. Stress dependencies of creep rate for hot-pressed CRE_2O_3/Al_2O_3-doped- Si_3N_4 ceramic, under 1250^{0}C (1523K), 1275^{0}C (1548 K) and 1300^{0}C (1573 K).

The value of the stress exponent, *n*, is determined to be 0.8~0.9 under a stress range of 200 to 300 MPa, independently of the temperature (1250 ^{0}C to 1300 ^{0}C). Lange *et al* (Lange, 1980) reported that Si_3N_4 material with small amount of the glassy phase showed a stress exponent, *n* , to be unity corresponding to the diffusional creep. As a result of the degradation of mechanical properties at high temperatures in majoritary Si_3N_4 ceramics, substantial investigations have been directed at the analysis of the creep behavior, in terms of diffusional mechanisms related to grain-boundary sliding along the glassy phase and cavitation (Yoon, 1996, Yoon-2, 1997, Crampon, 1997, Wiederhorn, 1999, Arellano-Lopéz, 2002, Silva, 1989). In the presence of a residual glassy phase, grain boundary sliding can be diffusionally accomodated either by viscous flow of the glassy phase or by the dissolution-diffusion-precipitation of material, and a stress exponent of *n* = 1 is found for pour Newtonian flow. Grain-boundary sliding controlled by viscous flow can be ruled out in the present study because it is revelead that the stress exponent of all conditions is determined to be about unity. Thus, the creep deformation is due to the diffiusional mechanisms.

b) Apparent activation energy (*Q*):

From previous studies on the creep deformation of Y_2O_3/Al_2O_3-doped Si_3N_4, in the domain where accommodated grain-boundary sliding exerts the major control of the creep rate, the solution-diffusion-precipitation on mechanism must be controlled by the diffusion of matter through the glass rather then by the transfer of atoms across the glass/crystal interface (Crampon, 1997, Yoon, 1997). In that case the activation energy for the mixed-control mechanism occuring during compressive creep failure remains consistent with that of the solution-diffusion-precipitation. The apparent activation energy for creep deformation determined from the creep rate under 200 to 300 MPa is obtained to be aruond 500 kJmol^{-1}, as shown in Fig. (5). This value corresponds to the heat of solution of the Si_3N_4 in glassy phase, wich was measured from the densification experiments (Crampon, 1997)Yoon, 1997).

The present experimental activation energy value is similar to others values of *Q* reported in the creep of Y_2O_3-Al_2O_3-doped-Si_3N_4 materials (Crampon, 1997, Silva, 1989, Silva, 1997, Yoon, 1997, Shibuya, 1998), for a grain-boundary sliding mechanism accomodated by a solution-diffusion-precipitation process, with the viscosity of the glassy phase as the rate-controlling step.

