Available online at www.sciencedirect.com
ScienceDirect
Journal of the European Ceramic Society 27 (2007) 1203–1209
www.elsevier.com/locate/jeurceramsoc

A silicon nitride reference material—A testing program of ESIS TC6

Tanja Lube[a,*], Jan Dusza[b,1]

[a] Institut für Struktur- und Funktionskeramik, Montanuniversität Leoben, Peter Tunner Straße 5, A–8700 Leoben, Austria
[b] Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, SK-04353 Košice, Slovak Republic

Available online 24 May 2006

Abstract

Silicon nitrides with sufficient strength for structural applications have been developed 20 years ago. A break-through in the use of these ceramics for structural applications did not yet take place. Most probably, the reason for this is a significant lack of design relevant data.

TC 6 “Ceramics” of the European Structural Integrity Society (ESIS) has established a research program in order to determine a complete set of material properties and data indispensable for design for a commercially available silicon nitride ceramic. The material chosen as the ESIS Silicon Nitride Reference Material is a gas pressure sintered silicon nitride containing ~3 wt.% Al_2O_3 and ~3 wt.% Y_2O_3.

The results available to the present indicate that this specific material has a good mechanical performance at room temperature and up to ~800 °C. Above this temperature the large amount of amorphous grain boundary phase causes a detrimental influence on the environmental assisted crack growth properties and the creep performance.

Keywords: Si_3N_4; Mechanical properties; Thermal properties; Wear resistance; Reference material

1. Introduction

The performance of structural ceramics was impressively improved during the last decades. Many ceramics now reach more than twice the strength measured 30 years ago.[1] Process technologies became less expensive and more reliable.[2] Higher toughness and the existence of a *R*-curve lead to more defect tolerant materials. The understanding of the interaction between the properties of the raw materials, processing and the microstructure steadily grows, allowing materials to be tailored for specific applications.[3] The performance of several commercial silicon nitrides meets the requirements of the designers.[4]

Parallel to the material development, concepts for a reliable design of ceramic components were established.[5–7] Such tools now take into account various failure modes and are implemented in FEM-software.[8–10] Nevertheless, the idea of using a ‘brittle and unreliable’ material such as ‘ceramic’ for structural parts still seems to be a strange idea for most engineers. A lack of personal experience with such materials combined with the uncommon design methodology may be the reason for this. In practice, a majority of applications of ceramics is technically rather unobtrusive (cutting tools, wear resistant parts[11]). Spectacular applications like the ceramic turbocharger rotor are rare.[12]

Several simple design studies exist, that use ‘typical material properties’ – especially for time or cycle dependent properties – to prove the principal suitability of ceramics.[13] These studies demonstrate the crucial influence of these properties on the long-term reliability of components. But data that characterise these properties are only very seldom included in manufacturers’ material data sheets. Only some ceramics have been studied in detail with respect to all kind of properties, for example NC132.[14–16] But often these investigations were focused towards other goals and have to be considered unsystematic with regard to the use of these data for design purposes. A consequence of this lack of data is a lack of implemented examples for appropriate ceramic design.[17]

The aim of this paper is to introduce the ‘Reference Material Testing Program’ (RMTP) of ESIS TC6. In the following sections a description of the concept and of the working tasks is provided. The state of the program together with some selected preliminary results is reported.

* Corresponding author. Tel.: +43 3842 402 4111; fax: +43 3842 402 4102.
E-mail addresses: tanja.lube@mu-leoben.at (T. Lube), dusza@imrnov.saske.sk (J. Dusza).
[1] Tel.: +421 55 7922 462; fax: +421 55 7922 408.

0955-2219/$ – see front matter
doi:10.1016/j.jeurceramsoc.2006.04.020

2. The Reference Material Testing Program of ESIS TC6

In the Technical Committee 6 "Ceramics" (TC6) of the European Structural Integrity Society (ESIS), the idea was born to establish a database containing all property data of one structural ceramic that are relevant for a successful design of components. It is expected, that this program will result in a complete database for mechanical design for that material. Such a database can be the basis for detailed design studies, give a baseline for further material development and will make a fair comparison between alternative materials possible. An enhanced use of ceramics in structural applications might result. The RMTP has now been a common task for ESIS TC6 for a considerable time period. It promoted collaboration between the partners and keeps attracting new potential participants. The high relevance of the RMTP activity is supported by the fact, that participation in the program is voluntary and not funded.

To produce a versatile database, a material that performs reasonable in a large number of possible applications is preferred to ensure a fair comparison with alternative ceramics. A candidate structural ceramic for the RMTP should therefore be applicable at ambient temperature as well as above 1000 °C and have a toughness around 5 $MPa\sqrt{m}$ or more. It should be commercially made by a large European producer and the production should have reached a stable quality. The existence of the material should be assured for several more years.

