Supporting Information for

Facile Nanoparticle Dispersion detection in Energetic Composites by Rare Earth Doped in Metal Oxide Nanostructures

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Experiment Details

TiO_2 nanoparticles were prepared using a hydrothermal synthesis from a titanium isopropoxide (TIP) precursor, and nitric acid stabilizer. The washed nanoparticles were suspended in an 80:20 water:ethanol mixture, and were spray dried with a Bucci spray drier with an ultrasonic nozzle to form spherical agglomerates. As such, the proposed method can be used with the differing spray-drying parameters to create tracers that mimic the behavior of pure, catalytic additives of a wide range of sizes. In this experiment, two sizes of tracers were made to mimic pure additives of two different particle sizes, large (~6 µm) and small (~3.5 µm) particles. After collecting spray dried power, the particles were either heat treated at 400 °C for 3 hours, or were used as is. These powders were used for some of the characterization (UV-Vis, photoluminescence, XRD, SEM), but to obtain truly representative characterization, the particles were dispersed in a common solid propellant binder, hydroxyl terminated polybutadiene (HTPB), and were mixed using the hand-mixing methods explained by Stephens et al. [1].

Illumination of the Embedded particles in a Rubber Binder

Sample strands were made using the different particle formulations, and with different mixing methods to evaluate if the particle fluorescence could be observed through the binder, and if qualitative information about the dispersion of the particles could be obtained using standard, inexpensive methods. A mercury arc lamp was used to generate light, which was subsequently smoothed with a convex lens and filtered with a 470 nm bandpass filter to generate blue light to illuminate the samples sufficiently and consistently. A Nikon D60 SLR camera with a red filter was used to image the illuminated strands, with an enclosure around the entire aparatus to prevent ambient light from affecting the imaging.

