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UV transmitters of aluminum polyphosphates prepared by high pressure technique at room temperature

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The possibility to obtain nanostructured ceramic materials from nanometric powders has been studied over the last few years. Such interest was motivated by the promise of potential improvement in optical and mechanical properties, like transparency, ductility, hardness and strength, among other potentially interesting properties [1–4]. These enhanced properties have been related to the nanostructure of these materials [5]. However, it is very difficult to retain the nanocrystalline grain size during the sintering process in order to produce a high-density bulk nanostructured ceramic. Therefore, it is necessary to find favorable conditions to enhance densification and limit grain grow. The high pressure technique has been used successfully to obtain nanostructured ceramic or composite materials with the desired properties [6–10].

Aluminum polyphosphate nanostructured systems have been used extensively as pigment for painting [11, 12], as matrix for composite materials [13], as well as ceramic foams [14]. However, as far as we know, there is no report in the literature concerning the preparation of aluminum polyphosphate ceramic materials by using the compaction of nanometric powders. In this work, we studied the required conditions to produce bulk ceramic materials from nanometric powders of aluminum polyphosphate, using a special high pressure cell configuration with a highly hydrostatic pressure transmitting medium and at room temperature. The samples were analyzed by Vickers microhardness, density, UV-VIS spectroscopy and X-ray diffraction.

Nanocrystalline aluminum polyphosphate with P:Al mol ratio $= 0.60 \pm 0.01$ in the reaction admixture, was obtained by precipitation from solutions of sodium polyphosphate, aluminum nitrate, and ammonium hydroxide, under strong stirring and at room temperature. Particle diameters were in the 2–6 nm range. The powder obtained was dried at 600 °C [15].

For the high pressure experiment the aluminum polyphosphate powder was initially pre-compacted in a piston-cylinder type die to approximately 0.1 GPa. The typical initial volume was about 300 mm^3 (diameter = 8 mm, height = 6 mm). These samples were then placed in a lead container that is extremely soft and acts as a pressure-transmitting medium, maintaining a very low shear strength with increasing pressure [8]. The containers were assembled in a toroidal-type high pressure chamber [16]. The compaction was then accomplished at pressures of 4.0, 6.0 and 7.7 GPa, at room temperature, for several samples. The pressure calibration was performed by the "fixed points" technique, using Bi and Yb, which allowed calibration of the pressure in the following three fixed points: Bi with phase transitions at 2.5 and 7.7 GPa, and Yb with a phase transition at 4.0 GPa. The pressure is considered accurate to ± 0.5 GPa. Detailed description of the high pressure calibration method is given elsewhere [17].

The compacts obtained were polished with sand paper (grade 1000 and 2500) and alumina paste for Vickers microhardness measurements, which were performed in a Shimadzu microindenter with loads of 100 g. The density of the samples were determined using the picnometry method. The water absorption was obtained from the ratio between the dry and the wet weight of the sample. The wet weight was measured after the sample was immersed during 30 min, at room temperature.

The samples were analyzed by UV-VIS spectroscopy in the range from 250 to 600 nm using a spectrophotometer Shimadzu UV-1601PC. The X-ray diffraction patterns were obtained using Cu K_α radiation in a Siemens diffractometer D500.

Aluminum polyphosphate compacts, crack-free and optically translucent were obtained by high pressure processing. They have a cylindrical form with height of 4 mm and diameter of 6 mm. In Table I are listed the results for microhardness, density and water absorption for samples compacted at pressures of 4.0, 6.0 and 7.7 GPa. The density changed with pressure and the highest value was measured for the sample compacted at 7.7 GPa, which was approximately 99.0%

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TABLE I Results for microhardness, density and water absorption for samples compacted at different pressures

Sample	Pressure (GPa)	Density (kg/m^3)	Microhardness (GPa)	Water absorption (wt%)
1	4.0	2310 ± 20	3.3 ± 0.1	1.1 ± 0.2
2	6.0	2360 ± 20	3.4 ± 0.1	1.1 ± 0.2
3	7.7	2550 ± 20	3.6 ± 0.1	1.0 ± 0.2

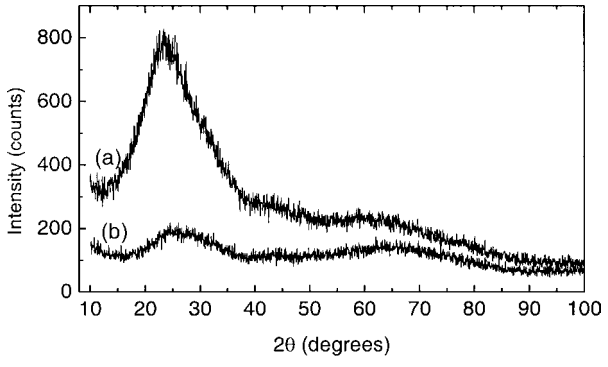

Figure 1 Typical X-ray diffraction patterns for powder sample (a) and compacted sample (b).

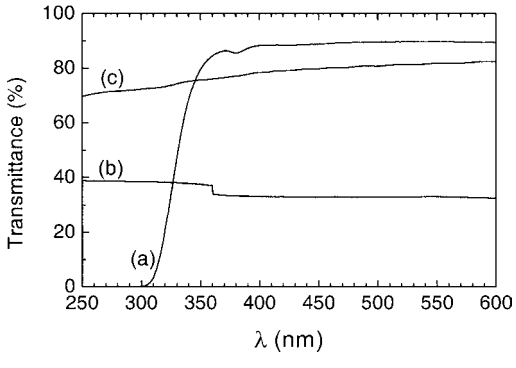

Figure 2 Transmittance spectra for microscope slab glass (a), aluminum polyphosphate compact (b), and a quartz slab (c).

of theoretical density (2570 kg/m^3). For these samples the water absorption was low, about 1.0%. This value is very impressive compared to the samples sintered at 1600 °C and ambient pressure, which showed a value of 3.4% for the water absorption.

Typical X-ray patterns for amorphous materials are shown in Fig. 1, for the aluminum polyphosphate powder and for the compact (sample 3). As can be observed, the high-pressure processing did not promote crystallization in the compacted sample.

Fig. 2 shows a comparison between the transmittance spectra of a quartz slab with thickness of 2.4 mm, a microscope slab glass of 2.0 mm and the aluminum polyphosphate compact with 2.6 mm. It was observed that the sample presents a transmittance about 40% over all of the range measured, due to light scattering. Although the absolute transmittance values are lower than for the quartz slab, with almost the same thickness, they showed a similar behavior, completely different from the microscope slab glass that has a strong absorption in the UV region. This is a very important result because there are few solids that exhibit this behavior, as for example, calcium and magnesium fluorides, and UV grade fused silica. Additionally, our samples present density and microhardness comparable to these optical materials, making the aluminum polyphosphate compacted sample a promising optical window for UV radiation.

In conclusion, aluminum polyphosphate compacts were produced using the high pressure technique, for the first time. They showed important mechanical and optical properties, being translucent, crack free, hard, dense, and can be polished to optical quality, showing a significant transmittance in the UV range. They can be used as an optical window in this wavelength range, which is a characteristic that only very few materials possess. In further research, a deeper insight into this property will be sought.

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