2.1. Organisation of the RMTP

The aspired goal of providing a complete set of design data involves a large experimental effort comprising state of the art standard tests as well a highly sophisticated experiments. Five working areas comprising the relevant properties were defined and co-ordinators were found for each topic. An overview of the structure of the program is given in Table 1. The working program for each topic was established in a bottom-up procedure. The experimental work should be shared within the group and only some experiments should be reproduced and verified by one or more participants. The co-ordinators take over the organisation of the work within their topic. They collect the results and review them. As specialists in the fields of their topic they are qualified to interpret measurements and identify contradicting data and missing information.

Table 1
Topics in the ESIS Reference Material Testing Program

Topic A	Project coordination, production of specimens, physical and thermal properties as functions of temperature from ambient temperature to 1200 °C
Topic B	Contact loading, hardness, indentation damage, friction and wear, the influence of machining on strength, chipping and related topics
Topic C	Strength at room to high temperatures, biaxial strength, strength distribution and volume effect, fracture toughness and *R*-curve
Topic D	Sub-critical crack growth at ambient to high temperatures, fatigue up to 10^6 cycles at different *R*-values
Topic E	Creep in bending and tension, oxidation and corrosion characteristics, thermal shock

To avoid an influence of specimen preparation samples are produced by one machine shop so far as possible. Tests are to be made according to standard or pre-standard methods if such are available and reasonable. Additional tests employing other methods will complete the database. Minimal results are specified by the RMTP, but each participant is free to perform more in-depth investigations beyond the agreed test extend.

In the starting phase a focus is put on room temperature properties and only screening tests are performed in the range up to 800 °C. Later a limited test program will be carried out at temperatures from 800 °C to 1300 °C. Until now 17 research teams from 10 European countries participate in the RMTP. They are listed in Table 2 as well as the main topics in which they are involved and possible duties as topic co-ordinators.

2.2. Investigated material

The material chosen as the ESIS Silicon Nitride Reference Material is produced by CeramTec (Plochingen, Germany) under the name SL200 B. It is a gas pressure sintered ceramic containing ~3 wt.% Al_2O_3 and ~3 wt.% Y_2O_3. The material is provided in the form of plates (47 mm × 11 mm × 102 mm). These plates have a light skin layer (~1.5 mm thick) and a darker bulk. Since it is not known if this colour difference is also responsible for any difference in mechanical properties, the position of the outer layer is recorded for all experiments. To avoid an influence of the specimen production, all specimens are produced at the same partner. Care was taken that for each individual specimen the plate from which it comes as well as the position within the plate is recorded during all production steps. Sample sets were put together randomly from a large number of specimens. About 1200 bend bars for strength tests were produced and distributed, as well as different pieces for wear tests and other investigations, approximately 80 plates have been used to produce these specimens.

3. Properties of the ESIS reference silicon nitride

3.1. Physical and thermal properties

The microstructure of the material consists of β-Si_3N_4 grains with an aspect ratio of ~3–5 and an intergranular glassy phase. It could be shown[18,19] that also a small amount (0.03–0.16 wt.%, depending on the position in the plate, see Section 2.2) of α-Fe remaining from the original powder is present. The volume fraction of glassy phase as determined by various techniques[20] is around 12%. The glass transition temperature of the amorphous silicate phase was estimated[19] by differential scanning calorimetry to be at 950 °C. No crystallisation takes place during heating up to 1400 °C in N_2. Young's modulus and Poisson's ratio were determined[19] using the IET-technique (see Table 3). The coefficient of linear thermal expansion α_{lin} and the specific heat c_p were measured in argon. Thermal diffusivity α was determined by the laser flash method. These data were used to calculate thermal conductivity λ according to $\lambda = \alpha\rho c_p$. The

Table 2
Participants in the ESIS TC6 Reference Material Testing Program

Participants name, organisation, country	Main working areas co-ordination duty (c:)
R. Danzer, T. Lube, Institut für Struktur- und Funktionskeramik, Montanuniversität Leoben, A	Organisation, specimens, strength, crack growth, c: topics A and C
J.-P. Erauw, Belgian Ceramic Research Centre, Mol, B	Wear, strength
G. Roebben[a], Department of Metallurgy and Materials Engineering, Katholieke Universiteit Leuven, B	Basic properties
A.-P. Nikkilä, Institute of Materials Science, Tampere University of Technology, SF	Fatigue
O. Rosenfelder, K.Friederich, CeramTec AG, Plochingen, BRD	Material
M. Bartsch, Institut für Werkstoff-Forschung, DLR, Köln, BRD	Toughness
R. Steinbrech, Institut für Werkstoffe und Verfahren der Energietechnik II, Forschungszentrum Jülich GmbH, BRD	Toughness
H. Klemm, Institut für keramische Technologien und Sinterwerkstoffe, Dresden, BRD	Crack growth
R. Westerheide, Fraunhofer Institut für Werkstoffmechanik, Freiburg, BRD	Wear, toughness, c: topic B
G. De Portu, Instituto di Richerce Tecnologiche per la Ceramica, Faenza, I	Wear
V. Sglavo, Dipartimento di Ingegneria dei Materiali, Universitá di Trento, I	Strength, toughness
J. Dusza, Institute of Materials Research, Slovak Academy of Science, Košice, SK	Strength, creep, crack growth, c: topic E
M. Anglada, J. Alcala, Departament de ciencia dels Materials i Enginyeria Metal.lurgica, Universidad Politénica de Catalunya, Barcelona, E	Fatigue, c: topic D
J. Kübler, Eidgenössische Materialprüf.- und Forschungsanstalt, Dübendorf, CH	Crack growth, creep, c: topic D
R. Morrell, National Physical Laboratory, Teddington, UK	Wear, basic properties, c: topic B
M. Reece, Department of Materials, Queen Mary and Westfield College, London, UK	Electrical properties
Z. Chlup, Institute of Sciences of the Czech Republic, Institute of Physics of Materials, Brno, CZ	Toughness