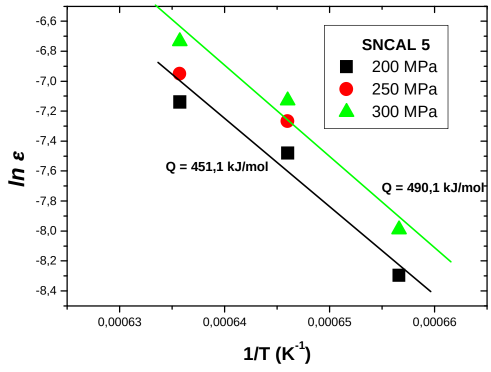
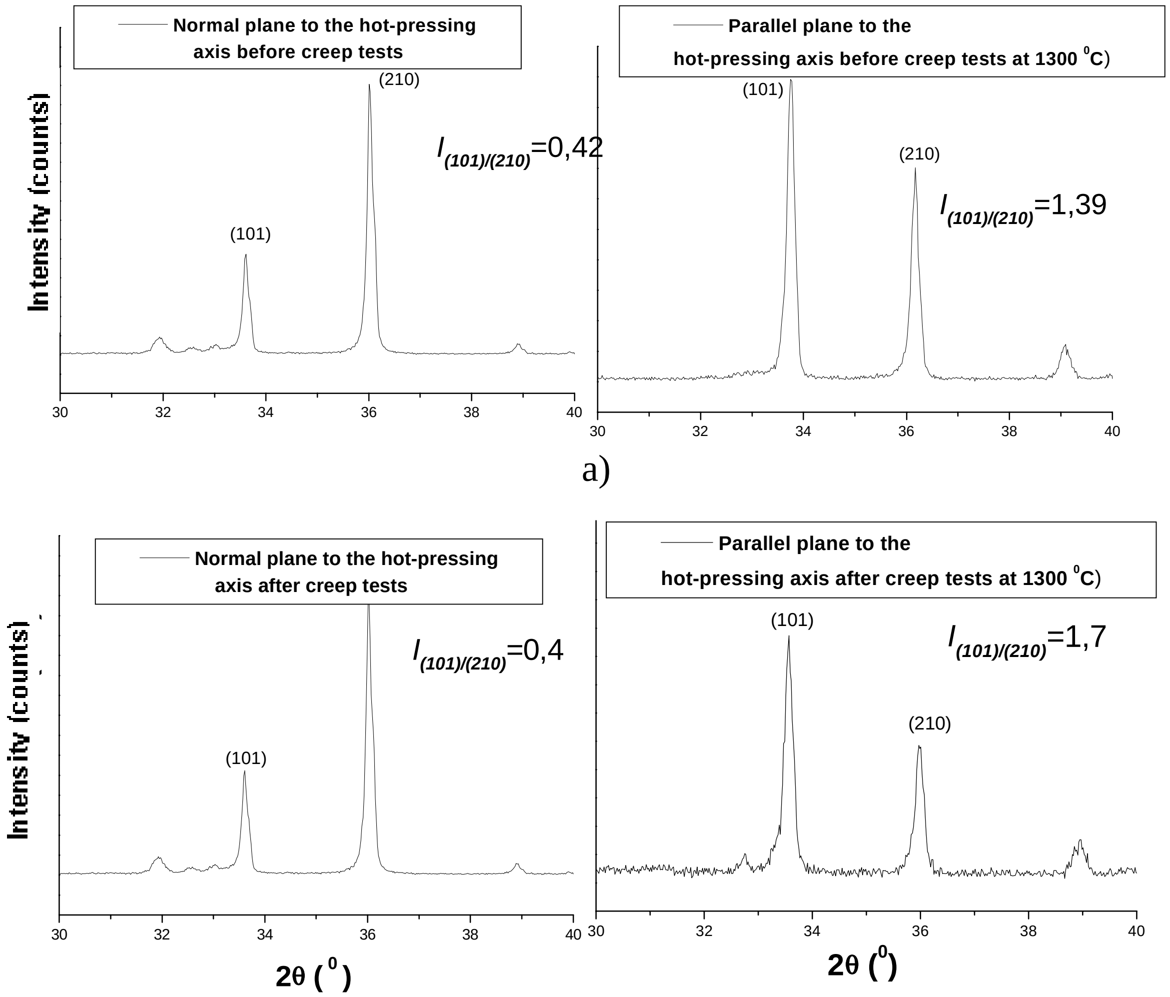


Figure 5. Temperature dependencies of creep rate for Si_3N_4 ceramics under 200, 250 and 300 MPa stresses.

c) XRD observations:

A technique comonly used to determine the anisotropy in planes parallel and perpendicular to the uniaxial hot-pressing direction by X-ray diffraction is based on the ratio of the peak areas between the (101) and (210) planes of the β–Si_3N_4 grains (Lee, 1992, Yoon, 1997).The texture development with grain orientation has been reported in previous studies (Yoon, 1997), wich contain two mechanisms for preferred grain orientation, including physical grain rotation and preferential grain growth due to the stress gradient. Considering rotation during the creep tests was the probable mechanism of texture development. Fig. (6) shows the change in the ratio of β-Si_3N_4 (101/(210) peak area on a bulk specimen before and after creep tests, respectively.



a)

b)

Figure 6. XRD patterns of specimens: a) before creep tests, in the perpendicular (normal) plane to the hot-pressing axis (N) and parallel plane to the hot-pressing axis (P); b) after creep tests at 1300^0C and 300 MPa, in the (N) and (P) planes.

It can be observed, through XRD patterns, that the parralel plane (P plane) of the samples, before the creep tests, shows a great variation on β-Si_3N_4 (101/(210) peak area when compared to N plane. The peaks intensity are 1.39 for the P plane, and 0.42 to the N plane. These values indicate an alignment of the resultant grains that comes from the hot-pressing process. After the creep tests at 1300^0C and under a pressure stress of 300 MPa, can be seen from XRD patterns, that the relation β-Si_3N_4 (101/(210) in the P plane increase from 1.39 to 1.7, indicating that there was a new and continuos global reorientation of the grains in the specimen, confirming the rotation of the grains during the creep test. This indicate the physical grain rotation for texture development is a preferential mechanism and is due to the viscous flow, where the viscous flow can redistribute the grain boundary glassy phase and thus then enables the adjacent grains Si_3N_4 to be rearranged.