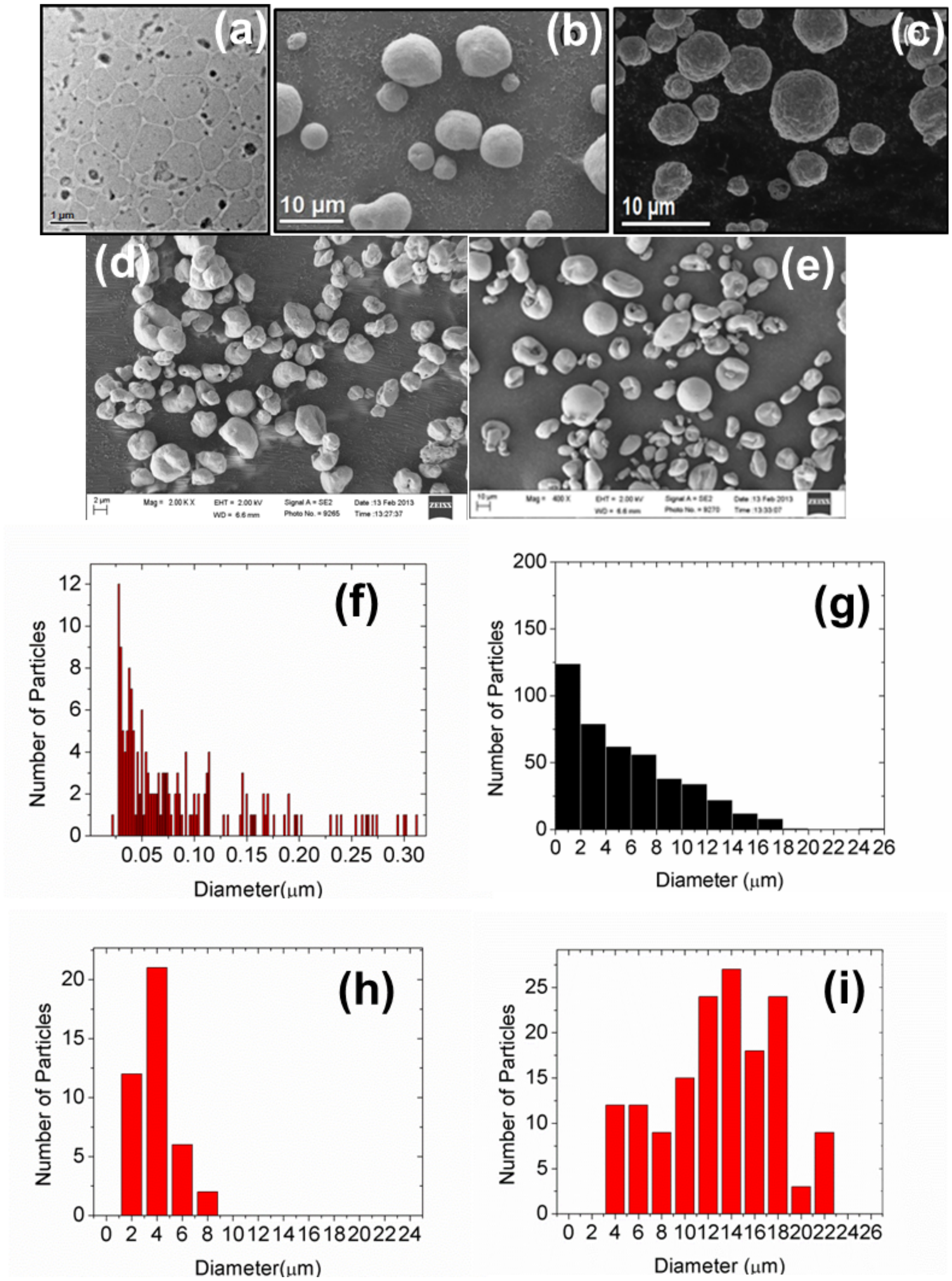

Figure S1 (a) TEM micrograph of the roughly spheroidal $EuTiO_2$ small agglomerates ($EuTiO_2$-Sm). These particles are around 90 nm in size, shown in histogram (f) which exhibits a truncated normal distribution. (b) SEM micrograph of $EuTiO_2$-H spray dried agglomerates, shown in the histogram in (g) to be around 6.2 µm in size. (c) Pure TiO_2 spray dried with the small agglomerate parameters (TiO_2-H) show that the Eu addition does not significantly affect the agglomerate size or morphology. (d) SEM micrograph of the $EuTiO_2$-Md spray dried agglomerates with accompanying size distribution in histogram (h). (e) SEM micrograph of $EuTiO_2$-Lg particles with accompanying size distribution in histogram (i).

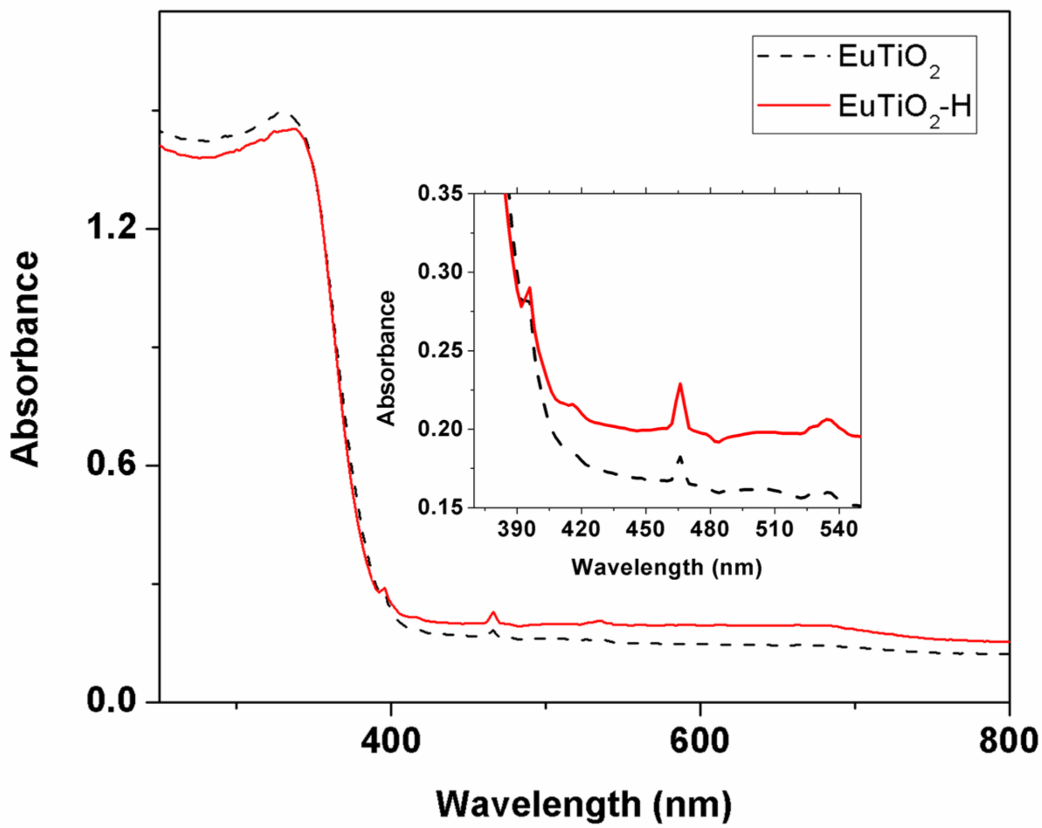

Figure S2 UV-Visible range diffuse reflectance spectroscopy on both the heat treated ($EuTiO_2$-H), and non-heat treated ($EuTiO_2$) Eu-TiO_2 powder shows excitation peaks at 394, 464, and 534nm.