[a] Now at Reference Materials Unit, Institute for Reference Materials and Measurements, Joint Research Centre of the European Commission, Retieseweg 111, B-2440 Geel, Belgium.

change of the density ρ with temperature was estimated using the approximation[21] that the volumetric expansion α_V equals three times the linear expansion α_{lin}, $\alpha_V = 3\alpha_{lin}$. The results are shown in Fig. 1 and in Table 3 together with the data from the manufacturers data sheet.

3.2. Hardness, wear and machining

Hardness tests using different indenters and different kinds of wear tests were conducted[22] on both type of surfaces, the light skin and the dark bulk. Results of the hardness measurements are included in Table 3. Erosion tests were conducted at 75 m s^{-1} air speed and 10.7 g s^{-1} feed rate of a 220 μm sand. Ball-on-disc wear tests were performed. The ball for these tests was substituted by pins made from the same silicon nitride with a tip curvature radius of 5.74 mm. The total sliding distance was 5 km, at a wear track diameter of 25 mm. Sliding speed was varied from 0.01 m s^{-1} to 0.1 m s^{-1} with applied load of 5 N or 10 N. The material proved to be very resistant to erosion, with the dark bulk showing an even lower mass loss at a given total mass of sand than the skin. Almost no specific wear was measured in ball-on-disc wear tests and no transition from mild to severe wear could be identified.

3.3. Strength, strength statistics and fracture toughness

At ambient temperature strength tests were performed according to EN 843 1[23] in 3- and 4-point loading with a 40 mm (40/20 mm) span and the tensile face close to the skin as well as in the bulk.[24] Additionally, biaxial strength was investigated using the ball-on-three-balls test.[25] Two specimen sets were tested: (a) diameter 43.4 mm and thickness 3.7 mm on an a support with diameter 34.6 mm and (b) 10 mm × 8.4 mm × 2 mm on a support with diameter 7.1 mm. The tensile surface of all strength specimens was ground with a D15 diamond disc. A more detailed description of the these tests can be found elsewhere.[24,26] The parameters of the Weibull distributions were determined for each set following the ENV 843-5.[27] The same type of defects

Table 3
Properties of the Reference Material—Manufacturers data and results from the RMTP (values in brackets correspond to 95% confidence limits)

Property	Unit	Manufacturer	ESIS Testing Program
Density	kg/m^3	3210	3190 ± 9
4-point bending strength	MPa	750	867 (852–881)
Weibull modulus	–	12	14 (11–16)
3-point bending strength	MPa	n.a.	985 (965–1006)
Weibull modulus	–	n.a.	16 (12–20)
Fracture toughness	MPa$\sqrt{m}$	7 (IF)	4.9 ± 0.1 (SEVNB), 7.9 ± 1.2 (IF)
Young's modulus	GPa	305	303 ± 1.3 (bulk) 307 ± 2 (skin)
Hardness HV10	GPa	16.2	14.3
Thermal conductivity	W/mK	21 (20–100 °C)	26 (20–100 °C)
Thermal expansion	10^{-6} K^{-1}	3.2 (20–400 °C)	2.6 (20–400 °C)
Specific heat	kJ/kg K	0.7 (20–100 °C)	0.76 (20–100 °C)

