Materials Science Forum Vols. 416-418 (2003) pp 579-583
Online available since 2003/Feb/15 at www.scientific.net

doi:10.4028/www.scientific.net/MSF.416-418.579

Elastic Modulus Determination of ß-$Si_{6-x}Al_xO_xN_{8-x}$ with Rare Earth Concentrate by Dynamic Mechanical Analysis

I. R. Ruiz, H. Takiishi, R. N. Faria, L. F. C. P. Lima and J. C. Bressiani

Department of Materials Engineering Energy and Nuclear Research Institute
C. P. 11049, Pinheiros – S. Paulo – SP- Brazil. CEP 05422-970.

Keywords: elastic modulus, SiAlONs, rare earth additives and DMA.

Abstract: In the present work the elastic modulus of β-$Si_{6-x}Al_xO_xN_{8-x}$ ceramics with 5% of rare earth concentrate has been studied by a dynamic mechanical flexural method. The obtained results are discussed in terms of the coefficient x values, which in this case varied from 0.39 to 1.5, as well as in terms of residual porosity of the sintered samples. In order to follow up the influence of the latter parameter several samples were subjected to post-sintering HIP treatment yielding fully dense material. Elastic modulus for the investigated materials varied from 257 to 312 GPa.

Introduction

It is well known that the elastic modulus (E) of a ceramic material can change from a low to high values accordingly to the ionic or covalent character of the chemical bonding [1]. The elastic modulus of silicon nitride with yttria and zirconia, at room temperature, has been found to be approximately 309 GPa [2]. In a similar manner, additions of Y_2O_3 and Yb_2O_3 to Si_3N_4 also yield a material with E values in the range of 287 to 312 GPa [3]. Sc_2O_3 additions [4] also resulted in a material with E in the range of 292 to 314 MPa, which were in good agreement with a previous work [5].It has been also shown [6] that the elastic modulus is strongly dependent on the phase composition of dense Si_3N_4 ceramics, in particular the α/β phase ratio due to the difference between intrinsic properties of the two phases. According to this paper [6], α and β-Si_3N_4 exhibited elastic modulus of 340 and 312 GPa, respectively. None of the previous works [1-6] have reported about investigations on β-SiAlON with rare earth concentrate. In the present investigation the elastic modulus of β-$Si_{6-x}Al_xO_xN_{8-x}$ ceramics with 5% of rare earth concentrate has been studied using a dynamic mechanical analysis (DMA).

Experimental

In the present study β-$Si_{6-x}Al_xO_xN_{8-x}$ samples with 5, 10, 15 and 20 eq. % aluminum additives (x = 0.39, 0.77, 1.15 and 1.5), and also with a constant amount of rare earth concentrate additives of 5 wt % were investigated. The exact composition of the rare earth concentrate used are given in Table I. Samples in the shape of bars were uniaxially and cold isostatically pressed, and subsequently sintered at 1973 K in nitrogen under normal pressure. Samples for elastic modulus

determination were diamond ground to the final dimensions of 4 x 1.4 x 45 mm rectangular bars. In order to study the effect of porosity on the elastic modulus some samples were, after sintering, hot isostatically pressed at 2023 K under 150 MPa nitrogen pressure.

Elastic modulus was measured in a dynamical mechanical analyzer Netzch DMA 242 using a three point bending flexural mode. For porous samples, the measured values of the elastic modulus were normalized to zero porosity level using the expression:

$$E = E_o (1-1.9P+0.9P^2) \quad \text{(Eq. 1)}$$

where E_o is the elastic modulus of a fully dense material, and P is the pore volumetric fraction. This relationship was initially developed for calculation of elastic modulus of porous materials containing up to 50 % of porosity and a Poisson rate of about 0.3 using table values of elastic modulus of fully dense materials and the measured porosity of sintered materials[1,7]. However, in our case this relationship was used inversely since the standard values of fully dense β-SiAlONs of various stoichiometries are not available, and exactly these values presented the goal of the present work.

Table I - Composition of rare earth concentrate.

Rare earth oxide	Wt %
Y_2O_3	89.5
Er_2O_3	3.9
Dy_2O_3	3.8
Lu_2O_3	1.8
Gd_2O_3	0.6
Ho_2O_3	0.2
Tm_2O_3	0.2

Results

Fig. 1 shows the variation of the elastic modulus (E and E_o) with aluminum concentration for pressure-less sintered samples. It can be seen that there is a decrease in the elastic modulus as the Al concentration is increased. Substantial difference between E and E_o is noted for lower Al concentrations where the porosity is higher. Such behavior can be attributed to the increase in substitution of Si^{+4} and N^{+3} for Al^{+3} leading to β-$Si_{6-x}Al_xO_xN_{8-x}$ solid solution formation, which have a higher degree of ionic bonding, and also to a higher yield of amorphous aluminosilicate intergranular phases.

