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Preparation and properties of enstatite ceramic foam from talc

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Abstract

The effective method of preparation, stabilization and high temperature treatment of enstatite ceramic foam is described in this work. The technique is based on foaming of suspension of talc, on the stabilization of foam structure and on final high temperature treatment after drying. The spontaneous delamination of aggregates and the redistribution of talc particles in foam are driven by decreasing surface energy. The changes of phase composition as well as the mechanism and the kinetics of processes which take place during the thermal treatment were described. The treatment within the temperature range from 1150 to 1250 °C provides the ceramic foam via sintering without melted phase, whereas a liquid phase sintering occurs at higher temperatures. The final temperature of sintering is 1300 °C. Increasing amount of melted phase supports the formation of enclosed porosity and formed glass stabilizes the high temperature protoenstatite polymorph in the foam.

Keywords: Enstatite; Ceramic foam; Talc; Porous materials

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1. Introduction

Steatite ceramics based on magnesium metasilicate ($MgO{\cdot}SiO_2$ or $MgSiO_3$) is widely used in high-temperature applications and electronics due to the low dielectric losses, high mechanical strength and high temperature resistance.[1] Enstatite containing glass ceramics is a promising material for the preparation of glass ceramics substrates for recording media due to its high strength, improved impact, vibration resistance and particularly low temperature expansion coefficient.[2,3]

Steatite ceramics together with ceramics based on magnesium orthosilicate forsterite ($2MgO{\cdot}SiO_2$ of Mg_2SiO_4) and cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$ or $Mg_2Al_4Si_5O_{18}$) ceramics belong to the ternary systems of $MgO–Al_2O_3–SiO_2$, which are prepared from the mixture of talc ($3MgO{\cdot}4SiO_2{\cdot}H_2O$) and kaolin ($Al_2O_3{\cdot}2SiO_2{\cdot}2H_2O$). $BaCO_3$ is often used as a non-alkaline type of flux which provides low dielectric losses of ceramics. Feldspars or barium carbonate added to talc generate the vitreous phase, hence the crystalline magnesium metasilicate grains are surrounded by the glass matrix after cooling.[1,4]

Steatite occurs in four polymorphic forms such as enstatite, protoenstatite, clinoenstatite and high-temperature clinoenstatite. Protoenstatite is thermodynamically stable at the temperatures higher than 985 °C but in ceramic body it is often stabilized by glassy phase. Therefore, if protoenstatite is not stabilized properly, an undesirable transformation to clinoenstatite takes place at the laboratory temperature. The volume change leads to the formation of cracks in the ceramic body and deteriorates the dielectric properties of material.[2,5]

The combination of properties of ceramics or glass-ceramics with highly porous foam structure enables such applications as high temperature carrier of catalysts[6–8] and photocatalysts,[9,10] refractory and thermal insulating materials,[11–13] filters for molten metals and hot gasses,[14,15] bioceramics and biological substrates.[16–18]

Open cell (reticulated) ceramic foams, which are defined as dispersion systems based on the interconnected bubbles of

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gas phase in continuous ceramic matrix, can be prepared via various manufacturing techniques[19,20] including the transformation of ceramic powder slurry into the solid foam,[21–23] the reticulated sponge method (replica technique)[24–27] or the gel casting.[28–31] The replica technique is based on the pyrolysis of polymeric sponge coated by ceramic slurry. The following high-temperature treatment leads to the ceramic bond via sintering. The gel casting method uses mechanical work and foaming agents in order to foam the ceramic suspension.

The paper deals with the technique of preparation of enstatite based ceramic foam from talc, where the formation of kaolinite framework is driven by decreasing of surface energy of the system and provides a mechanical support to the foam structure. The stabilization of foam by diatomaceous earth significantly increases the time-stability. The structure and the properties of foam as well as the processes occurring during the stabilization and further thermal treatment were investigated.

2. Experimental

2.1. Preparation of foam

Talc, high alumina cement (HAC, Secar 51, Lafarge) and diatomaceous earth (Holland) were used to prepare the solid framework of aqueous foam stabilized by the mixture of anionic surfactant of an alkyl sulfate-type with water (volume ratio 1: 22). Fig. 1 shows the flow chart diagram of the preparation process using foam of the volume density of 50 kg × m^{-3}.