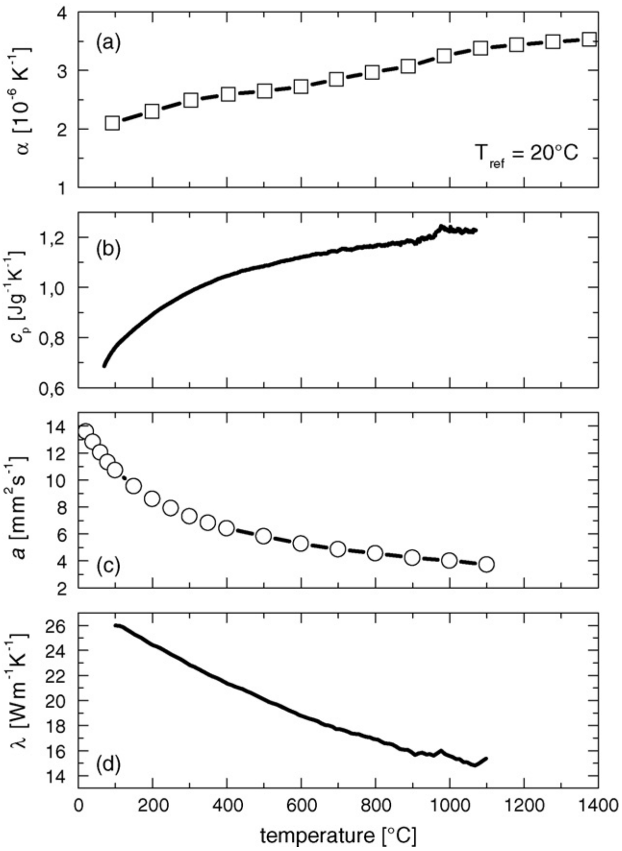

Fig. 1. Thermal properties. (a) Coefficient of linear thermal expansion for a reference temperature of 20 °C, (b) specific heat, (c) thermal diffusivity and (d) thermal conductivity.

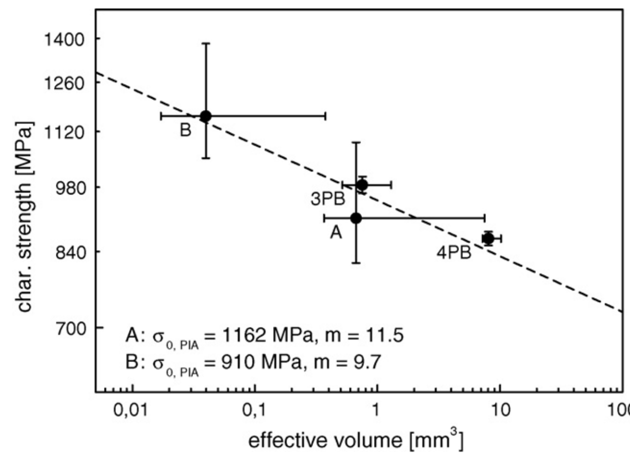

Fig. 3. Dependence of the characteristic strength of uniaxial and biaxial strength specimens on the effective volume. The scatter bars refer to the 95% confidence intervals for characteristic strength and effective volume, respectively. The dashed line indicates the behaviour predicted according to the Weibull theory on the basis of the bend tests.

(agglomerates of amorphous intergranular phase, regions with microporosity and iron inclusions) was detected as failure origins for all types of specimens. These of defects can also be found on polished sections as shown in an example in Fig. 2.

The results of the strength tests are summarised in Table 3 and in Fig. 3. In order to compare the biaxial data with the uniaxial data, the PIA criterion[28] was used to calculate an equivalent stress for the biaxial tests. From Fig. 3 it is obvious, that the biaxial strength follows the same volume dependence as the uniaxial strength.

The influence of test temperature on the strength can be obtained from Fig. 4. At increasing temperature, strength remains almost constant at a value of ∼870 MPa up to 800 °C. Above this temperature a drop can be observed. This behaviour can be explained by the softening of the amorphous phase, which takes place above 950 °C.[19] At high temperatures, an influence of loading speed on the strength was also observed[24,29,30] as indicated by the difference between tests with high and low loading rates.

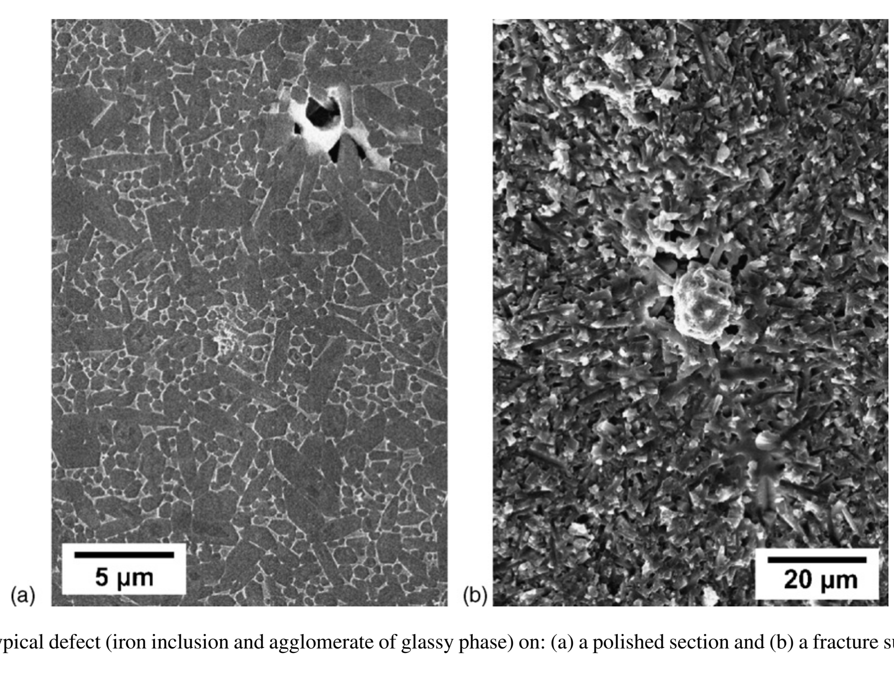

Fig. 2. Microstructure and typical defect (iron inclusion and agglomerate of glassy phase) on: (a) a polished section and (b) a fracture surface of a bending specimen.

