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5. Fracture Toughness Enhancement of Mullite-Ceramics Reinforced with Metals

J.P. Singh, Narottam P. Bansal, Amar S. Bhalla, Morsi M. Mahmoud, Navin Jose Manjooran, Gurpreet Singh, Jacques Lamon, Sung R. Choi, Gary Pickrell, Kathy Lu, Geoff Brennecka andTakashi Goto

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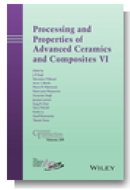

Processing and Properties of Advanced Ceramics and Composites VI: Ceramic Transactions, Volume 249

Published Online: 3 OCT 2014

DOI: 10.1002/9781118995433.ch5

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Refugio-García, E., Hernández, J. G. M., Rodríguez-García, J. A. and Rocha-Rangel, E. (2014) Fracture Toughness Enhancement of Mullite-Ceramics Reinforced with Metals, in Processing and Properties of Advanced Ceramics and Composites VI: Ceramic Transactions, Volume 249 (eds J.P. Singh, N. P. Bansal, A. S. Bhalla, M. M. Mahmoud, N. J. Manjooran, G. Singh, J. Lamon, S. R. Choi, G. Pickrell, K. Lu, G. Brennecka and T. Goto), John Wiley & Sons, Inc., Hoboken, NJ, USA. doi: 10.1002/9781118995433.ch5

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Publication History

Published Online: 3 OCT 2014
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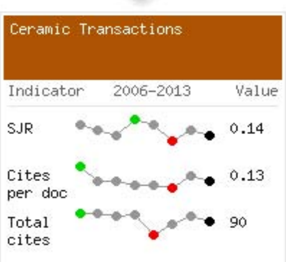

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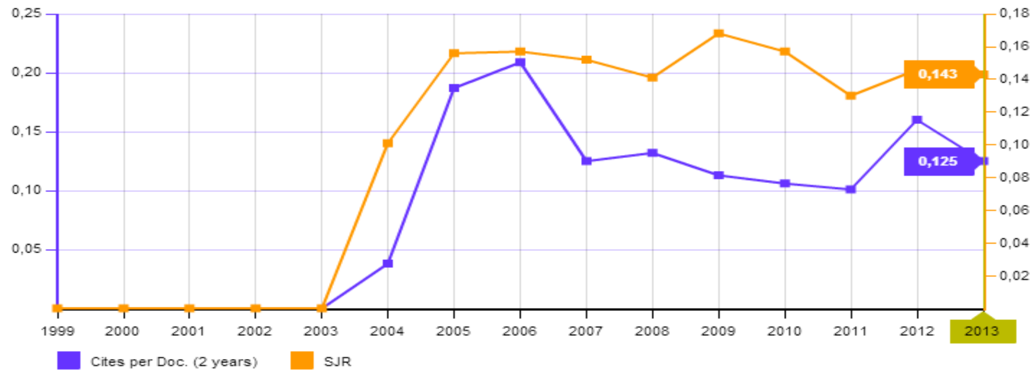

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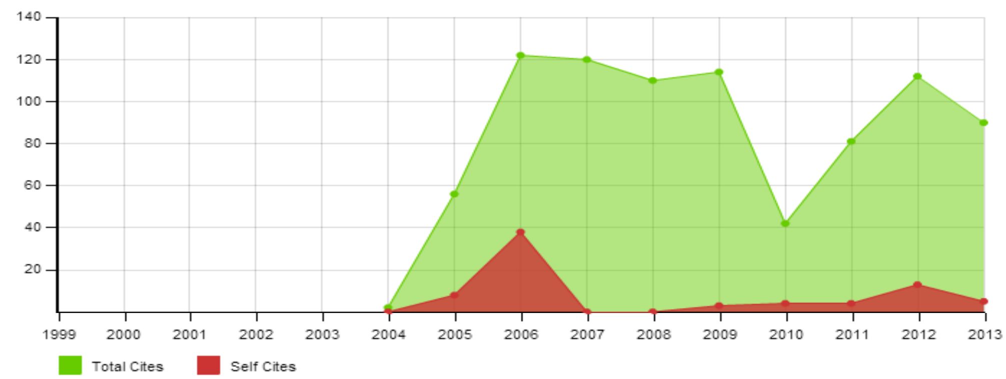

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The American Ceramic Society

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Library of Congress Cataloging-in-Publication Data is available.

ISBN: 978-1-118-99549-5
ISSN: 1042-1122

Printed in the United States of America.

