Preface.

Introduction.


Solvothermal Synthesis of Gadolinium Hydroxide and Oxide Powders and Their Potential for Biomedical Applications (Eva Hemmer, Yvonne Kohl, Sanjay Mathur, Hagen Thielecke, and Kohei Soga).


Effect of Nano-Silica on Acid Resistance Properties of Enamel and Its Connection to Energy Saving (Majid Jafari and Javad Sarraf).

Immobilization of Myoglobin with Regenerated Silk Fibroin/MWCNTs on Screen-Printed Electrode: Direct Electrochemistry and Electrocatalysis of H2O2 (Lei Zhang, Lei Shi, Wei Song, and Yi-Tao Long).

Liquid Phase Patterning and Morphology Control of Metal Oxides (Yoshitake Masuda).

Role of Nano-Structured Domain Derived from Organically Modified Silicate in Electrocatalysis (P. C. Pandey, D. S. Chauhan, and V. Singh).


Bioactive Glass-Ceramic/Mesoporous Silica Composite Scaffolds for Bone Grafting and Drug Release (Enrica Verné, Francesco Baino, Marta Miola, Giorgia Novajra, Renato Mortera, Barbara Onida, Chiara Vitale-Brovarone).


Synthesis of PbTe Nanowires with Enhanced Seebeck Coefficient (Wenwen Zhou, Hao Cheng, Aidong Li, Huey Hoon Hng, Jan Ma, and Qingyu Yan).
Dear Dr. Rocha-Rangel:

It is a pleasure to accept your manuscript entitled "Preparation and Their Mechanical Properties of Al2O3/Ti Composite Materials" in its current form for publication in the 8th Pacific Rim Conference on Ceramics and Glass Technology. The comments (if any) of the reviewer who reviewed your manuscript are included at the foot of this letter.

If you have not already done so, please complete and send the copyright transfer form to Greg Geiger at ggeiger@ceramics.org or fax 614/899-6109.

Thank you for your fine contribution. On behalf of the Editors of the 8th Pacific Rim Conference on Ceramics and Glass Technology, we look forward to your continued contributions to the proceedings.

Sincerely,

Prof. Sanjay Mathur
Symposium Chair, 8th Pacific Rim Conference on Ceramics and Glass Technology
sanjay.mathur@uni-koeln.de

Date Sent: 16-Aug-2009

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Preparation and their Mechanical Properties of Al₂O₃/Ti Composite Materials

Enrique Rocha-Rangel, Elizabeth Refugio-García, José G. Miranda-Hernández, and Eduardo Térres-Rojas
Departamento de Ing. Metalúrgica, ESQUIE-IPN, UPALM, Av. IPN s/n, San Pedro Zacatenco, México, D. F. 07738, Departamento de Materiales, Universidad Autónoma Metropolitana Av. San Pablo # 180, Col Reynosa-Tamaulipas, México, D. F., 02200, Laboratorio de Microscopía Electrónica de Ultra Alta Resolución, Instituto Mexicano del Petróleo, Eje Central Lazara Cárdenas # 152, Col San Bartola Atepehuacan, México, D. F., 07730

Abstract

The effect of different titanium additions (0.5, 1, 2 and 3 wt %) and the milling intensity (4 and 8 h) in a Symoloyer mil on the microstructure and fracture toughness of Al₂O₃-based composite materials is analyzed in this work. After high energy milling of Al₂O₃-Ti powder mixtures, it was obtained small particles with main size of about 150 nanometers. The microstructures observed by SEM present the formation of a small and fine metallic net inside the ceramic matrix. From fracture toughness measurements made by the indentation fracture method, it was observed that when the concentration of titanium increases, the fracture toughness of the composites also increases. Enhance in the fracture toughness is due to the bridging formation by the metal in the ceramic matrix. On the other hand, with milling times of 8 h combined with sintering temperatures of 1500°C during 1 h, they were obtained well consolidated bodies.

Introduction

In recent times, interpenetrating composites between ceramics and metals (cermets) have been considered to take advantage of the best properties of both phases. High wear resistance is achieved from ceramic/metal microstructures because of the high hardness and high wear resistance of the ceramic fraction in the composite. The metallic fraction increases the fracture toughness of the composite, which improves its damage tolerance. Interpenetrating composites have an advantage over other materials, because the homogeneous distribution of the metal in the ceramic matrix provides dimensional stability at high temperatures.¹⁻² Interpenetrating cermets can be fabricated by a number of techniques such as: direct oxidation of a metal,³ metal infiltration of a ceramic perform,⁴⁻⁵ reactive metal penetration⁶⁻⁷ and by hot pressing.⁸ However, most of these processes are costly, present low productivity and they are complex in their procedures. Therefore, simple and cheaper processes are now in development for the production of high amounts of cermets. High-energy ball milling combined with pressureless sintering can be a substitute low-cost method for the production of cermets, and conventional powder-techniques can be applied for forming and densification. Milling can be performed under vacuum, air or inert atmosphere as well as under dry or wet conditions, using an organic milling agent,⁹ in addition, it
allows for variation of the metallic phase or the ceramic volume fraction in a wide range. In this way, the properties of the composite can be modified to suit the desired purpose.