Setting of cement provides the mechanical properties necessary for the manipulation with raw foam before the thermal treatment. In order to avoid the undesirable effect of surfactant on the hydration process, AC was firstly mixed with water. Within the induction period when the hydration had just begun but its rate was very small, talc and diatomaceous earth were stirred into the reaction mixture. Foam (1.5 dm^3) was added at least. After 15 min of stirring the foamed slurry was poured into the mould. After 2–3 days of free curing the samples gained sufficient mechanical properties for manipulation. Further treatment included drying followed by firing to the temperature within the sintering range.

2.2. Sample analysis

The structure and the properties of foam were investigated by scanning electron microscopy (SEM) using Field emission microscope Jeol JSM-7600F. High temperature X-ray diffraction analysis (HT-XRD, X'pert Empyrean, PANanalytical) with high temperature chamber HTK 16 (Anton Paar) and simultaneous TG-DTA and EGA (Q600, TA Instruments connected to infrared spectrometer iS10, Thermo Scientific) were used to investigate the processes and changes in the phase composition during high temperature treatment. The distribution of particle size of kaolinite and diatomaceous earth was investigated by Helos (Sympatec) particle size analyzer.

The behaviour during sintering and melting was investigated by the horizontal type heating microscope EM 201 (Leitz). The cylinder shaped specimen of the diameter and the height of 3 mm was pressed out and put down by the sample holder. The sample was heated under the heating rate of 5 °C min^{-1} up to the temperature of 1400 °C. The behaviour of the specimen during the thermal treatment was investigated via the relative change of area (A):

$$A\ (\%) = 100\left(1 - \frac{A_T - A_0}{A_0}\right) \tag{1}$$

where A_0 and A_T are the initial specimen area and the area measured at the temperature T, respectively. The picture recorded by CCD camera with the step of 25 °C was used to determine the temperatures of standard points which are defined for heating microscopy and from which the points of sintering (SP), deformation (DP), hemisphere (HP) and flowing (FP) were identified.

The apparent density and porosity were assessed by the evacuation method and the hydrostatic weighting and pycnometric measurements were used for the determination of true density. The specimens were prepared in laboratory furnace under the heating rate of 2 °C min^{-1} to the temperatures within the sintering range assed by heating microscopy (1150, 1175, 1200, 1225 and 1250 °C). After 60 min at final temperature the samples were cooled to 800 °C under the cooling rate of 2 °C min^{-1} and next they were left in the furnace to cool down spontaneously to the laboratory temperature. The thermal conductivity at ordinary temperature was measured by TCi (C-Therm) analyzer without the contact agent.

2.3. Kinetics and mechanism of processes during thermal treatment

The apparent activation energy (E_a) and the frequency factor (A) of investigated process were evaluated by the mechanism-free method based on the Kissinger kinetic approach:[32,33]

$$\ln\left[\frac{\Theta}{T_m^2}\right] = \ln\left[\frac{AR}{E_a} n(1-\alpha_m)^{n-1}\right] - \frac{E_a}{RT_m} = \text{const.} - \frac{E_a}{RT_m} \tag{2}$$

where T_m is the peak temperature measured under applied heating rate $\boldsymbol{\Theta}$, n is the empirical reaction order (kinetic exponent), α_m is the fractional conversion reached for the temperature T_{m} and R is the universal gas constant.

The non-isothermal kinetics experiments were performed using TG-DTA analyzer SDT Q600. 30 mg of sample were heated under the heating rates (Θ) of 2, 3, 5, 7, 10, 15 and 20 °C min^{-1} up to 1100 °C. The points on the plot of ln (AR/E_a) versus $T_m{}^{-1}$ were fitted by the straight line with the slope equal to $-E_{\mathrm{a}}/R$ whereas the intercept yields to the constant term of Eq. (2).

The mechanism was estimated from the shape of DTG peak via the value of kinetic exponent ($\boldsymbol{n}$) which is related to the empirical order of reaction.[32] The exponent can be calculated from the equation:[34,35]

$$n = \frac{2.5RT_m^2}{w_{1/2}E_a} \tag{3}$$