10 9 8 7 6 5 4 3 2 1

Contents

FRACTURE TOUGHNESS ENHANCEMENT OF MULLITE-CERAMICS REINFORCED WITH METALS

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ABSTRACT

With the idea to determine ways of tailoring mullite-ceramics ($3Al_2O_3 \cdot 2SiO_2$) in order that one or more toughening mechanisms are activated in service, investigations about the production of mullite-based composites with different reinforcement metals (Co, Ni, Ti) have been carried out. The synthesis of composites materials has been made by means of pressure less sintering of an intensive mechanical mixture of powders. With the use by separate of those metals in the chemical formulations, significant improvements in ceramic toughness have been obtained. From the fracture toughness measurements and microstructural observations, it can be commented that the toughening mechanism in the mullite/metal reinforced composites is due to crack bridging and crack deflection, owed to the presence of ductile particles in the ceramic matrix. On the other hand, the presence of metals in the composites helps densification of mullite.

INTRODUCTION

In all its applications, ceramic materials are valued for their ability to resist heat and chemical attack, as well as its high hardness. These virtues are due to the strong links that keep the constituent atoms in their positions of balance[1-3]. The nature of these links also adds a critical drawback which is the fragility, causing that ceramics are particularly sensitive to the presence of minimal imperfections in its microstructure, defects that act as points of cracks initiation[2]. Therefore, many ceramic research efforts have been devoted to develop new processes that minimize these microscopic defects; and it has insisted on the design of new compositions and microstructures that prevent the growth of cracks, where they have result that the combinations between ceramic composites and metals present good mechanical properties[4-9].

Mullite is a cheap refractory ceramic with a nominal composition of $3Al_2O_3 \cdot 2SiO_2$. The raw materials for produce mullite are easily obtainable and are reasonably priced. It has excellent high temperature properties with improved thermal shock and thermal stress resistance owing to the low thermal expansion, good strength and interlocking grain structure[2]. Values of the main properties of the mullite are shown in Table 1.

Table 1. Main properties of pure mullite at room temperature[10].

Property	Value
Thermal expansion coefficient	$5x10^{-6}K^{-1}$
Flexural resistance	200MPa
Fracture toughness (K_{IC})	$2MPam^{-1/2}$
Young modulus	231MPa
Density	$3.16gcm^{-3}$

As seen in this table, a characteristic in which mullite is deficient is its low fracture toughness (K_{IC}). Furthermore, to obtain dense bodies of mullite, it is required long sintering treatment at temperatures above 1700°C[10], this is due to the high value of the activation energy necessary for ion diffusion occurs through the network of mullite[10]. Therefore, as all ceramics, mullite presents high sensibility to minimal imperfection in its structure, being the main reason for its high fragility. For these reasons, they have been sought alternative methods of production and strengthening of mullite, varying reinforcement materials, the manufacturing process, sintering treatments and even the use of sintering additives that improve the mechanical properties principally fracture toughness[11]. The objective of this work is to manufacture mullite-based composites reinforced with different metals such as: Titanium, nickel and cobalt, in order to enhance their fracture toughness. This through a combination of techniques: mechanical grinding-pressing cold and sintering without pressure.

EXPERIMENTAL

Raw materials for the production of composites were: Mullite powders (99.9 %, 1 μm, Sigma-Aldrich, USA), titanium, cobalt and nickel powders (99.9 % purity, 1-2 μm, Sigma-Aldrich, USA). The amount of metallic powders used to obtain the desire composite was that allows at the end of the processing let to obtain a Mullite-10 vol. % Me, where Me represents the corresponding metal used as a reinforcement. The powders were milled and dry mixed in a horizontal mill (Cole-Parmer jar mill, 115 VAC/60 Hz, USA), using a rotation speed of 300 rpm, during 12 hours, with the help of ceramic jars and using YSZ's balls as grinding elements, the ratio; weight of balls/weight of powder was 20:1. Cylindrical samples were fabricated by uniaxial pressing with the milled powders, using 300MPa. Dimension of samples were: 20 mm diameter and 3 mm thickness (Montequipo 10,000 Ton. México). Afterwards, samples were pressureless sintered at 1400°C during 2 hour in an electrical furnace with argon atmosphere (Carbolite RHF 16/3, 1600°C, UK). The rates of heating and cooling were kept constant and equal to $10°Cmin^{-1}$. The characterization of the synthesized products was as follows: density was evaluated by the Archimedes' principle[12], microhardness measurements were evaluated with the help of a Vickers indenter (Mitutoyo, MVK-H2 H3 HM114, Japan), using a load of 1Kgf with penetrations of 15s. Fracture toughness measurements were carried out by the fracture indentation method[13-15]. The microstructure of the composites was observed with an optical microscope (Nikon, Eclipse MA 100, Japan). Finally, phases present in the composites materials were determine by X-ray diffraction (Philips, D-5000, Germany).