**Experimental Procedure**

The starting materials were α-Al₂O₃ powder (99.9%, 1μm, Sigma, USA) and Ti powder (99.9%, 1-2μm, Aldrich, USA). The final titanium contents in the composites produced were 0.5, 1, 2 and 3 vol. %. Powder blends of 20g were prepared in high energy mill (simoloyer) with ZrO₂ media, the rotation speed of the mill was of 400 rpm two different milling times were studied (4 and 8 h). With the milled powder mixture, green cylindrical compacts 2 cm diameter and 0.2 cm thickness were fabricated by uniaxial pressing using 270 MPa pressure. Then pressureless sintering was performed under argon flux of 10cm³/min and at two different temperatures (1500 and 1600 °C) during 1 h. Densities of sintered specimens were determined by using Archimedes method. The microstructure was observed by scanning electron microscopy (SEM). The hardness of samples was evaluated as micro-hardness using Vickers indentation, fracture toughness was estimated by the fracture indentation method,¹⁰ (in all cases ten independent measurements per value were carry out).

**Results and Discussion**

**Density**

The results of density measurements in all samples appear in Figure I. Here it is possible to be observed that the effect of the diffusion during the sintering causes the densification of all samples. Nevertheless, if the formation of the liquid phase of titanium is considered for samples sintered at 1600°C. From these values it is considered that some pore formation occur during sintering, the pore formation can be provoked in the sample since the titanium is well known that not wet ceramics [11], due to this there is the presence of some remained porosity in samples. On the other hand, also is observed that the best values of density are reached by samples prepared with powder milled during 8 and sintered at 1500°C.
Hardness

The measured micro-hardness of the samples as a function of titanium content in the composite are reported in Figure 2. In this figure it is seen an unusual behavior, because hardness in all samples with 0.5 % Ti present a strong reduction, however for contents of 1 and 2 % Ti the hardness experiment an increment in their values, finally values of hardness for samples with 3 % Ti exhibit intermediate values. This behavior is difficult to explain because it was waited, a decrement in the hardness of samples with the increments of titanium.
Fracture toughness

The results of mechanical test show that the fracture toughness is enhanced when in the composites’ microstructure there is the presence of a ductile metal. The influence of the milling time and the sintering temperature also are very important in the resulting final fracture toughness values, because samples prepared with powder milled during 8 h and sintered at 1500°C display best values of fracture toughness in comparison with those samples prepared with powder milled during 4 h and sintered at 1600 °C. This can be explained by the finest metallic particle size achieved in the later mentioned samples. Several authors have been documented that the enhance in fracture toughness in cermets may be due to plastic deformation of the metallic phase, which forms crack-bridging ligaments.\(^4, 9, 12\)

![Fracture toughness of sintered cermets as a function of titanium content.](image)

Microstructure

Figure 2 shows scanning electron micrographs of different processed cermets. Suitably refined and homogeneous microstructures are achieved in all samples. The ligament diameter (titanium) ranges from less than 1 μm and it appear be independent of the amount of titanium in the composite. The grain size of alumina ranges from 3 to 12μm and it appears to grow up with the increments of Ti in the composite. In all samples, the alumina-matrix and reinforcing metals were identified with the help of EDS analysis performed during SEM observations. In these pictures it is possible to observe some porosity in the samples.
Conclusions

- **Al₂O₃/Ti cermet**s with interpenetrating microstructures can be manufactured by the combination of high energy milling and pressureless sintering of Al₂O₃ and Ti powders.
- The best process conditions are 8 h high energy milling at 400 rpm and sintering at 1500 °C during 1 h.
- Homogeneous composites with fracture toughness as high as 5.2 MPa·m¹/² for a composite with 3 vol % Ti could be obtained and show better damage tolerance than does monolithic Al₂O₃.
- The refined and homogeneous incorporation of a ductile metal (Ti) in a hard ceramic matrix (Al₂O₃) improves its fracture toughness.
References


