Fluoride based transparent ceramics by spark plasma sintering

Mythili Prakasam[1*], Emmanuel Petit[1], Inka Manek-Hönninger[2], Alain Largeteau[1]

[1] CNRS, Univ. Bordeaux, ICMCB, UMR 5026, F-33600 Pessac - FRANCE

[2] CELIA, Univ. Bordeaux-CNRS-CEA, UMR 5107, F-33405 Talence - FRANCE

**Corresponding author: Mythili.prakasam@icmcb.cnrs.fr*

Highlights

- Optimizing sintering parameters to yield transparent fluoride based ceramics at T < 550°C
- High vacuum helps with pyrohydrolysis of fluoride
- > 85% of transparency in infrared and 60% in visible region

1. Introduction

Recent applications of Infrared (IR) radiation for various applications for designing optical components ranging from mirrors, lenses, prisms, infrared detectors and CO_2 lasers has increased the search for new materials and its quality. Various transparent materials such as crystals, ceramics and glasses are used for fabrication of IR devices. Conventional transparent materials have a strong absorption in the infrared region making them unsuitable in this spectral range. In the present scenario, crystal though are ideal for applications, primarily in terms of compactness and user friendly, these types of crystals are difficult to be grown due to the high temperature growth issues, which limit size and quality. The Visible-infrared transmission of the materials obtained by this method is similar to that of conventionally used materials such as zinc selenide single crystal (ZnSe), zinc sulfide (ZnS) and germanium (Ge) single crystals. In order to overcome difficulties due to the toxic materials involved in the coating of such materials, alternative materials such as fluorides are used instead of ZnSe and ZnS. The lower value of the refractive index of fluoride ceramics leads to a lower Fresnel impairment at their surface, so that antireflective coatings (AR) required for the manufacture of optical systems currently employed can be avoided. This aspect leads to a simplified process of fabrication of lenses done with nanosized particles (20-30nm) of powder to obtain nanostructured ceramics in terms of number of steps involved. The usage of toxic material for the coating is also avoided. In the present work, we will present about a low temperature route of fabricating transparent fluoride ceramics by spark plasma sintering [1].

2. Methods

Commercial powders of CaF_2 and BaF_2 from M/S Fox chemicals with the grain sizes in the range of 20-40 nm was used for fabrication of transparent ceramics by using spark plasma sintering. To sinter we employed commercial DR. SINTER LAB Spark Plasma Sintering system, Model SPS-511S / SPS-515S equipped with high vacuum system capable of reaching 10^{-3}Pa. The fabricated transparent ceramics of CaF_2 and BaF_2 were tested for optical characterization (Figure 1) including UV-Vis-IR transmission, laser damage threshold and laser testing at various wavelengths.

3. Results and discussion

Transparent ceramics processing with nanosized ceramic powders and advanced densification technology provides an alternative approach to overcome the disadvantages/ limits of conventional single-crystal growth methods. It would be much easier to elaborate polycrystalline ceramics with a full densification state and a homogeneous chemical composition under sintering temperature much lower than its melting point with a relative low cost and size flexibility. In order to obtain the transparent ceramics, it is important to obtain maximum density and porosity in the orders of 100 ppm/ < 0.01 vol %. Various sintering parameters interplay to obtain the desired result, where the prime factors being sintering temperature (T_s), dwell time (t) and Pressure (P_s) and the point of pressure application (A_P). In the present work, we optimized the sintering parameters to obtain homogeneous transparent ceramics of CaF_2 and BaF_2 at temperatures < 550°C.

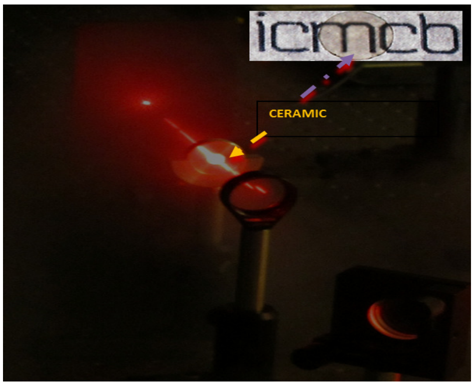

Figure 1. CaF_2 ceramic showing a good transmission in Red and infrared region tested with Laser beam wavelength 633 nm

4. Conclusions

A methodology [1] was defined capable of yielding fluoride transparent ceramics with commercial fluoride powders from M/S Fox chemicals nanopowders, which were fabricated by SPS at temperatures 450°C< T<550°C. The polycrystalline material obtained is of optimal quality with regards to optical homogeneity. The transparency in the infrared region is ~ 85% and visible region ~ 60%. The microstructure, optical transparency and laser measurements will be presented in detail.

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References

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Transparent ceramics; infrared optics; Laser