RESULTS

Density

The results of density measurements, evaluated by the Archimedes' principle are shown in Figure 1. In this figure it can see that in all three cases in which the mullite was reinforced with a metal, got a degree of densification superior compared to pure sample. This can be due to the fact that having metal particles homogeneously distributed in the mullite ceramic matrix, the conduction of heat in the samples is best, favoring transport of material during sintering of samples, which is reflected in samples with higher densification. Specimens reinforced with titanium and nickel reached relative densities of 95% and 94% respectively, while the sample with cobalt reached 91% of densification. Furthermore, pure mullite densified slightly less than 86%.

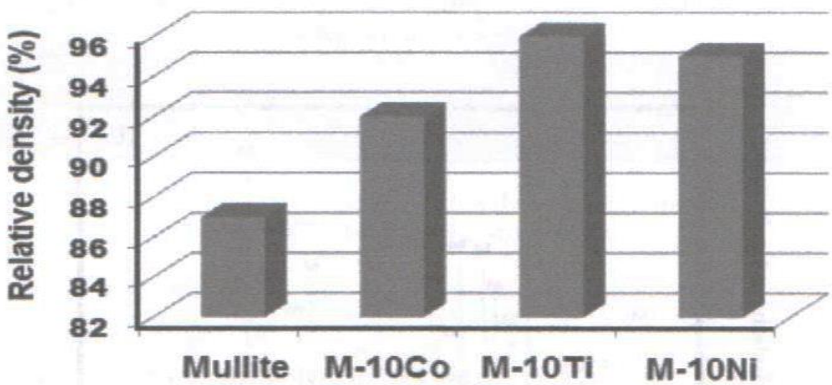

Figure 1. Relative density of the sintered samples at 1400°C during 1 hour.

X-ray diffraction

Results of X-rays analysis diffraction of the samples are showed in Figure 2. In these figures for each case clearly shows the presence of intense peaks of mullite. Meanwhile, that also clearly visible in each diffraction pattern the presence of the corresponding reinforcing metal used; as is the case of titanium in Figure 2a, nickel in Figure 2b and cobalt in Figure 2c. In these three diffraction patterns it is not observed the presence of any other crystalline component in the samples, as would be any metal oxide used, which may have formed during the sintering, which indicates that the argon atmosphere used for inhibiting the oxidation of metals complied well with its function, of these diffraction patterns is determined that neither one has any contamination in samples, that may come from any of the processing steps, particularly the grinding.

