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Preparation of cerium fluoride suspensions for grain-orientation by a strong magnetic field assisted slip-casting process

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Abstract

To achieve highly transparent CeF_3 scintillation ceramics for high-energy physics applications, a grain-oriented structure, which could be obtained by a slip-casting process assisted by a strong magnetic field, was necessary to decrease the birefringence effects caused by the CeF_3 hexagonal phase structure. The preparation of well-dispersed CeF_3 suspensions, which is a key factor for the grain-orientation, was studied in this paper, and the factors that influenced the dispersing property of the suspensions, including properties of raw powders, pH of the suspension and amount of the dispersant, were also discussed. Optimized experimental parameters were screened out by comparing the viscosity of the suspensions and were used to prepare well-dispersed CeF_3 suspensions. The resulting suspensions were then slip-cast under a strong magnetic field of 9 T used to align the particles. X-ray diffraction (XRD) results indicate grain-oriented ceramics that were obtained after sintering at 950 °C in vacuum for 3 h, with the calculated Lotgering factor f of 94.35%, compared with 3.67% for the sample obtained without magnetic field.

Keywords: A. Suspensions; Ammonium polyacrylate; Cerium fluoride; Grain-orientation

1. Introduction

Transparent CeF_3 ceramics may become a good potential candidate for calorimetry at the High-Luminosity Large Hadron Collider (HL-LHC) at CERN, the European Organization for Nuclear Research [1–4]. Unfortunately, CeF_3 has a hexagonal phase structure with a space group of P6$_3$/mcm (193), the difference between its refractive indices is 0.008 at 352 nm [5], and the light scattering caused by birefringence effects at the grain boundaries precludes the achievement of full transparency. Thanks to the development of superconducting magnets, with the help of which grain-oriented ceramics with a non-cubic phase structure have been fabricated successfully [6–10]. Grain-orientation could ease the birefringence effects and improve the transparency of the non-cubic ceramics. With the assistance of a magnetic field of 12 T during the slip-casting process, a grain-oriented structure and a significant increase of the optical transmission of hexagonal Al_2O_3 ceramics were achieved [11]. Some other transparent non-cubic polycrystalline ceramics were also obtained through a similar process, e.g. for Nd^{3+}:$Ca_{10}(PO_4)_6F_2$ [12]. In the case of strong magnetic field assisted grain-orientation technology, a magnetic torque is calculated by $T_m=0.5\mu_0 H^2\Delta\chi\sin 2\theta$ [12], where μ_0 is the magnetic permeability in vacuum, H is the intensity of the magnetic field, $\Delta\chi=\chi_c-\chi_{a,\ b}$ is the anisotropy of the magnetic susceptibility, and θ is the angle between the magnetic field and the magnetic easy axis, and is generated and applied on the crystalline particles having a magnetic anisotropy under the magnetic field. A rotation will be achieved and the magnetic easy axis of the grains will align along the magnetic field direction when the T_m is larger than the energy of thermal motion, kT. Due to the small susceptibility of

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the non-magnetic material, interactions between the particles in a flocculated suspension prevent the particles from rotating when a magnetic field is applied, so it is important to disperse the particles in the suspension to allow the magnetic field to exert its effects. Typically, when powders are dispersed into a suspending medium, such as water, the attractive van der Waals forces result in the particles aggregating to form flocculated clusters. A well-dispersed system can be achieved by modifying the surface of the particles in the suspension to provide some repulsive potential energy to offset the attractive van der Waals forces. There are three main sources of repulsive potential energy [13]: electrostatic, resulting from electrostatic interactions between charged particle surfaces, steric, from steric interactions between particle surfaces coated with adsorbed polymeric species, and electrosteric, a combination of the former two. Dispersant is widely used to introduce the repulsive potential energy.