4. Conclusion

In this work, a natural yttrium and rare earth oxide solid solution, CRE_2O_3, produced at FAENQUIL-DEMAR, was used as sintering additive of the hot-pressed Si_3N_4 ceramics. The use of the mixed yttrium and rare earth oxide solid solution, CRE_2O_3, as sintering aid was efficient for the obtaining of dense, hard and toughned ceramics.

The creep behavior indicated that grain-boundary sliding controlled by viscous flow can be ruled out in the present study because it is revelead that the stress exponent of all conditions is determined to be about unity. The apparent activation energy is obtained to be aruond 500 $kJmol^{-1}$ and this value corresponds to the heat of solution of the Si_3N_4 in glassy phase. Both, the stress exponent, *n,* and apparent activation energy value, *Q*, are within the range of values obtained in other studies of the compressive creep of Y_2O_3-Al_2O_3-doped-Si_3N_4 ceramics. The creep behavior indicate that this additive can substitute pure Y_2O_3 in the fabrication of ceramic materials, resulting in equivalent properties.

From the results presented in this work, we conclude that the use of the mixed rare earth oxide solid solution, CRE_2O_3, is a viable alternative, low cost sinter additive, enabling the obtention of creep resistant Si_3N_4 ceramics by hot-pressing sintering

5. Acknowledgrment

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References

Arellano-López, A. R., Varela-Feria, F. M. , Martinez-Férnandez, J., Singh, M., 2002, “ Compressive creep of silicon nitride with different secondary phase compositions”, Materials Science and Engineering A, 332, 295-300.

Barsoum, M., 1997, *“Fundamentals of Ceramics,* Mat. Science and Engeneering Series, 668 p.

Bellosi A, 1999, “Design and Process of non-oxide ceramics – case study: factor affecting microstructure and properties of silicon nitride” , in: Gogotsi Y G, Andrieuski R A, Editors, *Materials Science of carbides, nitrides and borides,* Kluwer Academic Publishers, Netherlands, Vol 68 : 285-304.

Björklund H, Falk L. N. L, Rundgren K and Wasén J, 1997, “β-Si_3N_4 grain growth, part I.: Effect of metal oxide sintering additives”, *J Eur Ceram Soc*; 17: 1285-1299.

Cannon, W.R. and Langdon, T.G., 1983, “Review - Creep of Ceramics, part - I” *J Mat Sci* 18 [1], 1-50.

Coble, R. L., 1970, “Diffusion models for hot pressing with surface energy and pressure effects as driving forces”, Journal of Applied Physics, 41 (12), 4798-4807.

Crampon, J., Duclos, R., 1997, “Compressive creep and creep failure of $8Y_2O_3/3Al_2O_3$-Doped hot-pressed silicon nitride”, J. Am Ceram Soc., 80 (1) 85-91.

Evans A.G, Charles E. A, 1976, “Fracture Toughness Determination by Indentation”, *J Am Ceram Soc*; 59: 7-8.

Evans, R.W., Wilshire, B., 1993, “*Introduction to Creep”,* London, The Isntitute of Matertials, 115p.

German R. .M, 1985, *“Liquid Phase Sintering”*, New York, Plenum Press: 127-155.

Herrmann M, Schulz I, Hermel W, Schubert Chr, Wendt A, 2001, “Some new aspects of microstructural design of β−Si_3N_4 ceramics”, *Z. Metallkd,*; 92 (7): 788-795.

Komeya K, 1988, “Fabrication and Properties of Silicon Nitride ceramics”, *Fine Ceramics,* Edited by Saito S, Elsevier, Japan: 175-188.

Lai K. R, Tien T. Y, 1993, “Kinetics of β-Si_3N_4 Grain Growth in Si_3N_4 Ceramics Sintered under High Nitrogem Pressure”, *J. Am. Ceram. Soc.* 76 (1): 91-96.

Lange, F. F., Davis, B. I., Clarke, D. R., 1980, J. Mater. Science, 15, 601.

Lee F., and Bowman, K.J., 1992, “Texture and Anisotropy in Silicon Nitride”, *J.Am.Ceram.Soc,*75 (7) 1748-1755.

Mukhopadhyay A. K, Datta S. K, Chakraborty D, 1999 “Fracture toughness of structural ceramics”, *Ceramics International* ; 25: 447-454.