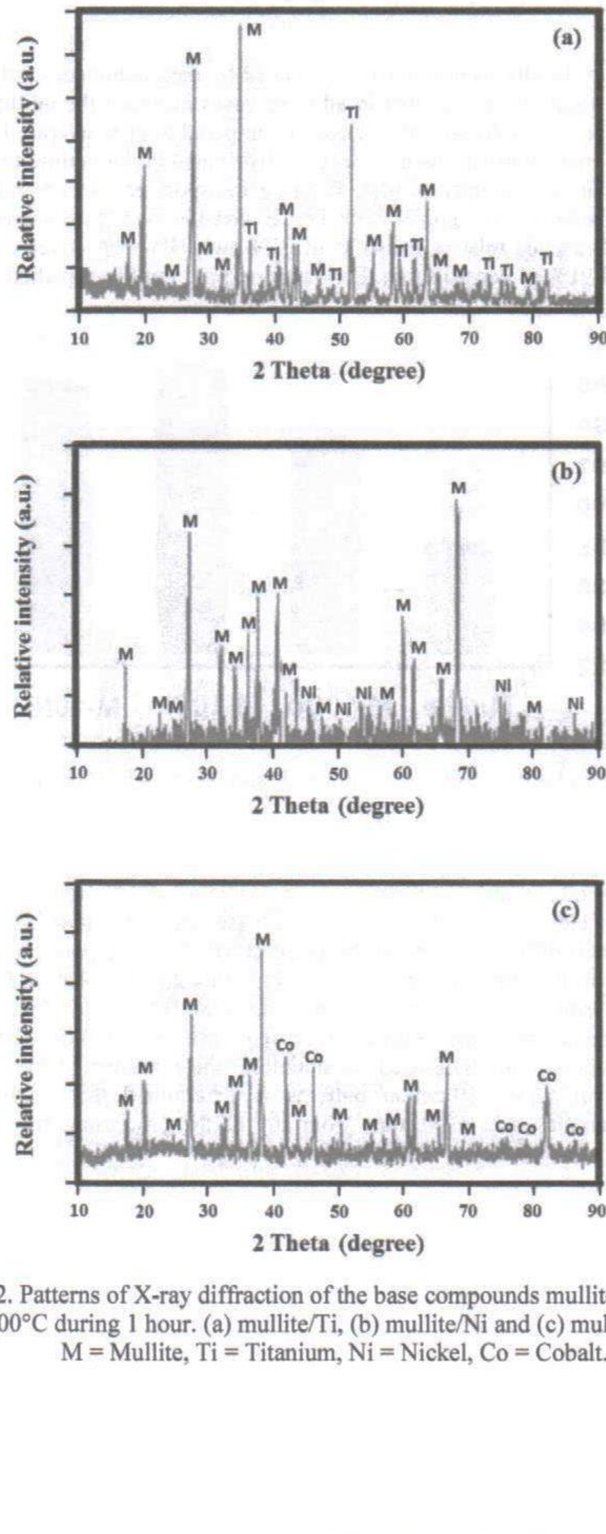

Figure 2. Patterns of X-ray diffraction of the base compounds mullite sintered at 1400°C during 1 hour. (a) mullite/Ti, (b) mullite/Ni and (c) mullite/Co M = Mullite, Ti = Titanium, Ni = Nickel, Co = Cobalt.

Microstructure

Figure 3 shows micrographs taken with an optical microscope in the three study materials. In these micrographs, are observed two phases, the opaque corresponds to the ceramic matrix (mullite) whereas the clear and bright phase corresponds to the reinforced metal used in each case. In samples with nickel and cobalt they are observed the formation of porosity, while in the sample reinforced with titanium porosity is not observed. In each case there is a good and homogeneous distribution of the metallic phase; even in the case of the titanium sample, metal distribution is better than in the other two cases.

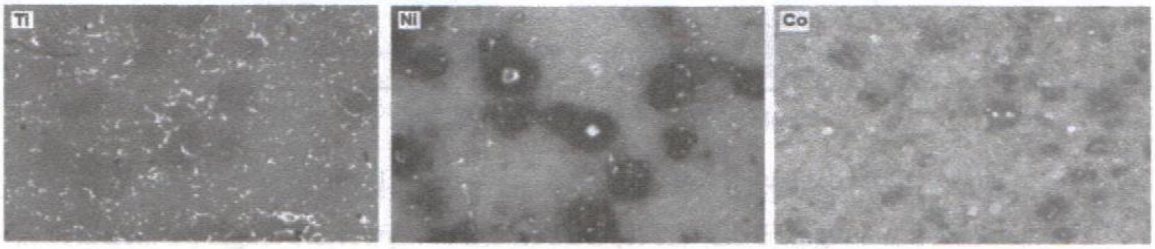

Figure 3. Optical micrographs of the three studied materials.
Mullite/Ti, Mullite/Ni, Mullite/Co.

Mechanical properties (HV and K_{IC})

Measurements results of micro hardness (HV) and fracture toughness (K_{IC}) evaluated in studied materials are presented in Table 2. The sample reinforced with titanium presents greater hardness, the values of micro hardness of the other two samples have similar values for magnitude, although the sample with additions of cobalt is a little harder than that with nickel. Pure mullite hardness value is above these last two, and bit below the sample with titanium. A clear effect of the metals used as reinforcement on the hardness of mullite is shown in Figure 4. Where apparently when they were used titanium or cobalt are not significant differences in the hardness of mullite. However, when nickel is added to this the same hardness decreases considerably.

Table 2. Relative density, hardness and fracture toughness of study materials.