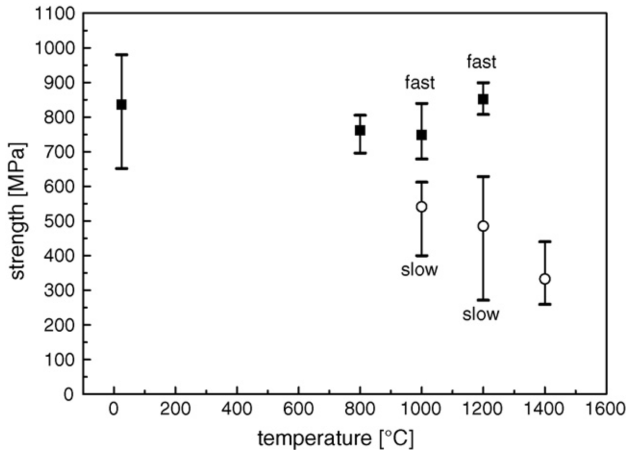

Fig. 4. The influence of test temperature on the strength. Bars indicate the scatter of the data (minimal and maximal values).

Fracture toughness of the bulk determined with the SEVNB[31] method is $K_{Ic,SEVNB} = 4.9 \pm 0.1\,MPa\sqrt{m}$. Fracture toughness by the Chevron-notch (CNB) method[32] is $K_{Ic,CNB} = 5.2 \pm 0.1\,MPa\sqrt{m}$. No significant difference in fracture toughness between skin and bulk was found[24] using the IF-method.[33] A slightly rising *R*-curve was reported[24] from stable crack growth experiments and fracture toughness tests using long cracks. This *R*-curve can explain the higher values of $K_{Ic,CNB}$ compared to $K_{Ic,SEVNB}$. During CNB tests stable crack growth takes place prior to fracture thus leading to a toughness result on the intermediate or upper part of the *R*-curve.[34]

3.4. Time dependent failure

The influence of time on strength at room temperature was investigated[24] by measuring the time to failure in static bend loading (static bend tests) and by determination of the influence of loading rate on strength (dynamic bend tests). At 800 °C, 1000 °C and 1200 °C the time to failure in static bending was determined. Tests at ambient temperature were conducted in deionized water, at high temperatures in air. The data were evaluated assuming a power-law relation for the dependence of the crack growth velocity v on the applied stress intensity $K_I : v \propto K_I^n$. Corresponding plots are shown in Fig. 5. At room temperature and 800 °C the exponent $n_{RT} \approx 42$, at 1000 °C $n_{1000} \approx 22$ and at 1200 °C $n_{1200} \approx 6$. The low value of n_{1200} is an indication that creep may play an important role in failure at this temperature. It was shown,[30] that at 1200 °C the lifetime is controlled by sub-critical growth of a single crack at high (200–300 MPa) applied stresses. At low applied stresses (~150 MPa) failure is caused by non-localized creep damage and multiple crack growth.

Cyclic loading experiments were conducted in an ambient air environment (25 °C, relative humidity 50%) with a sinusoidal load wave of frequency 117 ± 4 Hz and loading ratio ($R = P_{min}/P_{max}$) of 0.1.[35] Three different stress levels in 4-point bending (40 mm outer and 20 mm inner spans) were used. The time to failure at a given stress level exhibits a variation of about three orders of magnitude. Such difference in lifetime might even increase if the tests were not suspended after 10^7 cycles (equivalent to 10^5 s). By comparison with the static lifetime data it can be argued that the cyclic lifetimes are fundamentally governed by underlying environmentally assisted (static fatigue) effects. Simple calculations using the frequency and load ratio, *R*, indicate that, in the absence of true mechanical damage under cyclic loads, cyclic lifetimes shall be an order of magnitude longer than static lifetimes. Overall, the results are similar to those obtained by Ohya et al.[36] and Jacobs and Chen[37] where environmental effects are found to play a significant role in the cyclic behaviour. It seems sensible to propose that, for structural applications of the ESIS Si_3N_4 reference material, cyclic lifetimes could be estimated on the basis of static results. A different behaviour can however be expected for other loading ratios, especially for $R = -1$.

