Forming Mullite-Ceramics Reinforced with ZrO_2-t Starting from Mullite-ZrO_2-t and Kyanite-Al_2O_3-ZrO_2-t Mixtures

Narottam P. Bansal, J. P. Singh, Song Won Ko, Ricardo H. R. Castro, Gary Pickrell, Navin Jose Manjooran, K. M. Nair andGurpreet Singh

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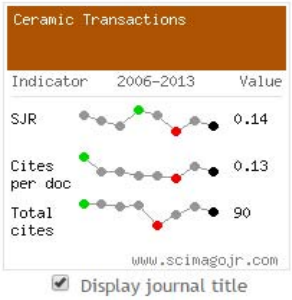

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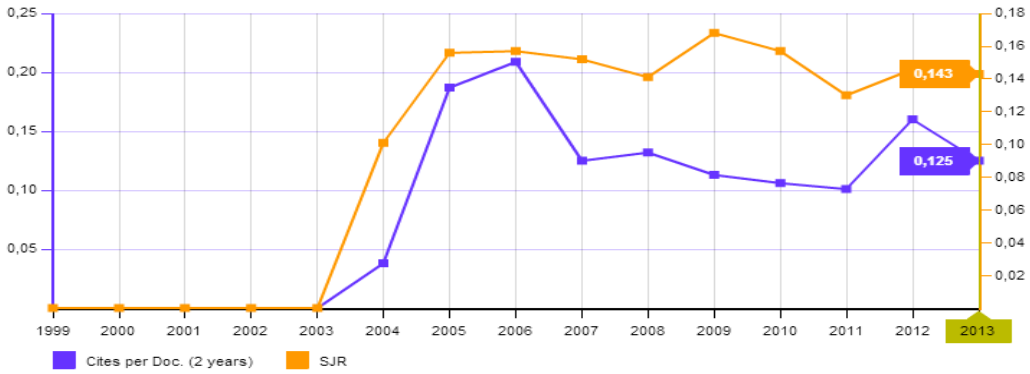

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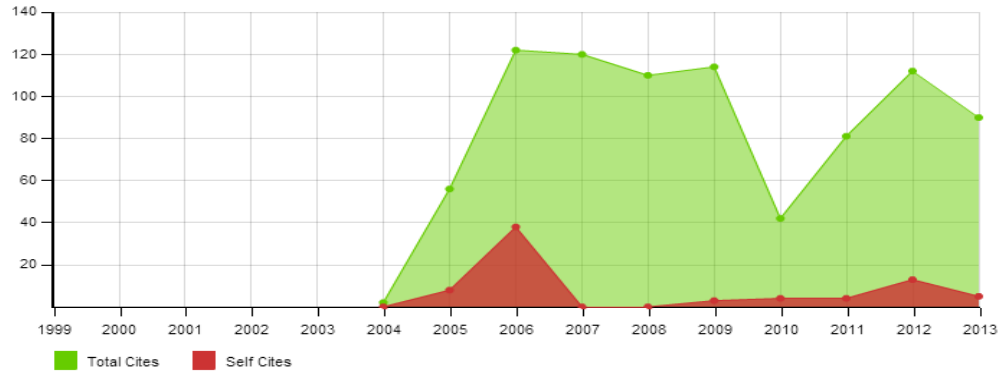

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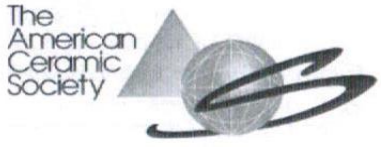

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Contents

Preface ix

CERAMIC MATRIX COMPOSITES

Development of Continuous SiC Fiber Reinforced HfB_2-SiC Composites for Aerospace Applications 3
Clifford J. Leslie, Emmanuel E. Boakye, Kristin A. Keller, and Michael K. Cinibulk

Effect of Primary Grain Size of $SrZrO_3/ZrO_2$ Nano-Dispersed Composite Abrasive on Glass Polishing Properties 13
Takayuki Honma, Koichi Kawahara, Seiichi Suda, and Masasuke Takata

