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## Phase development during thermal treatment of a fast-setting cordierite-mullite refractory

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### Abstract

Cordierite based materials are widely used in high temperature applications due to their good thermo-mechanical properties and thermal shock resistance. They are generally employed in the kiln furniture (shelves, brackets, bearing plates) for firing ceramic pieces. Because of its low expansion coefficient and dielectric properties, cordierite is also used in advanced ceramics, catalyst supports automotive, industrial waste gas purification and parts subjected to sudden temperature changes. The low intrinsic strength of the cordierite may be compensated by the presence of mullite, forming a composite material of cordierite-mullite, extending its use to somewhat higher temperatures, at the expense of a slight increase in the thermal expansion coefficient. In this work a cordierite-mullite precursor was prepared from a mixture of magnesium oxide, calcined alumina, silica fume and monoaluminum phosphate solution that produces fast setting at room temperature (~20 minutes) and then, by calcination, cordierite-mullite is generated. The evolution of the mineralogical phases was studied from room temperature to 1350 °C by X-ray diffraction, differential thermal analysis and thermogravimetry. A possible sequence of chemical reactions throughout the heat treatment is also proposed. Adding refractory aggregates to this precursor leads to obtaining a fast-setting concrete, suitable for the formation of individual pieces or repair service at moderately high temperatures. A summary of its main properties is also included.

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## 1. Introduction

Cordierite ceramics ( $2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$ ) are generally employed in the kiln furniture (shelves, brackets, bearing plates) for firing ceramic pieces, due to its excellent thermal shock resistance. Because of its low coefficient of expansion and dielectric properties, cordierite is also used in advanced ceramics, catalyst supports automotive, industrial waste gas purification and parts subjected to sudden temperature changes. The low intrinsic strength of cordierite can be somehow compensated by the presence of mullite ( $3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ), forming a composite material of cordierite-mullite, which extends their use to somewhat higher temperatures at the expense of a slight increase in the expansion thermal coefficient. Materials generally high in cordierite are synthesized from mixtures of talc, plastic clays, alumina and water, being formed by casting in plaster molds, by pressing or by extrusion.

Furthermore, the binding properties of various phosphatic materials have been known for about a century with the advent of dental cements. Among the materials with chemical bond, the magnesia-phosphate cements (known as MPC) are of interest in small-scale applications where traditional cement based materials have limitations. Due to the fast setting and high early strength, they have been widely used in rapid repair of damaged concrete structures (roads, bridges, harbors, airfields, industrial flooring, sealing perforations, etc.), where it matters that the downtime be minimized (hours instead of days). Although the cost of the MPC is superior to that of the normal hydraulic cements, often the costs caused by the diversion of traffic or disruption of industrial production, are considerably superior to the same repair. In recent years, its use as material for encapsulation of hazardous waste has also been studied.

Responsible for the setting reaction of MPC at room temperature is an acid-base reaction between magnesia and various phosphate solutions. The reaction product, typically a salt or hydrogel, forms a cementitious matrix in which aggregates are embedded. Setting time (generally ranging from a few minutes to a few hours) can be controlled with the type and reactivity of the magnesia used, with the concentration of the phosphate solution, with magnesia/phosphate ratio and the use of retarders. Ammonium acid phosphates are the binders used in applications at room temperature, see Sharp and Winbow (1989), Popovics et al. (1987), Abdelrazig and Sharp (1988), Cassidy (1977). For the refractory application proposed here, these binders are not appropriate due to the release of ammonia produced during thermal treatment. In the refractory industry, phosphate bond is also used due to the high melting point of some compounds, using compositions that set at room temperature or by heating. Phosphoric acid and monoaluminum phosphate are the most commonly used.

In this work a cordierite-mullite precursor using chemical magnesia-monoaluminum phosphate bond to achieve rapid setting of the material at room temperature was prepared. We studied the evolution of the mineralogical phases and the probable sequence of chemical reactions during heating to 1350 °C, where the desired cordierite-mullite phases are finally obtained. The addition of refractory aggregates to this precursor produces concrete suitable for the conformation of individual pieces or repairs in service for moderately high temperatures.

## 2. Materials and methods

The cordierite based precursor employing the magnesia-phosphate bond was prepared from a mixture of silica fume, calcined alumina and magnesium oxide in the stoichiometric ratio of cordierite ( $\text{SiO}_2$ : 51.4%,  $\text{Al}_2\text{O}_3$ : 34.9% and  $\text{MgO}$ : 13.7%). These solids were added to an aqueous solution of monoaluminum phosphate ( $\text{Al}(\text{H}_2\text{PO}_4)_3$ , MAP 100L, density 1.48 g/cm<sup>3</sup>), pH 1.5 and mixed manually at room temperature. Relations in weight were  $\text{MgO}/(\text{Al}(\text{H}_2\text{PO}_4)_3)=0.86$  and  $\text{water}/\text{solid}=0.49$ . The paste was allowed to set and dry at room temperature (20 °C), in an oven at 110 °C (24 hours) and then it was submitted to 450, 600, 750, 1100 and 1350 °C for 2 hours in an electric furnace. The mixture of solids with the phosphate solution was called P-Al precursor.

The alumina used was calcined type, Alcan S3G with a mean particle size of 5 microns, specific surface determined by the Brunauer-Emmett-Teller or BET method of 1 m<sup>2</sup>/g and 0.49%  $\text{Na}_2\text{O}$ . X-ray diffraction (XRD) patterns showed that alumina ( $\alpha\text{-Al}_2\text{O}_3$ ) was accompanied by a small amount of  $\beta$ -alumina ( $\beta\text{-NaAl}_3\text{O}_7$ ) coming from Bayer process.

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