As far as we know, very little attention has been paid so far on the fabrication of grain-oriented CeF_3 ceramics, despite its promising high-energy physics applications. Aiming to achieve these ceramics, efforts must be focused on the preparation of appropriate suspensions, which is a key factor for the grain-orientation in the magnetic field. In the present work, ammonium polyacrylate was used as a dispersant, and the factors influencing the dispersing property of the suspensions were also studied. The motivation is to try finding out the optimized experimental parameters giving rise to well-dispersed suspensions, which could be used to slip-cast under a strong magnetic field to align the particles. XRD measurements were conducted on the obtained ceramics to demonstrate the grain-orientation results.

2. Experimental materials and methods

2.1. Raw materials

CeF_3 powder with high purity ($\geq 99.9\%$) provided by Suzhou Pujing Co., Ltd. (Suzhou, China) was used as received. Deionized water was used as the dispersion medium and ammonium polyacrylate, produced by R. T. Vanderbilt Company, Inc. (Norwalk, USA), was used as a dispersant. Analytically pure ammonium hydroxide, NaOH and HCl produced by Sinopharm Chemical Reagent Co., Ltd. (Beijing, China) were used to adjust the pH of the suspension. The raw materials were weighed with an accuracy of 0.001 g and the pH was measured on the suspensions after ball-milling with an accuracy of 0.01. The morphology of the CeF_3 powder was observed by a scanning electron microscope (SEM) of type JSM-6700F produced by JEOL Ltd. (Tokyo, Japan). The particle size distribution was measured by a particle size analyzer of type XDC produced by Brookhaven (Holtsville, USA). Electron diffraction pattern measurement was conducted on the CeF_3 powder to analyze the crystalline structure by a transmission electron microscope (TEM) of type JEM-2100F produced by JEOL Ltd. (Tokyo, Japan).

2.2. Zeta potential measurements

The Zeta potential of the CeF_3 powders in a suspension with or without dispersant was measured by a Zeta potential analyzer of type Zetaplus produced by Brookhaven (Holtsville, USA). The measurements were conducted on suspensions with a solid loading of 0.5 vol%, prepared by ultrasonic agitation provided by a KQ-500DB ultrasound unit from Kun Shan Ultrasonic Instruments Co., Ltd. (Kunshan, China) using 500 W at 40 kHz for several minutes. HCl and NaOH were added to adjust the pH of the suspension.

2.3. Suspension preparation and viscosity measurements

Each suspension was prepared by the following procedure. The dispersant ammonium polyacrylate was firstly dissolved into deionized water, and ammonium hydroxide was added to adjust the pH of the solution. It took several minutes to achieve a homogeneous mixture. Then, a ball-milling process was used to disperse the CeF_3 powders into the resultant solution and to break up the agglomeration between the particles. The viscosity measurements were conducted on suspensions with a solid loading of 40 vol% by a Rheometer of type MCR301 produced by Anton Paar (Graz, Austria). The shear rate accelerated from 0.01 to 1000 s^{-1} and then slowed down to 0.01 s^{-1} again.

2.4. Slip-casting and heating

In Tarì's study [14], the optimized experimental parameters were found to be independent of solid loading. As the solid loading increases, the viscosity of the suspension increases, and the effective parameter range gets smaller, so the optimized parameters were screened by using the suspensions with higher solid loading of 40 vol%. By using these parameters, well-dispersed suspensions with a solid loading of 30 vol% were prepared and then poured into porous plaster molds, and slip-casting was carried out in a vertical static magnetic field of 9 T. The casting process lasted several hours long, and the magnetic field was maintained throughout the whole process. After casting, the wet bodies were dried at 200 °C in a CF_4 atmosphere for 5 h and then sintered at 950 °C in vacuum for 3 h. For comparison, some CeF_3 ceramics were prepared in a similar process without the presence of a magnetic field.

2.5. Characterization of grain-orientation

The grain-orientation of the obtained ceramics was characterized by an XRD instrument of type D8 ADVANCE from Bruker (Karlsruhe, Germany) on the surface and on cross sections of the samples. The Lotgering factors f were calculated using the PDF 08-0045 data and the XRD data obtained.