Thermal Effect Studies on Flexural Strength of $SiC_f/C/SiC$ Composites for Typical Aero Engine Application 21
Vijay Petley, Shweta Verma, Shankar, S.N. Ashritha, S. N. Narendra Babu, and S. Ramachandra

Effect of Phase Architecture on the Thermal Expansion Behavior of Interpenetrating Metal/Ceramic Composites 33
Siddhartha Roy, Pascal Albrecht, Lars Przybilla, Kay André Weidenmann, Martin Heilmaier, and Alexander Wanner

High Temperature Interactions in Platinum/Alumina System 45
Ali Karbasi, Ali Hadjikhani, Rostislav Hrubiak, Andriy Durygin, and Kinzy Jones

Fracture Mechanics of Recycled PET-Based Composite Materials Reinforced with Zinc Particles 55
Jessica J. Osorio-Ramos, Elizabeth Refugio- García, Víctor Cortés-Suarez, and Enrique Rocha-Rangel

INNOVATIVE PROCESSING

Fabrication of GaSb Optical Fibers 65
Brian L. Scott and Gary R. Pickrell

Characterization and Synthesis of Samarium-Doped Ceria Solid Solutions 71
Aliye Arabacı

Influence of Precursors Stoichiometry on SHS Synthesis of Ti_3AlC_2 Powders 79
L. Chlubny and J. Lis

Chemical Vapor Deposition and Characterization of Thick Silicon Carbide Tubes for Nuclear Applications 87
P. Drieux, G. Chollon, A. Allemand, and S. Jacques

Uniform Microwave Plasma Pyrolysis for the Production of Metastable Nanomaterials 99
Kamal Hadidi, Makhlouf Redjdal, Eric H. Jordan, Olivia A. Graeve, and Colby M. Brunet

Characterization of the Conductive Layer Formed During µ-Electric Discharge Machining of Non-Conductive Ceramics 105
Nirdesh Ojha, Tim Hösel, Claas Müller, and Holger Reinecke

Forming Mullite-Ceramics Reinforced with ZrO_2-t Starting from Mullite-ZrO_2-t and Kyanite-Al_2O_3-ZrO_2-t Mixtures 111
Elizabeth Refugio-García, Jessica Osorio-Ramos, José G. Miranda-Hernández, José A. Rodríguez-García, and Enrique Rocha-Rangel

Impact of Nanoparticle-Microstructure on Cosmeceuticals UV Protection, Transparency and Good Texture 119
Yasumasa Takao

Piezoelectric Thick-Film Structures for High-Frequency Applications Prepared by Electrophoretic Deposition 131
Danjela Kuscer, Andre-Pierre Abelard, Marija Kosec, and Franck Levassort

Low Temperature Growth of Oxide Thin Films by Photo-Induced Chemical Solution Deposition 143
Tetsuo Tsuchiya, Tomohiko Nakajima, and Kentaro Shinoda

The Effect of Active Species during TiN Thin Film Deposition by the Cathodic Cage Plasma Process 149
Natália de Freitas Daudt, Júlio César Pereira Barbosa, Danilo Cavalcante Braz, Marina de Oliveira Cardoso Macêdo, Marcelo Barbalho Pereira, and Clodomiro Alves Junior

FORMING MULLITE-CERAMICS REINFORCED WITH ZrO_2-t STARTING FROM MULLITE-ZrO_2-t AND KYANITE-Al_2O_3-ZrO_2-t MIXTURES

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ABSTRACT

Mullite-ceramics reinforced with 8 and 10 vol. % YZT were prepared starting from two different powder mixtures. The first one was a mixture of pure mullite and YZT, and the other was a mixture of kyanite, alumina and YZT. The production of ceramics consisted of the pressureless-sintering of mixtures powders which were thoroughly mixed under high energy ball-milling. During sintering, kyanite and alumina of the second mixture react between them to form mullite. Measurements of density, microhardness and K_{IC} were carry out in all produced materials, from it was obtained that samples produced with pure mullite reach major densities, microhardness and toughness in comparison with the values displayed for samples prepared with kyanite and alumina. The reason of this has its explanation since during sintering of the kyanite and alumina samples; there is a competition between two phenomena; the reaction to form mullite and the sintering of the product. As the reaction occurs at low temperature and has high energy consumption, the activation energy necessary for diffusion during sintering is not reached; therefore this phenomenon is the controlling agent during the processing.

