

Preparation of mullite whiskers derived from topaz doped with rare earth oxides for applications in composite materials

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Abstract

Mullite whiskers were obtained by means of the thermal decomposition of natural colorless topaz powder, both pure and doped with 5% by weight of La_2O_3 and/or Y_2O_3 , treated at 1400 °C for 1 h. The analyses by thermogravimetry (TG) and differential scanning calorimetry (DSC) showed that the mullitization temperature of the topaz is 1315 °C; this temperature was reduced to 1298 °C and 1111 °C as a result of doping with Y_2O_3 and La_2O_3 , respectively. The x-ray diffraction (XRD) analysis only exhibited, as an end product from the thermal decomposition of the topaz, mullite when doping with La_2O_3 ; the final product was mullite and alumina and, when doping with Y_2O_3 , it was mullite, alumina and $\text{Y}_2\text{Si}_2\text{O}_7$. In the doped samples, an SEM micrograph proved there to be a reduction in the aspect ratio of the whiskers and the presence of a small residual glassy phase, when compared with the pure mullite. An investigation was conducted with the intention of obtaining whisker development and disaggregation from the ceramic body. The conclusion of this investigation was that the higher porosity in the ceramic body, the better the development and disaggregation of the whiskers is.

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1. Introduction

Whiskers are very fine single crystal materials and, as a consequence of their small dimensions, have a high degree of crystalline perfection that gives them extremely high strengths, near theoretical values and a high modulus of elasticity. The greater the aspect ratio of the whisker, the greater its strength. This means that making its thickness as small as possible should result in fewer cracks, which can spread when subjected to some kind of force [1]. Whiskers can be elastically elongated up to 3% without any permanent deformation, compared with less than 0.1% for the ceramic mass [2]. Whiskers are used as reinforcing elements in composite ceramics so as to improve the mechanical properties and also to prevent the spread of cracks [3,4]. A 15% by volume quantity of whiskers is sufficient to significantly improve the flexural strength and fracture toughness in the ceramic materials [5,6].

Mullite is the single thermodynamically stable phase in the phase diagram of the SiO_2 – Al_2O_3 system, in the range of 60 to 66.7% mol of Al_2O_3 . In this range, the $\text{Al}_2\text{O}_3/\text{SiO}_2$ molar ratio of mullite varies, respectively, from 1.5 to 2, and, as a result of there being limited natural deposits of the material, mullite can only be obtained by way of synthesis [7].

There are several methods to synthesize mullite whiskers, but they all base themselves on two fundamental formation processes. The first process is the forming of a glassy phase, with the alumina dissolving in this glassy phase and precipitating itself in the form of mullite whiskers. The glassy phase is formed due to the impurities that exist in the precursor oxides of alumina and silica that are used during mullite synthesis. It is also possible to add glass-forming oxides in order to achieve the glassy phase. The second process is from gas transport reactions, which is mainly achieved by means of a volatile fluoride compound. Fluoride, at high temperatures, draws the Si and Al atoms from the oxide precursors, transforming them into mullite whiskers [8].

The mullite formation process, by means of oxide precursors, using the high-energy ball grinding process is relatively the easiest process for producing mullite whiskers. High-energy grinding reduces the mullitization temperature by up to 300 °C, when compared with other conventional processes for producing mullite, such as aluminosilicate gels and solid-state reactions [9].

After the whiskers have been formed, regardless of the synthesis process used, the most critical step is the disaggregation of these whiskers from the ceramic body that formed them. Disaggregation is required when the whiskers are intended to be used in a loose, separated form so that they can be mixed into a ceramic or metallic matrix, where they will be used as a reinforcing element. The disaggregation process always begins with a fragmentation of whisker clusters that exist in the ceramic body, using any type of grinding equipment for this action. During this initial step, many whiskers are broken, thereby generating a large volume of fragments and, consequently, substantially reducing the volume of whiskers. Fragment generation is inherent in this process and increases production costs. For this reason, *in situ* growth of whiskers in a ceramic matrix

has been the most widely used technique for producing ceramic composites [10].

In this paper, the thermal decomposition of colorless natural topaz was used as an alternative methodology for producing mullite whiskers. Topaz is an aluminosilicate that is abundant in several Brazilian regions, the chemical formula of which is $\text{Al}_2\text{SiO}_4 (\text{OH}_{1-x}\text{F}_x)_2$ [11,12]. Many studies, described as using topaz to obtain mullite, in fact used fluoro-topaz to synthetically obtain the mullite, in accordance with US patent #4911902 [13]. The fluoro-topaz $\text{Al}_2(\text{SiO}_4)\text{F}_2$ is a fluorinated topaz, in which the OH ions are replaced by fluorine. It is obtained by using a mixture of SiO_2 , Al_2O_3 and AlF_3 , which is heated between 750 and 950 °C. When the fluoro-topaz is heated to a temperature above 1200 °C, mullite whiskers are formed.

Rare earth oxides, when added to aluminosilicates, form a glassy phase [14,15], which promotes nucleation and whisker development. Because of this property, natural colorless topaz powder was mixed with 5% by weight of La_2O_3 or Y_2O_3 , with the purpose of achieving better whisker formation inside the ceramic body. The development, aspect ratio and disaggregation of these whiskers were analyzed and compared with pure mullite whiskers.

Most studies report that the whiskers formed on the outer surface of a sample, while few report the whiskers forming inside this same sample, but almost none discuss how to disaggregate these whiskers. This work involved a study that sought to obtain greater whisker formation in the inside of the samples and the disaggregation of these whiskers for their application in composite materials.

2. Experiment

The 99.99% pure La_2O_3 and Y_2O_3 powder was provided by Vetec Química Fina Ltd. and Sigma-Aldrich do Brasil Ltd., respectively. The colorless topaz was extracted from a mine in the northeastern region of the Brazilian state of Minas Gerais; this material was then subjected to wet autogenous grinding for 73 h in a mill manufactured by NTK Technical Ceramics. The topaz powder particle size analysis was performed in a CILAS particle size analyzer, the diameter (D_{50}) was 3.42 μm . The topaz chemical analysis was performed at the Lakefield Geosol Ltda laboratory and revealed the topaz to be 99.93% pure with a concentration in weight of 54.9% of Al_2O_3 and 32.55% of SiO_2 . The powders were compressed in a uniaxial press (model 3912 from Caver Laboratory Equipment), at a pressure of 41 MPa, forming 1.3 cm diameter pads.

The topaz powder, pure and doped with 5% by weight of La_2O_3 or Y_2O_3 , in its compacted and non-compacted (loosened) form, were treated at 1400 °C for 1 h, in static air, in a tubular furnace, manufactured by INTI – Equipamentos Termoelétricos, with a heating rate of 10 °C/min. In order to analyze the best formation and disaggregation of whiskers in the inside of the samples, the powders' thermal treatment was done using two different methods. In the first method, the powders' treatment was performed without adding starch. In the second method, 40% by weight of wheat starch was added to the powders,

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