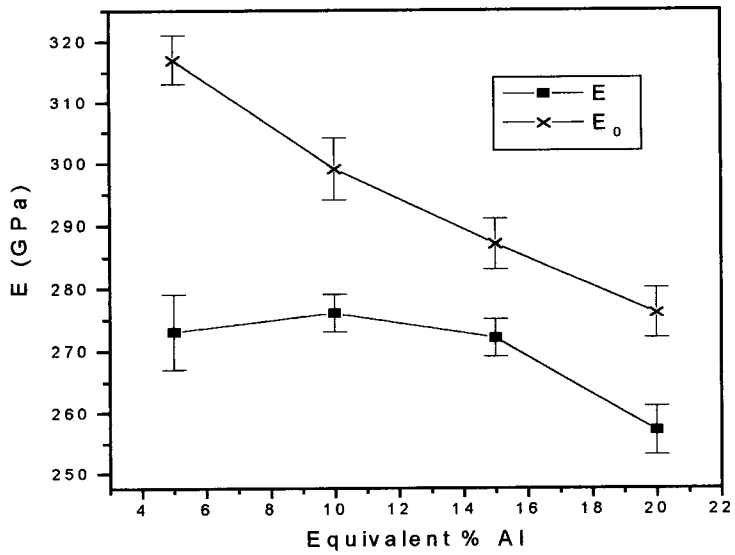

Fig. 1 – Elastic modulus (E and E_0) with variation of the aluminum concentration for samples pessure-less sintered.

Fig. 2 shows the variation of the elastic modulus (E and E_0) with aluminum concentration for samples sintered and subsequently subjected to HIP. In this case there is a better densification and consequently a lesser disparity between E and E_o. Some of the properties of the studied β-$Si_{6-x}Al_xO_xN_{8-x}$ materials are summarized in Table II.

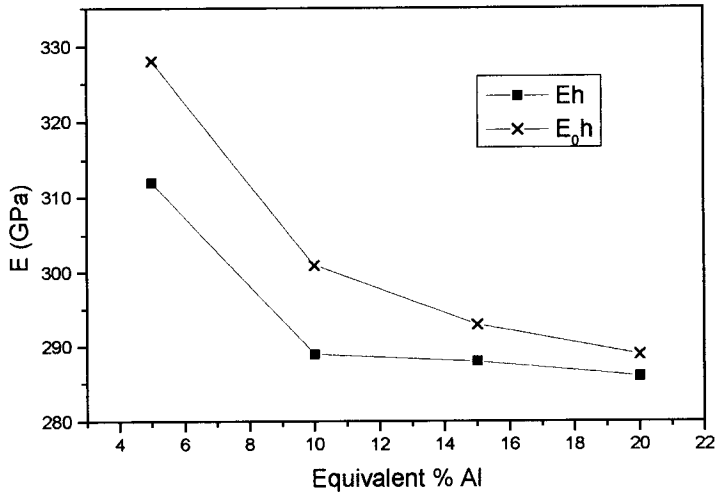

Fig. 2 - Elastic modulus (E and E_0) with variation of the aluminum concentration for samples sintered and subsequently subjected to HIP.

Table II – Some properties of sintered and sintered and HIP-ed SiAlONs.

X values	Eq. % Al	SINTERD				SINTERED AND HIP-ed			
		d_T %	Pore %	E (GPa)	E_0 (GPa)	d_T %	Pore %	E (GPa)	E_0 (GPa)
0.39	5	92.5	7.5	273±6	317±4	97.3	2.7	312	328
0.77	10	95.8	4.2	276±3	300±5	97.9	2.1	289	301
1.15	15	97.1	2.9	272±3	287±4	99.1	0.9	288	293
1.50	20	96.2	3.8	257±4	276±4	99.4	0.6	286	289

Conclusions

Increasing the amount of aluminum concentration on the β-SiAlON compound causes a decrease in the elastic modulus of the specimens. As expected, a lower the porosity yields a material with higher elastic modulus. Specimens that were hot isostatically pressed after sintering showed superior elastic modulus. DMA proved to be an excellent method to study these materials.

Acknowledgements

The authors would like to express their gratitude to CNPq, PRONEX/FINEP, and FAPESP for financial support. Thanks one also due to Dr. V. A. Izhevskiy for the help with the final revision of the work.

References

[1] D. W. Richerson, in Modern Ceramic Engineering – Properties, Processing and Use in Design. Second Edition (1992).

[2] C.T. Bodur, *J. Mat. Sci.*, v. 30, p. 980-988, 1995.

[3] H. Klemm; G. Pezzotti, *J. Am. Ceram. Soc.*, v. 77, n. 2, p. 553-561, 1994.

[4] S.R. Choi; W.A. Sanders; J.A. Salem; V. Tikare, *J. Mat. Sci. Let.*, v. 14, p. 276-278, 1995.

[5] W.A. Fate, *J. Appl. Phys.*, v. 46, n. 6, p. 2375-2377, 1975.

[6] S.K. Lee; K.S. Lee; B.R. Lawn; D.K. Kim, *J. Am. Ceram. Soc.*, v. 81, n. 8, p. 2061-2070, 1998.

[7] W. D. Kingery; H. K. Bowen; D. H. Uhlmann, in Introdution to Ceramics. Second Edition. John Wiley & Sons, N. York, (1976).

Advanced Powder Technology III
doi:10.4028/www.scientific.net/MSF.416-418

Elastic Modulus Determination of ß-Si_{6-x}Al_xO_xN_{8-x} with Rare Earth Concentrate by Dynamic Mechanical Analysis
doi:10.4028/www.scientific.net/MSF.416-418.579