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Structural, optical and thermal stability of the GSN nanocrystals

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Crystals of GSN were grown by a slow evaporation technique published by the authors elsewhere [1]. A crystal was then selected and observed by scanning electron microscopy (SEM) in order to analyze the morphologic and surface characteristics. Fig. 1 shows the image obtained by SEM of a GSN nano-monocrystal using a JEOL JSM-5800LV microscope, with an acceleration voltage of 15 kV and a current of 40μA. A crystal with vertices and defined angles was observed, with facets with flat surfaces, which also possessed parallel facets with an elongated form. Moreover, a series of impurities were noticed, mainly white spots on the surface of the faces, which correspond to the precipitated salts that did not obtain favorable crystallization

The SHG signal of GSN was measured by the powder technique of Kurtz and Perry [2]. The crystal was ground into powder and densely packed between two transparent glass slides. The second harmonic output was generated by irradiating powder samples by a pulsed-laser beam. The source is Nd:YAG Quanta ray INDI sries laser emitting 1064 nm, generating an 8 ns pulse and it was operated at 6 mJ/pulse and a repetition rate of 10 Hz. The second harmonic signal was analyzed with a Jobin-Yvon monochromator Triax320 and detected with a Hamamatsu R928 photomultiplier tube. Then, EGG/PAR 165 boxcar averager and readout processed it on a strip-chart recorder. The Fig. 2 presents the preliminary result of signal SHG experiment when the emission spectrum shows a strong signal around the 532 nm, wavelength corresponding to the half of 1064 nm used with the Nd-YAG laser beam. This measurement establishes that the frequency dubbing has been obtained.

GSN differential thermal and thermogravimetric (DTA-TGA) analysis is shown in Fig. 3. The sample was analyzed in a TA Instruments STD 2960 using simultaneous DTA-TGA mode, and was heated at rate of 15°C/min in 50cm^3 of air. From DTA curve, it has been found that GSN is stable up to 198°C, because endothermic signals were not observed, which characterize crystalline transformations. From the 198°C to 226°C a endothermic signal was observed that corresponded to the beginning of a phase change, until it finally melted completely, which were followed by two exothermic decomposition signals with temperatures of 302.5°C and 396.6°C, which corresponds to the combustion of glycine molecules and to the decomposition of the $NaNO_3$ respectively. TGA spectrum shows a loss of material at 226°C, which is in accordance with the decomposition of the organic part. At an elevated temperature this decomposition process continues up to 700ºC. Also was observed that 2.4% of the water contained in the material, was lost before being fused.

Fig. 4 displays the diffraction pattern and the (h k l) planes of the sample before and after heating. . The plane reflections meet the experimentally reported structure which belongs to the monoclinic system space group Cc with cell parameters a = 14.329 (3) Å, b = 5.2662 (11) Å, 9.1129 (18) Å, β = 119.10° (3) and cell volume V=600.9 (2) $Å^3$. The peaks in the XRD pattern were indexed, and differences in the positions of reflection of the planes with the diffraction pattern previously reported [3]were observed. No structural changes are present.

References

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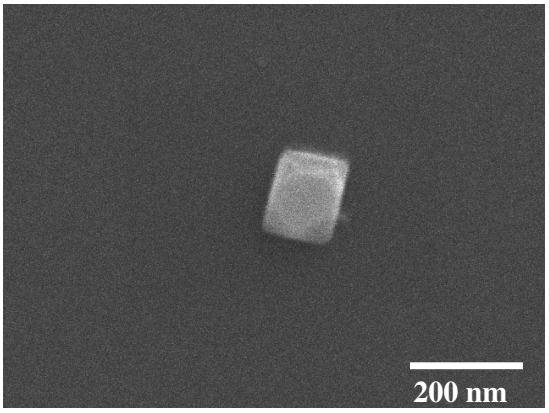

FIG. 1 SEM image of GSN nano-monocrystal

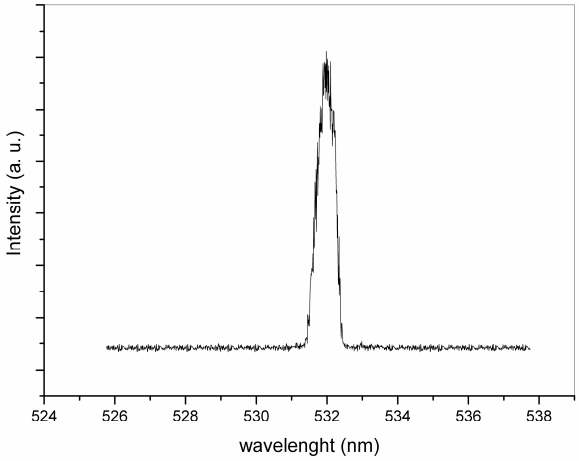

FIG. 2. 532 nm emission spectrum of GSN

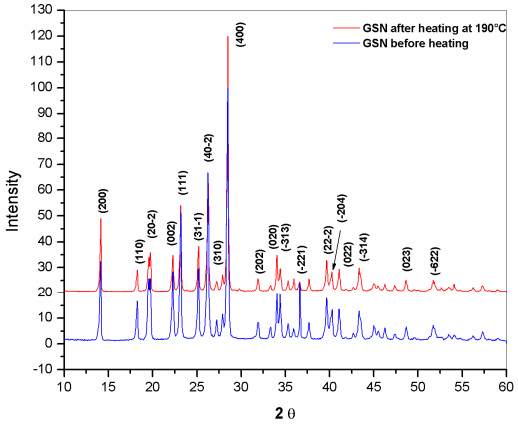

FIG. 3. XRD spectra of GSN before and after heating

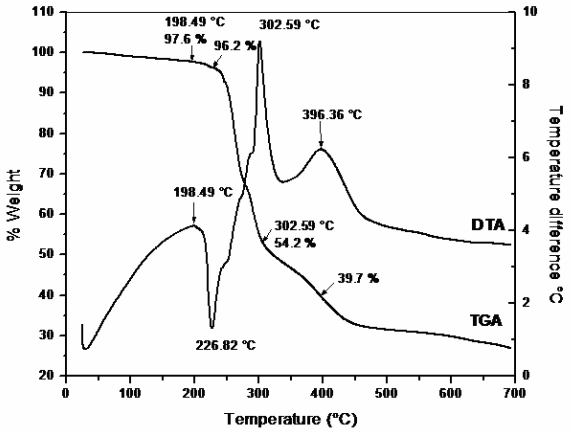

FIG. 4. DTA/TGA spectra of GSN