Experimental report

14/02/2017

Proposal: 1-02-201 **Council:** 4/2016

Title: Texture analysis of magnesium potassium phosphate ceramics

Research area: Materials

This proposal is a new proposal

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Samples: MgKH2PO4(D2O)6

Instrument	Requested days	Allocated days	From	To
D1B	4	4	29/09/2016	03/10/2016

Abstract:

Mg-K phosphate ceramics are chemically-bonded ceramics for applications. They harden at room temperature through an acid-base aqueous reaction between magnesium oxide and a potassium phosphate. The elongated crystal habit of the reaction product exerts a strong control on the mechanical properties. Small angle neutron scattering indicated that less reactive MgO (obtained by calcination of magnesium carbonate) yields coarser microstructure. Evolution of texture in function of the MgO employed and the link with the anisotropy of the mechanical properties and the fraction of crystalline product over amorphous fraction in Mg-K phosphate ceramics, is the aim of this experiment. Although X-rays diffraction and electron backscattering diffraction can be used to retrieve the textural information, neutron diffraction is the only one able to allow for the investigation of a representative volume of sample in a non-destructive fashion.

Introduction

Mg-K phosphate ceramics are chemically-bonded ceramics attractive for applications like waste encapsulation, bone repair, natural fibre composites. When MgO reacts with potassium phosphate (KDP) in solution, formation of K-struvite (MKP) occurs: MgO + KH2PO4 + 5H2O = MgKPO4•6H2O. MKP crystal habit is elongated and prone to orient preferentially (Fig.1). Preferred orientation, in terms of both lattice and shape of minerals, exerts a control on the anisotropy of the physical properties. Mechanical properties of this material have been reported, but essentially for the study of the role played by additives and liquid/solid. Microstructure, texture, and the influence on them of an important parameter for application: the temperature of annealing of MgCO3 (from which MgO is obtained), have not been investigated. The latter is connected with the reactivity of MgO and, in turn, with the rate of the setting reaction, the amount of amorphous phase formed, and the resulting microstructure.

Experiment

Texture of MKP in Mg-K phosphate ceramics, has been investigated at instrument D1B. Cubic 1 cm side deuterated samples investigated were:

Sample produced in a standard way

Sample produced between a temperature gradient (between 4 and 20 C). Due to dependence of setting reaction rates from temperature, this is a first attempt to induce texture.

Sample produced in a magnetic field.

Sample produced from MgO with different reactivity (which influence reaction rates and crystal mean size)

Results

Measurements have been conducted at D1B instrument, operated at wavelength 2.52 Å. The intensity in function of 2 theta has been measured with the available curved position sensitive detector. Counting time was 1 minute for scan. About 1400 scans were collected to cover the orientation space in the range 0-355º phi and 0-90 º chi with 5 º steps. Texture, coherent domain sizes and shapes, microstrains, and structural variations were investigated. Data were analyzed within the combined analysis formalism implemented in the MAUD software. The overall texture strength is evaluated through the texture index [4] which is expressed in m.r.d.^{2} units and varies from one (random powder) to infinity (perfect texture or single crystal) and used to compare the texture strength of different samples exhibiting similar Orientation Distributions (OD). Such normalized pole figures are calculated from the OD of crystallites, refined using the E-WIMV algorithm after extraction of the peak intensities during the Rietveld cycles. The results from a standard sample did not show any residual strains within experimental resolution, that is, the residual stresses, if existing, are estimated lower than 10 MPa. The pole figures for the crystallographic directions of MKP (Fig. 2) are showing weak preferred orientation in the two samples. They illustrate a 4-fold

symmetry which will be further investigated. Data analysis and interpretation for other samples is underway.

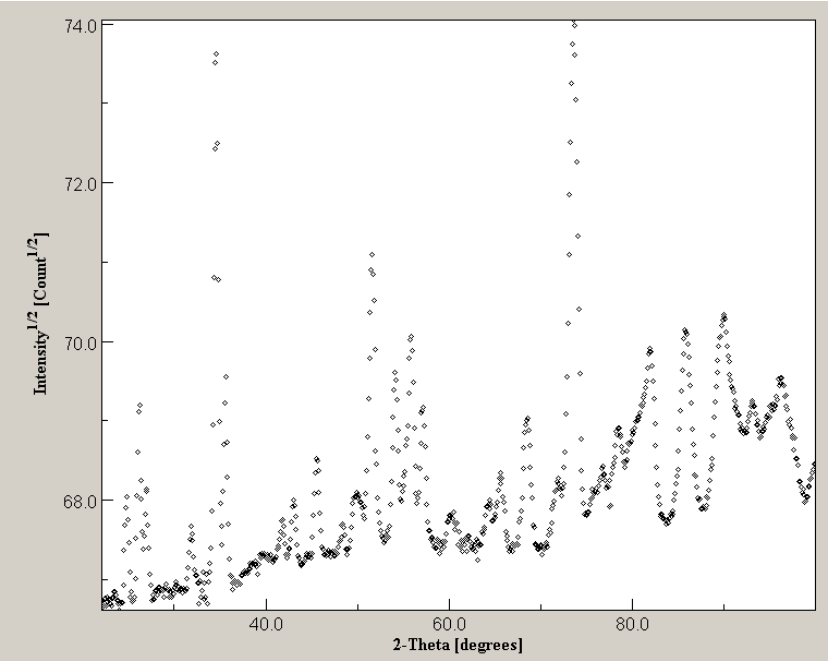

Fig. 1. Diffraction pattern collected at D1B from sample.

(a)

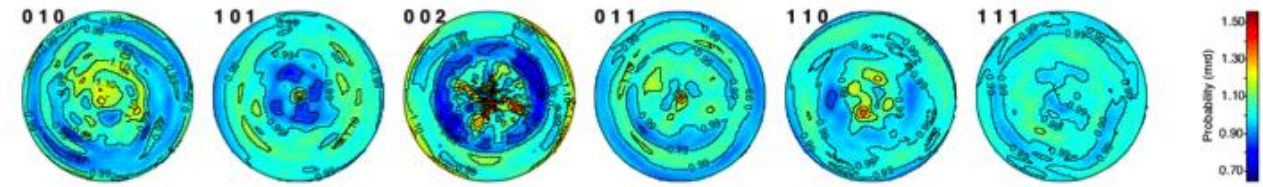

Fig. 2. MKP pole figures for the analyzed sample obtained after OD refinement (linear density scale, equal area projection)