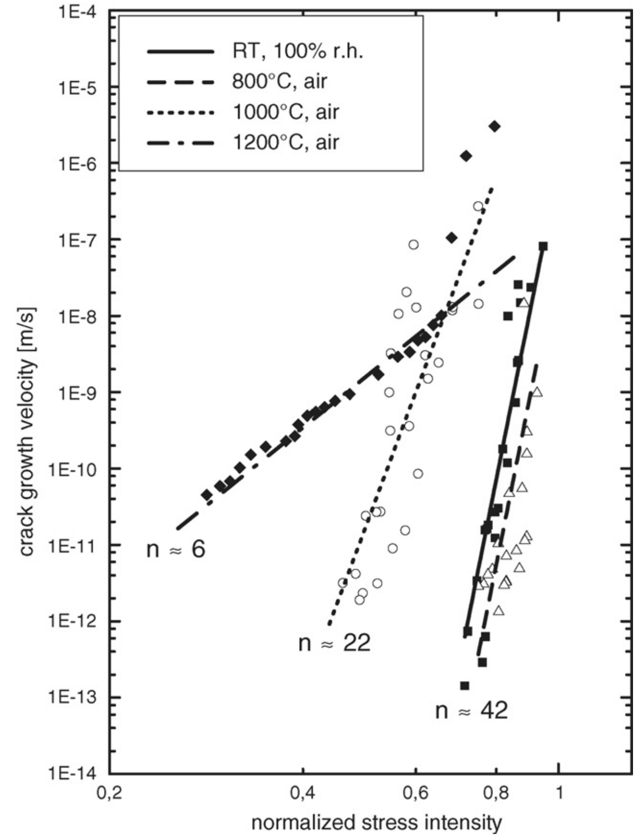

Fig. 5. Dependence of crack growth velocity on applied stress intensity (v–K curves) at different temperatures.

3.5. Creep and high temperature behaviour

Creep tests were conducted in 4-point bending (40/20 mm spans) in air at temperatures of 1150 °C, 1175 °C and 1200 °C with applied stresses from 50 MPa to 175 MPa. The creep curves exhibit all three stages of creep up to 125 MPa at 1175 °C. Examples are given in Fig. 6. The stress exponent for stationary power-law creep was determined to be $n_{creep,s} = 2.3$–4.2, for low and high temperatures, respectively (see Fig. 7). The activation energy was found to be ~820 kJ/mol. These results indicate that the material has a rather poor creep performance (as compared

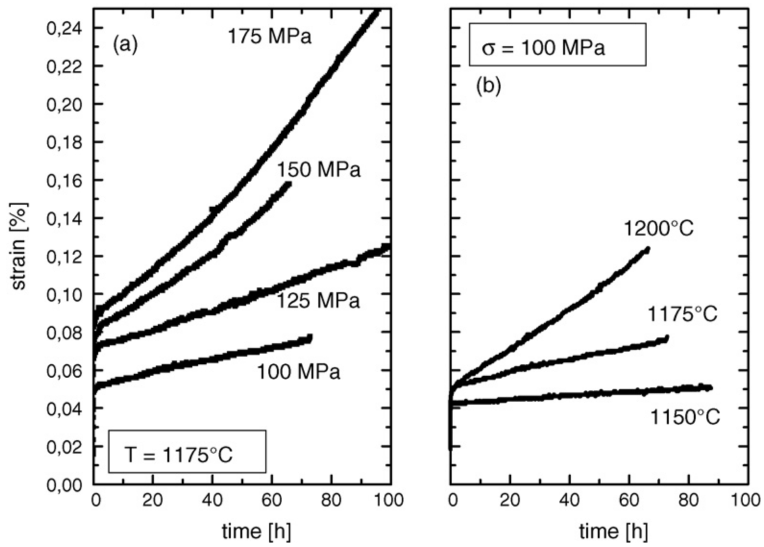

Fig. 6. (a) Creep curves at 1175 °C and (b) comparison of creep strain for an applied stress of $\sigma = 100$ MPa for different temperatures.

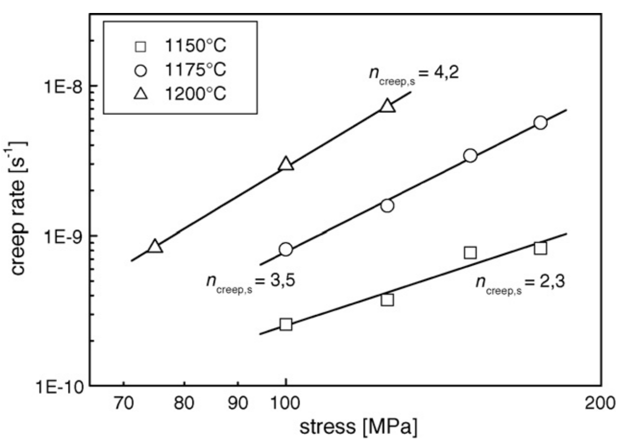

Fig. 7. Stationary creep rates as functions of elastically calculated outer fibre stress for different temperatures.

to modern commercial silicon nitrides optimized for high temperature applications[38]) which can most probably be attributed to the large amount of amorphous grain boundary phase and the iron inclusions.