INTRODUCTION

Besides its traditional uses, mullite has attracted attention in recent years, as a material for high temperature structural applications, mainly because at these temperatures it may retain a significant portion of the mechanical strength that it has at room temperature, also, because it has a low thermal expansion coefficient, low dielectric constant, high melting point, high creep resistance and high chemical stability[1]. Table 1 shows some of the values of the main properties of the mullite.

Table 1. Main properties of pure mullite at room temperature[1].

Property	Value
Thermal expansión coefficient	$5X10^{-6}$ K^{-1}
Flexural resistance	200 MPa
Fracture toghness (K_{IC})	2 $MPam^{1/2}$
Young modulus	231 MPa
Density	3.16 gcm^{-3}

As can be seen in this table, fracture toughness (K_{IC}) is a characteristic in which mullite is deficient. Furthermore, to obtain dense bodies of mullite, it is required long sintering treatment at elevated temperatures (>1700 °C)[2], this is due to the high value of the activation energy necessary for ion diffusion occurs through the network of mullite[3]. Because of these difficulties, over the past 15 years in many countries around the world, they were conducted a series of investigations in the seek for new processing methods by which they could obtain dense mullite bodies. To increase its tenacity, in some of these studies, it has proposed the use of ZrO_2 as reinforcing material[4-10], thus values of K_{IC} that have been obtained, varying between 3 and 3.2 $MPa \cdot m^{1/2}$ for 10 to 20 wt. % ZrO_2 content.

Some researchers[6] have mentioned that large amounts of ZrO_2 on mullite-based composites cause thickening of the microstructure and thus the difficulty in the retention of the tetragonal form of ZrO_2. This retention of the ZrO_2 is important, because it has been suggested that one method by which the mullite matrix is reinforcing by ZrO_2 is the transformation of ZrO_2-tetragonal to ZrO_2-monoclinic[11]. However, other authors have suggested different possible reinforcement mechanisms, such as microcracking induced by the same transformation of the ZrO_2[9-11], strengthening grain boundaries caused by a metastable solid solution of ZrO_2[10-12] and deflection of cracks due to the presence of acicular microstructure[13].

The aim of this work is to study the formation of mullite composites reinforced with ZrO_2-t, starting from mixtures of mullite + ZrO_2-t and Kyanite + Al_2O_3 + ZrO_2-t, using conventional methods of sintering without the application of pressure in an electric furnace.

Because pure millita is a highly expensive mineral, it is suggest the use of kyanite as mullite precursor material. Kyanite mineral is cheaper than the mullite mineral, with chemical formula ($Al_2O_3 \cdot SiO_2$), this mineral when is heated at high temperatures (~ 1300 ° C) decomposes forming mullite + silica, for this reason in the mixture with kyanite is added certain amount of alumina to compensate silica excess resulting from the decomposition of kyanite in order to form more mullite.

EXPERIMENTAL PROCEDURE

Starting materials were: Mullite powder (99%, 1μm, Virginia Milling Corporation, USA), Kyanite powder (99%, 3-5 μm, Virginia Milling Corporation, USA), Al_2O_3 powder (99.9 %, 1μm, Sigma, USA) and YZT powder (99.9 %, <1 μm, Tosho, Japan). Final YZT contents in the produced composites were: 8 or 10 vol. %. Powder blends of 20 g were prepared in a ball mill with ZrO_2 media, the rotation speed of the mill was of 300 rpm, and the studied milling time was 12 h. With the milled powder mixture, green cylindrical compacts 2 cm diameter and 0.2 cm thickness were fabricated by uniaxial pressing, using 300 MPa pressure. Then pressureless sintering in an electrical furnace was performed, at 1500 °C during 2 h. Characterization of sintered samples was as follows: Densities were measured using the Archimedes' principle. The microstructure was observed by scanning electron microscopy (SEM), equipped with an energy dispersive spectroscopy analyzer (EDS). Phases present in the sintered composites were determined by X-ray diffraction (XRD). Fracture toughness was estimated by the fracture indentation method[14], (in all cases ten independent measurements per value were carrying out).