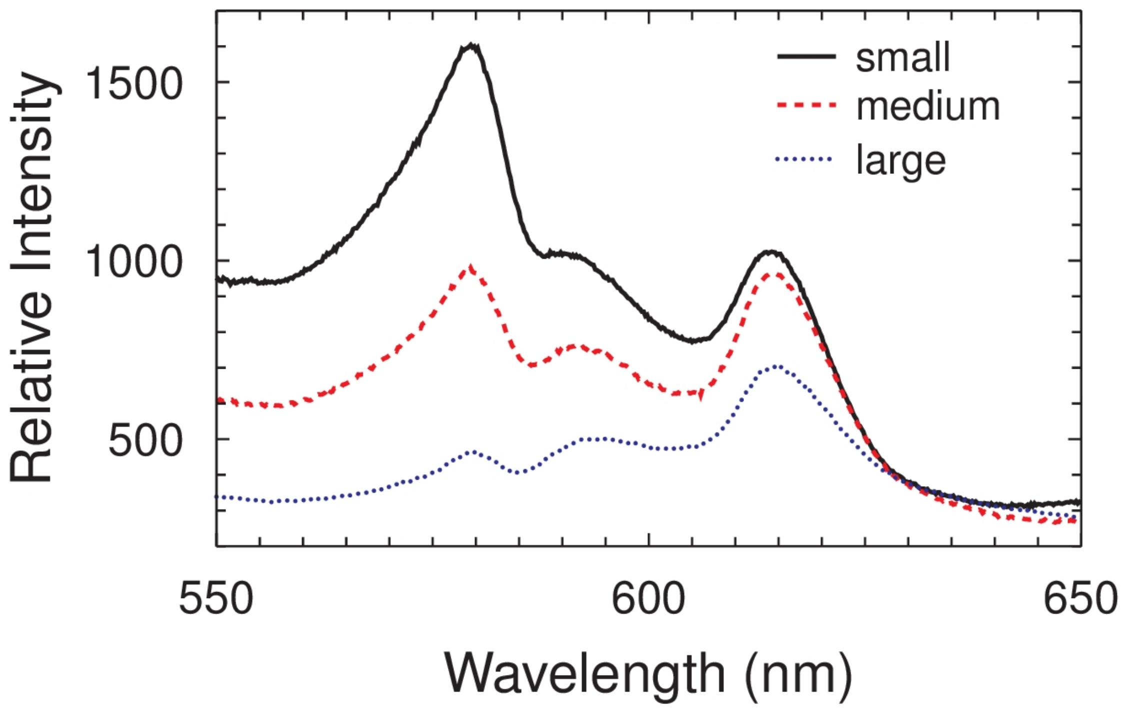

Figure S3 Photoluminescence data for the small, medium, and large samples

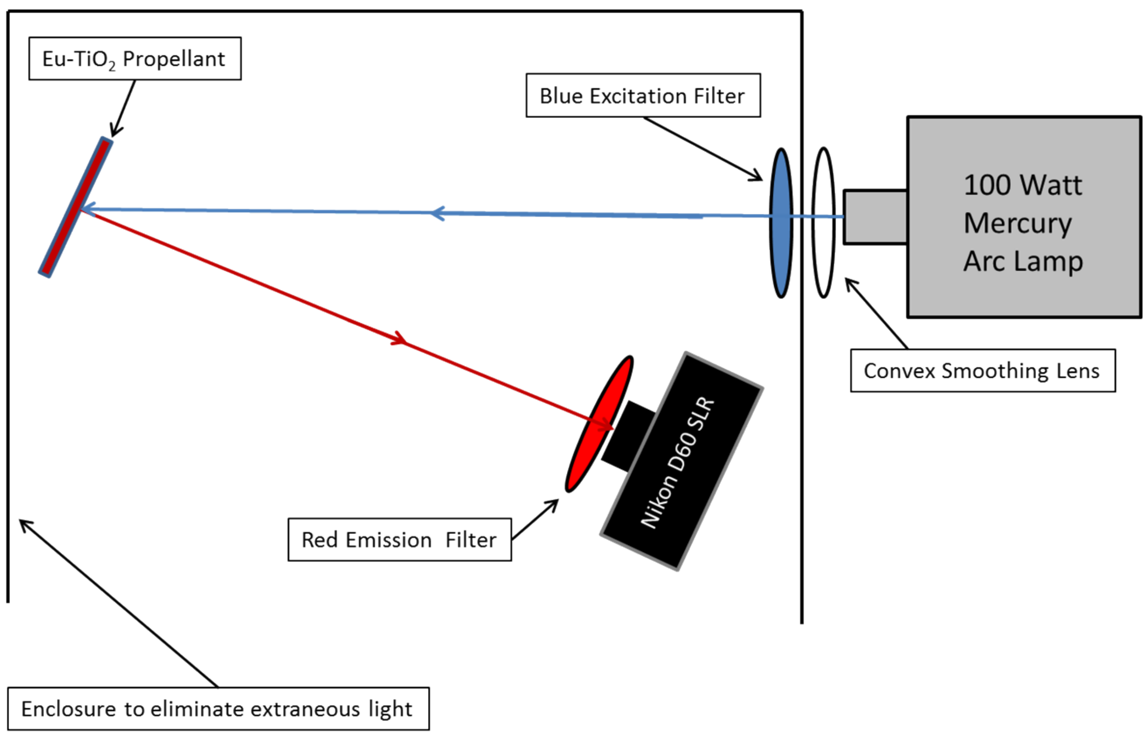

Figure S4: Schematic of shrouded illumination setup for inducing fluorescence in bulk strands

References

1. M. A. Stephens, E. L. Petersen, R. Carro, D. L. Reid and S. Seal, Propellants, Explos. Pyrotech. 2010, **35**, 143–152.