The material was subjected to oxidation between 1000 °C and 1300 °C during 250 h. The effect of oxidation was characterised by the specific weight gain and the retained room temperature strength. While the specific weight gain increases with increasing oxidation temperature, the retained strength is lowest after exposure at 1100 °C (63% of the original strength) and rises to 70% of the original strength at 1300 °C.

4. Summary and outlook

An ESIS Reference Material Testing Program has been established with the aim to measure the basic data relevant for design of a commercial silicon nitride (SL 200B, CeramTec, Plochingen BRD). The project is a carried out by TC6 “Ceramics” of the European Structural Integrity Society. Participation is voluntary and not funded. At the present time a majority of the specimens has been manufactured and distributed. A considerable part of the experiments is finished by now.

At room temperature the material has a 4-point bend strength of 867 MPa, a Weibull modulus of 15 and a fracture toughness around 5 MPa$\sqrt{m}$. The exponent of the power law for subcritical crack growth is approximately 42. Mechanical performance deteriorates at temperatures above ~800 °C. At high temperatures the large amount of amorphous grain boundary phase exerts a detrimental influence on the environmental assisted crack growth properties and the creep properties. First wear test results indicate that the wear behaviour at room temperature is excellent.

Detailed results of individual tests are published separately. A complete collection of data will be available in soon

Acknowledgements

The authors acknowledge the efforts of all participants who contributed to the present state of the program. They thank W. Preis of Lehrstuhl für Physikalische Chemie, Montanuniversität Leoben, Austria, for c_p determination.

References

1. Hamano, Y., Progress in structural applications of silicon nitride. In *Silicon-Based Structural Ceramics*, ed. B. W. Sheldon and S. C. Danforth. The American Ceramic Society, Westerville, 1994, pp. 3–14.
2. Mörgenthaler, K. D., Ceramic valves—a challenge? In *Ceramic Materials and Components for Engines*, ed. K. Niihara, S. Hirano, S. Kanzaki, K. Komeya and K. Morinaga. Japan Fine Ceramic Association, Tokyo, 1998, pp. 46–51.
3. Hoffmann, M. J., Analysis of microstructural development and machanical properties of Si_3N_4 ceramics. In *Tailoring of Mechnical Properties of Si_3N_4 Ceramics*, ed. M. J. Hoffmann and G. Petzow. Kluwer Academish Publishers, Dordrecht, 1994.
4. Savitz, M., Commercialization of advanced structural ceramics. *Am. Ceram. Soc. Bull.*, 1999, **78**(1), 53–56.
5. Weibull, W., *A Statistical Theory of Strength of Materials*. Royal Swedish Institute for Engineering Research, Stockholm, 1939.