To identify study samples, it was used the following code;
C8Z: Sample prepared with Kyanite + Al_2O_3 + 8 % vol. ZrO_2
C10Z: Sample prepared with Kyanite + Al_2O_3 + 10 % vol. ZrO_2
M8Z: Sample prepared with Mullite + 8 % vol. ZrO_2
M10Z: Sample prepared with Mullite + 10 % vol. ZrO_2

Table I. Summary of the dimensions of the notches in the specimen under study.				
Samples	Aperture [mm]	Depth [a] [mm]	Angle [α] [degrees]	Radius [r] [mm]
PEZI 0a	1.742	1.869	42 45	0.214
PEZI 0b	3.117	3.587	39 46	0.224
PEZI 10a	2.575	2.806	45 22	0.209
PEZI 10b	2.226	2.322	44 31	0.295
PEZI 10c	2.132	2.359	41 38	0.201
PEZI 30a	3.127	3.703	43 00	0.240
PEZI 30b	3.336	3.830	42 39	0.295
PEZI 30c	2.639	2.742	46 26	0.197

Bending test.

To apply the theoretical models of Peterson, Irwin and Griffith, it was first necessary to test the 8 specimens, previously prepared and tested in three-point bending to find that maximum force could with stand the test pieces before breaking. This test was conducted in the Instron universal machine, mod. 5500R, using a rate of 0.5 mm / min.

Thereafter, defining the encoding of the specimens based on their geometry, which corresponds to a flexure beam as shown in Figure 2[8].

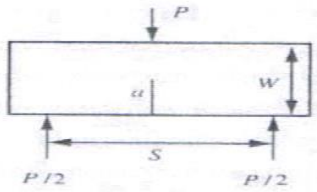

Figure 2. Geometry of a beam in bending.

Where:
W: the height of the sample [m]
P: is the applied force [N]
S: is the spam formed between the support [m]
a: is the depth of the crack [m]
Besides using other values are also useful.
B: thickness of the sample [m]
r: the radius [m]
d = W - a [m]

Thus using this coding will be able to obtain other parameters in order to find the value of K_{IC}.

Using the model of Peterson it is possible to get K_{tn}, σ_{max} and σ_{nom}.

Where:
K_{tn}: The factor of elastic stress concentration
σ_{max}: The effort at the root hub of efforts
σ_{nom}: The nominal stress

In the concentration stress charts developed by Peterson[9] it can be found the value of K_{tn}, relating r with d and W with d. However, also it can be obtained applying the equations of the curves and formulas mentioned below[9].

$$K_{tn} = C_1 + C_2(a/W) + C_3(a/W)^2 + C_4(a/W)^3 \quad \text{---------- (1)}$$

In the case where $2.0 < a/r < 20$, the following equations are applied.

ray diffraction patterns do not indicate complete reaction, since there are several peaks correspond to Al_2O_3 and SiO_2 in both cases, however these peaks are very low in intensity, therefore reaction is almost complete. The most important observation here is the presence of ZrO_2 in its monoclinc form, and this situation could be a problem, because the reinforcement by transformation of zirconia would not operate in this situation.

