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Investigation of Nanoparticle Reactions with Laser Heating by *In situ* TEM

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Nanoscale materials have significantly different and enhanced properties compared to their micro or macroscopic counterparts. As a result, they have been investigated in many studies [1]. In particular, Al nanoparticles has attracted significant attention, especially in the study of propellants, explosives and pyrotechnics, due to their high energy density [2], non-toxicity, commercial availability and low cost [3]. Fluorine, which is often called as material of extremes because of its high reactivity, is generally preferred as an additive in Al nanoparticle systems. Their reaction produces one of the strongest bonds ever determined and improve the general performance of the propellants [4]. The structural changes in fluorine-based polymers during their reactions with nano-aluminum particles can be observed via transmission electron microscopes (TEM) [5].

TEM is an essential tool for material characterization, and with the development of Ultrafast Transmission Electron Microscopy (UTEM), high resolution dynamic process analysis at ultrafast time scales became possible. In UTEM, the electron beam is generated via photoemission when a UV laser pulse hit the cathode and the stimulation of the specimen is achieved via a second laser pulse, called the sample excitation laser. The time difference between two pulses can be controlled by a computer controlled optical delay line (Figure 1). This configuration allows us to perform diffraction analysis [6-7], real space imaging [8,9], and electron energy loss spectroscopy [10] at ultrafast time scales. UTEM is especially useful in identifying intermediate states that occur during complex processes before a system reaches equilibrium enabling us to improve our understanding of fast processes in a variety of scientific topics.

In this work, we utilized a UTEM to perform an in-situ laser heating experiment on nano-aluminum-THV (tetrafluoroethylene, hexafluoropropylene, and vinylidene fluoride with the empirical formula $C_{2.29}F_{4.33}H_{0.25}$) composite sample using the sample excitation laser pulse. A 532 nm wavelength pulsed laser beam with Gaussian profile is used to increase the temperature of the sample and initiate the reaction between nano-Al and THV. The sample was stimulated with 1 nanosecond long 25000 laser pulses in 1 second, and the reaction between Al nanoparticles and THV was observed and monitored via high resolution TEM imaging.

Figure 2 depicts sequential snapshots taken by the UTEM at different times into the reaction to document the progress of the reaction throughout the observed time period. The structural change caused by the chemical reaction of the nano-Al particles and fluorine based composite can be seen clearly in these images. Diffraction analysis performed after the reaction showed the presence of exotic η-AlF_3 phase. We believe the formation of the η-phase is because of the nonlinear heating of the nanosecond laser pulses. Reflectance measurements for nanoaluminum-THV sample conducted and finite elements analysis were performed to determine the temperature change of the particles over time. In this presentation, the results of the dynamic in-situ TEM analysis, reaction temperature calculations, diffraction and spectroscopy analysis for the nano-Al THV reaction will be discussed.

References:

[1] R. Yetter, G. A. Risha and S. F. Son, Proceedings of the Combustion Institute **32** (2009) p. 1819..
[2] F. Noor *et al*, Phys. Chem. Chem. Phys. **15** (2013) p. 20176.
[3] C. Rossi *et al*, Journal of Microelectromechanical Systems **16** (2007) p. 919.
[4] J. Ken and G. Stead, Journal of Fluorine Chemistry **104** (2000) p. 5.
[5] H. S. Park *et al*, Ultramicroscopy **110** (2009) p. 7.
[6] M. S. Grinolds *et al*, PNAS **103** (2006) p. 18427.
[7] A. Yurtsever and A. H. Zewail, Science **326** (2009) p. 708.
[8] J. S. Baskin, H. Liu and A. H. Zewail, PNAS **29** (2014) p. 10479.
[9] O. Kwon *et al*, Nano Letters **8** (2008) p. 3557.
[10] F. Carbone, O. Kwon and A. H. Zewail, Science **325** (2009) p. 181.

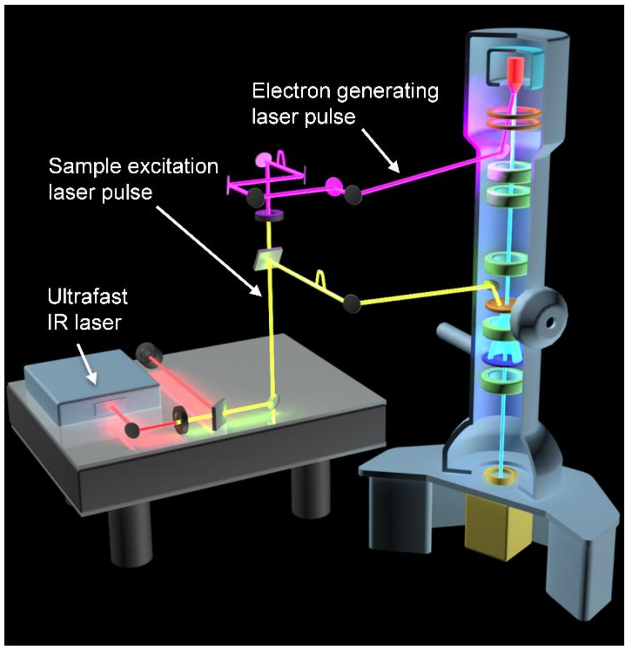

Figure 1. Modified 200 kV TEM with ultrafast laser system. Time delay between sample excitation and electron generating laser pulses is controlled by an optical delay line.

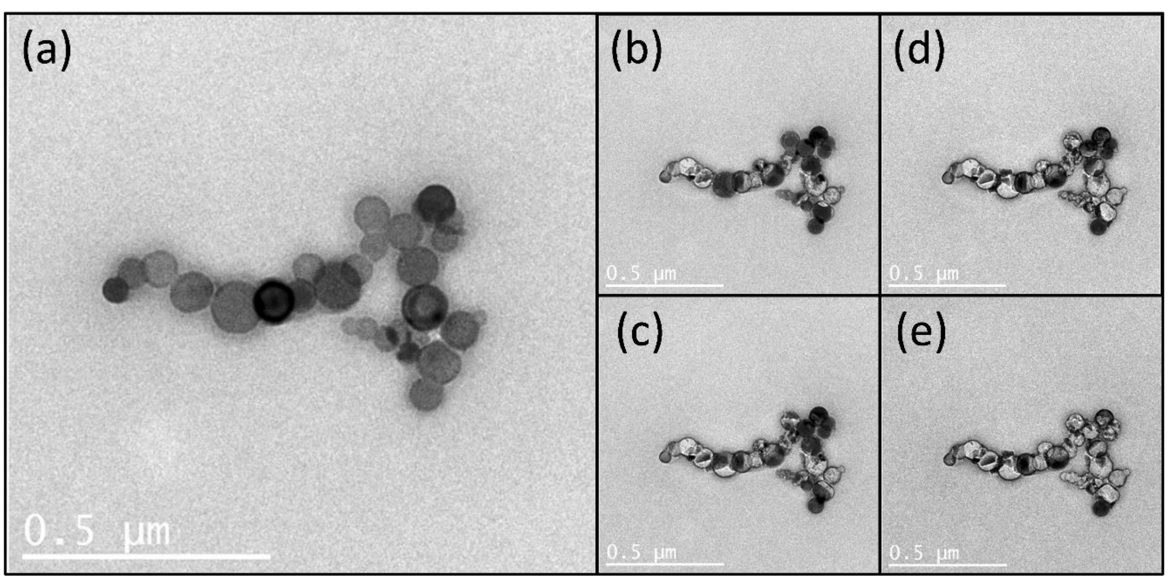

Figure 2. Sequential TEM images showing the reaction between the nano-Al and THV.

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