6. Davidge, R. W., *Mechanical Behaviour of Ceramics*. Cambridge University Press, Cambridge, 1979.
7. Matsui, M., The reliability evaluation of structural ceramics. *FC Annual Report for Overseas Readers*, 1989, pp. 20–26.
8. Nemeth, N. N., Manderscheid, J. M. and Gyekenyesi, J. P., Designing ceramic components with the CARES computer program. *Am. Ceram. Soc. Bull.*, 1989, **68**(12), 2064–2072.
9. Brückner-Foit, A., Heger, A. and Munz, D., Evaluation of failure probability of multiaxially loaded components using the STAU postprocessor. *Ceram. Eng. Sci. Proc.*, 1993, **14**(7–8), 331.
10. Brückner-Foit, A., Heger, A. and Munz, D., Strength and lifetime distributions of ceramic parts under multiaxial loading. In *Mechanische Eigenschaften Keramischer Konstruktionswerkstoffe*, ed. G. Grathwohl. DGM Informationsgesellschaft, Oberursel, 1993, pp. 43–54 [in German].
11. Jack, K. H., A reappraisal of nitrogen ceramics for engine applications. In *Ceramic Materials and Components for Engines*, ed. K. Niihara, S. Hirano, S. Kanzaki, K. Komeya and K. Morinaga. Japan Fine Ceramic Association, Tokyo, 1998, pp. 203–207.
12. Tatsumi, T., Takehara, I. and Ichikawa, Y., Ceramic gas turbine "CGT302" development summary. In *Ceramic Materials and Components for Engines*, ed. J. G. Heinrich and F. Aldinger. Wiley/VCH Verlag, Weinheim, 2001, pp. 45–50.
13. Hempel, H. and Wiest, H., *Structural Analysis and Life Prediction for Ceramic Gas Turbine Components for the Mercedes-Benz Research Car 2000*. ASME Paper No. 86-GT-199, 1986.
14. Quinn, G. D. and Quinn, J. B., Slow crack growth in hot-pressed silicon nitrde. In *Fracture Mechanics of Ceramics*, ed. A. G. Evans, D. P. H. Hasselman and F. F. Lange. Plenum Press, New York, London, 1993, pp. 603–636.
15. Quinn, G. D. and Gettings, R., Standard reference material 2001: ceramic fracture toughness. *Ceram. Eng. Sci. Proc.*, 1999, **20**(3), 513–523.
16. Choi, S. R., Powers, L. M., Holland, F. A. and Gyekenyesi, J. P., Creep of silicon nitride under various specimen/loading configurations. In *Ceramic Materials and Components for Engines*, ed. J. G. Heinrich and F. Aldinger. Wiley/VCH Verlag, Weinheim, 2001, pp. 291–298.
17. Rubeša, D. and Danzer, R., The peculiarities of designing with brittle materials—weak points and deficiencies. In *Fracture from Defects*, ed. E. R. d. l. Rios and K. J. Miller. EMAS Publishing, West Midlands, 1998, pp. 455–460.
18. Lube, T., Danzer, R. and Steen, M., A testing program for a silicon nitride reference material. In *Improved Ceramics Through New Measurements, Processing and Standards*, ed. M. Matsui, S. Jahanmir, H. Mostgaci, M. Naito, K. Uematsu, R. Waesche and R. Morrell. The American Ceramic Society, Westerville, 2003, pp. 259–268.
19. Roebben, G. *et al.*, Microstructure characteristics related to the high temperature fracture resistance of the ESIS silicon nitride reference material. In *Fracture Beyond 2000*, *Vol III*, ed. A. Neimitz, I. V. Rokach, D. Kocanda and K. Golos. EMAS Publications, Sheffield, 2002, pp. 77–84.
20. Roebben, G., Sarbu, C., Lube, T. and Van der Biest, O., Quantitative determination of the volume fraction of intergranular phase in sintered silicon nitride. *Mater. Sci. Eng.*, 2004, **A 370**, 453–458.
21. Speyer, R. F., *Thermal Analysis of Materials*. Marcel Dekker, Inc., New York, Basel, 1994, pp. 165.
22. Morrell, R., de Portu, G. and Erauw, J.-P., Subtask B—contact mechanics. Personal communication, 2004.
23. European Standard EN 843-1, *Advanced Technical Ceramics – Monolithic Ceramics – Mechanical Properties at Room Temperature: Part 1—Determination of Flexural Strength*, 1995.
24. Lube, T. *et al.*, Strength and fracture toughness of the esis silicon nitride reference material. In *Fracture Beyond 2000*, *Vol II*, ed. A. Neimitz, I. V. Rokach, D. Kocanda and K. Golos. EMAS Publications, Sheffield, 2002, pp. 409–416.
25. Börger, A., Supancic, P. and Danzer, R., The ball on three balls test for strength testing of brittle discs—stress distribution in the disc. *J. Eur. Ceram. Soc.*, 2002, **22**(8), 1425–1436.
26. Harrer, W., *et al.*, The ball on three balls test—strength, fractography and failure analysis of different materials, this issue.
27. European Standard ENV 843-5, *Advanced Technical Ceramics – Monolithic Ceramics – Mechanical Properties at Room Temperature: Part 5—Statistical Evaluation*, 1997.
28. Börger, A., Eine Methode zur biaxialen Festigkeitsprüfung von Scheiben aus sprödem Werkstoff. Dissertation, Montanuniversität, Leoben, 2004.
29. Lube, T. and Danzer, R., The ESIS silicon nitride reference material testing program. *CESP*, 2003, **24**(4), 337–342.
30. Kovalcík, J., Dusza, J., Lube, T. and Danzer, R., Delayed failure behaviour of the ESIS silicon nitride reference material at 1200 °C in air. *Mater. Lett.*, 2004, **58**(6), 871–875.
31. Kübler, J., *Procedure for Determining the Fracture Toughness of Ceramics Using the Single-Edge-V-Notched Beam (SEVNB) Method*, GKSS-Forschungszentrum on behalf of the European Structural Integrity Society, June 2000.
32. Chlup, Z., Fracture toughness of Si_3N_4 by CNB method. *Pers. Commun.*, 2004.
33. Niihara, K., Morena, R. and Hasselman, D. P. H., Further reply to comments on 'elastic/plastic indentation damage in ceramics: the median/radial crack system'. *J. Am. Ceram. Soc.*, 1982, **65**(7), C-116.
34. Munz, D. and Fett, T., *Ceramics*. Springer, Berlin, Heidelberg, 1999, pp. 298.
35. Lube, T., Alcala, J., Dusza, J. and Klemm, H., A silicon nitride reference material for ceramic design. In *Proceedings of 11th International Conference on Fracture*, ed. A. Carpinteri, 2005.
36. Ohya, K., Ogura, K. and Takatsu, M., Cyclic fatigue properties of sintered Si_3N_4. *Fatigue of Advanced Materials*. Materials and Components Engineering Publishers Ltd., Edgbaston, 1991, pp. 239–253.
37. Jacobs, D. S. and Chen, I.-W., Mechanical and environmental factors in the static and cyclic fatigue of silicon nitride. *J. Am. Ceram. Soc.*, 1994, **77**(5), 1153–1161.
38. Wiederhorn, S. M. and Ferber, M. K., Silicon nitride for gas turbines. *Curr. Opin. Solid State Mater. Sci.*, 2001, **5**(4), 311–316.