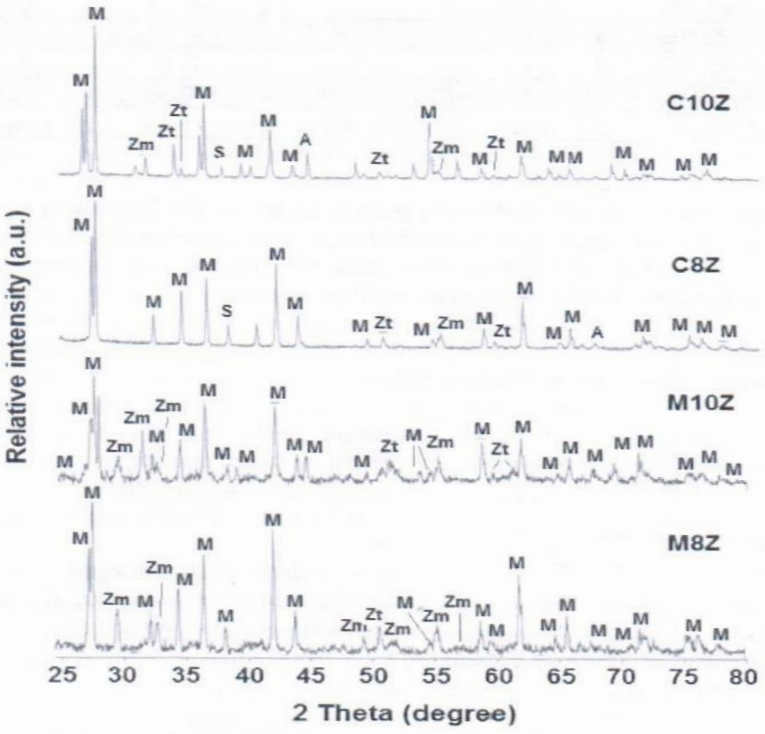

Figure 3. X-ray diffraction patterns of different prepared composites.
M-mullite, S-silica, A-alumina, Zt-tetragonal- ZrO_2, Zm-monoclinic- ZrO_2.

Microhardness

The microhardness evaluated in different prepared samples is reported in Figure 4. In this figure it is seen that those samples prepared with mullite + ZrO_2-t exhibit higher hardness values, compared to the samples prepared with the mixture of kyanite + Al_2O_3. The greater degree of densification and the situation of has a more homogeneous material in chemical composition in samples prepared with ZrO_2 and mullite, are the cause responsible for this condition. Since materials made from kyanite besides being less dense, have a more heterogeneous chemical composition, as have been indicated by XRD results, where phases such as mullite, monoclinic and tetragonal ZrO_2, Al_2O_3 and SiO_2 are present.

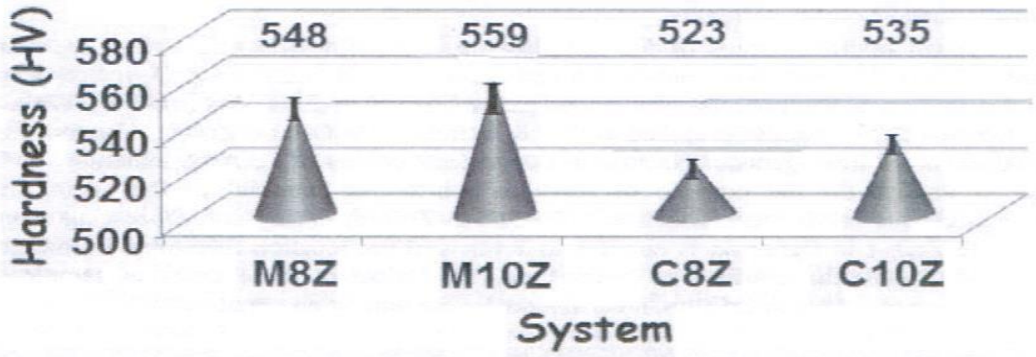

Figure 4. Hardness of samples sintered at 1500°C, during 2 h.

Fracture toughness

Figure 5 shows the values of fracture toughness determined in different samples prepared here. As expected samples prepared with mullite + ZrO_2-t exhibit larger values of fracture toughness because therein ZrO_2 is present in tetragonal form, giving the feasibility of the reinforcement by the transformation of ZrO_2-t to ZrO_2-m exist. On the contrary, the fracture toughness is less in the samples prepared with kyanite + Al_2O_3 + ZrO_2-t, because it was not possible the ZrO_2-t retention at the end of processing.The reason of these behaviors have its explanation since during sintering of the kyanite and alumina samples; there is a competition between two phenomena; the reaction to form mullite and the sintering of the product. As the reaction occurs at low temperature and has high energy consumption, the activation energy necessary for ion diffusion through the network of mullite during sintering is not reached; as a result this phenomenon is the controlling agent during the processing.