Peillon F. C and Thevenot F, 2002, “Microstructural designing of silicon nitride related to toughness”, *J Eur Ceram Soc,* 22: 271-278.

Perera D.S, Mitchell D.R.G,Leung S, 2000, “High aspect ratio β-Si_3N_4 grain growth” *J EurCeram Soc* ; 20: 789-794.

Pyzik, A. J., Braman, D. R., 1993 ,”Microstructure and Properties of self-reinforced Silicon Nitride.” *J.Am.Ceram.Soc.*, 76 [11] 2737-44.

Ribeiro S, 1997 “Estudo comparativo das propriedades do nitreto de silício sinterizado com óxido de ítrio/ óxido de silício e concentrado de óxidos de ítrio e terras raras/óxido de silício”. Faculdade de Engenharia Química de Lorena-FAENQUIL, *PhD Thesis*. 194p.

Ribeiro, S., Strecker, K., Vernilli Jr., F., 1997, *“Estudo comparativo da oxidação do nitreto de silício sinterizado com óxidos de ítrio e de terras raras”*In:Encontro Nacional da Sociedade Portuguesa de Materiais, 8., Materiais’97, Marinha Grande - Portugal, Anais. Ed. L. Guerra Rosa. Aveiro: Sociedade Portuguesa de Materiais, v.2, p. 133-141

Ribeiro, S., Vernilli Jr, F., Strecker, K., 1998, “Microestrutura e fases presentes em Si_3N_4 aditivado com Y_2O_3/SiO_2 ou concentrado misto de terras raras $(RE_2O_3)/SiO_2$*”.Cerâmica ABC* (ISSN-0366-6913), v.44, n.285/286, p.43.

Riley F. L, 2000, “Silicon Nitride and Related Materials”, *J.Am.Ceram.Soc*; 83 (2): 245-65.

Strecker K, Gonzaga R, Ribeiro S, Hoffmann M. J, 2000, “Substitution of Y_2O_3 by a rare earth oxide mixtures as sintering additive of Si_3N_4 ceramics”, *Mat. Lett*; 45: 39-42.

Santos C, Ribeiro S, Strecker K, Silva C. R. M, 2003; "Substitution of pure Y_2O_3 by a mixed concentrate of rare earth oxides (CRE_2O_3) as sintering additive of Si_3N_4: A comparative study of the mechanical properties", *J.Mat.Proc.Techn*, in revision.

Santos, C., Strecker, K., Baldacim, S.A., Silva, O .M.M., Piorino, F., Silva, C.R.M., 2003, "Comportamento à fluência de Si_3N_4 produzido com um aditivo de sinterização alternativo – CRE_2O_3.", Anais, 47 CBC - Congresso Brasileiro de Cerâmica, João Pessoa – PB, junho 2003.

Shibuya, N.H., 1998, "Fluência compressiva de cerâmicas de Si_3N_4" Tese de Doutorado – Instituto Tecnológico da Aeronáutica - ITA, S.J.Campos - SP, 185 p.

Silva, C.R.M., 1989, "*Compressive Creep of Silicon Nitride", PhDThesis*, Univ. of Manchester, UK.

Silva, C.R.M., Ying An.C., Piorino, F., 1997, "Energia da ativação para fluência do nitreto de silício", *Anais do 41^o Congresso Brasileiro de Cerâmica*, 3 a 6 junho 1997, S.Paulo-SP, vol I, pp. 218-220.

Wiederhorn, S. M., Hockley, B. J., French, J. D., 1999, " Mechanisms of deformation of silicon nitride and silicon carbide at high temperatures", J. Eur. Ceram. Soc, (19), 2273-2284.

Wötting G, Kanka B, and Ziegler G, 1986, "Microestructural Characterization, and Relation to Mechanical Properties of Dense Silicon Nitride"; in *Non-Oxide Technical and Engineering Ceramics*. Ed. by Hampshire S. Elsevier, London, U.K., 83-96.

Yoon, S.-Y., Akatsu, T., Yasuda, E., 1996 "The microstructure and creep deformation of hot-pressed Si_3N_4 with different amounts of sinteing additives", J. Materials Research, vol 11, No 1, 120-126.

Yoon, S. Y. , Akatsu, T., Yasuda, E., 1997, "Anisotropy of creep deformation rate in hot-pressed Si_3N_4 with prefereed orientation of the elongated grains", Journal of Material Science, 32, 3813-3819.

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