Sample	**Relative density (%)**	**Hardness (GPa)**	**K_{IC} ($MPam^{-1/2}$)**
Pure mullite	86	9.75 +/- 0.24	1.51 +/- 0.23
Mullite/10% vol. Ti	95	9.92 +/- 0.17	3.27 +/- 0.25
Mullite/10% vol. Ni	94	8.95 +/- 0.22	2.12 +/- 0.21
Mullite/10% vol. Co	91	9.56 +/- 0.21	2.43 +/- 0.27

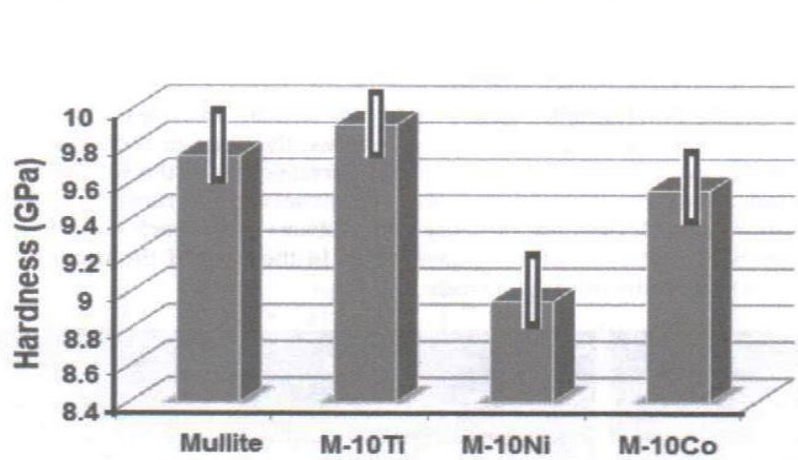

Figure 4. Effect of metals reinforcement on the micro hardness of mullite.

In the Table 2 and Figure 5 they are reported results of the fracture toughness measured in the same samples. Figure 5 shows that the mullite fracture toughness was just 1.51MPam$^{-1/2}$, well below the theoretical value of 2MPam$^{-1/2}$ reported for the same in the literature[10], the main reason for this difference is the low density of the processed samples of mullite here. However, when mullite is reinforced with a metal such as titanium their fracture toughness increases up to 3.27MPam$^{-1/2}$, which is equivalent to a 100% improvement in this property. On the other hand, when mullite is reinforced with nickel or cobalt their fracture toughness increases to 2.12 and 2.43MPam$^{-1/2}$ respectively, equivalent to 33% and 61%, being very significant improvements in this mechanical property improvements. This situation may be due to several factors, the first is the degree of densification reached by samples when a metal is added in them. The second would be the good distribution of the metallic particles in the ceramic matrix. A third is surely good adhesion between the metals used and the ceramic mullite. Altogether, these factors significantly improve the toughness of mullite. Different authors have reported that the mechanism of reinforcement in ceramic/metal composites is because of crack bridging and crack deflection caused by the reinforcing metal particles[4, 10].

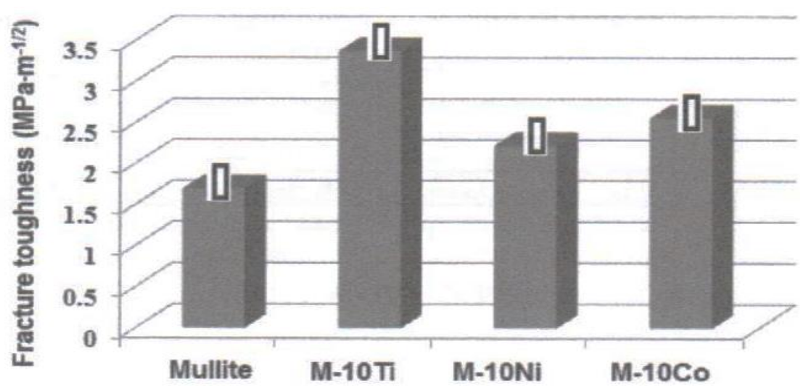

Figure 5. Effect of metals reinforcement on the fracture toughness of mullite.

CONCLUSIONS

- Through the proposed methodology, is possible the manufacture of mullite-based composites reinforced with metal particles.
- X-ray diffraction patterns indicate the presence of two crystalline phases in the three studied cases, where mullite is the main component.
- The mullite composite reinforced with titanium particles presents best degree of densification and therefore greater hardness and fracture toughness.
- In the three case studies the fracture toughness of mullite was considerably improved, the mechanism of strengthening is due to crack bridging and crack deflection

Acknowledgments. The materials processing laboratories facilities given by UAM-A, UAEM and UPV is appreciated. Authors wish to thank to the Department of Materials of UAM-A for the financial support given through project 2260235.

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