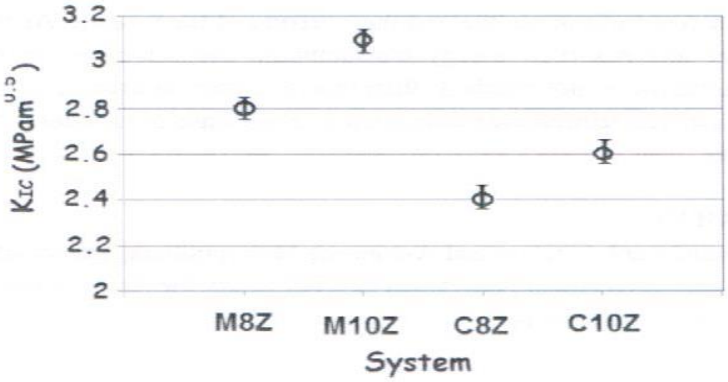

Figure 5. Fracture toughness of samples sintered at 1500°C, during 2 h.

Microstructure

Typical microstructures of the samples prepared with 10% vol. ZrO_2-t are shown in Figure 6. Figure 6a which corresponds to sample prepared with mullite + ZrO_2-t, clearly shows a good distribution of fine particles of a second phase (ZrO_2-t) in the matrix (mullite). While figure 6b corresponds to a sample prepared with the kyanite + Al_2O_3 and ZrO_2-t mixture, the ZrO_2 distribution is not homogeneous because it is appreciate colony of ZrO_2 agglomerates. ZrO_2 was added to mullite for the purpose of serve as reinforcing material of the same, situation unsuccessful, due to the transformation of ZrO_2-t to ZrO_2-m during any processing stage. The size of the ZrO_2-t in figure 6a is about 1 to 3 μm and the location thereof is in intergranular regions of the mullite matrix. The reinforcing mechanism in these kinds of samples is the transformation of ZrO_2-t to ZrO_2-m, mechanisms before widely documented[2,4,6,7,8,11].

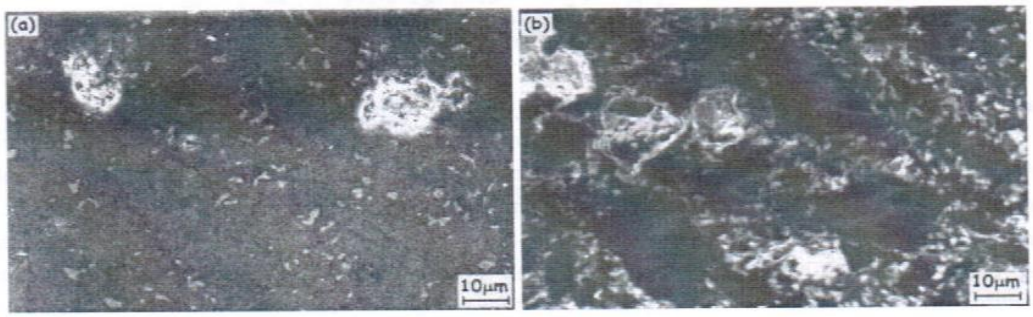

Figure 6. Microstructure observed in scanning electron microscope of samples sintered at 1500°C, during 2 h. (a) mullite + ZrO_2-t, (b) kyanite + Al_2O_3 + ZrO_2-t.

CONCLUSIONS

Samples produced with pure mullie reach major values of density, microhardness and toughness. During sintering of kyanite-alumina mixtures; there is a competition between two phenomena; the reaction to form mullite and the sintering of the product. As the reaction occurs at low temperature and has high energy consumption, the activation energy necessary for diffusion during sintering is not reached; therefore this phenomenon is the controlling agent during the processing. The reinforcing mechanism in these kinds of samples is the transformation of ZrO_2-t to ZrO_2-m.

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