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Special Issue

DOD Researchers Provide

A Look Inside Nanotechnology

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Nanoenergetics: An Emerging Technology Area of National Importance

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Introduction

Energetic materials are a major component of weapons systems used by all branches of the US military. Their primary use is in explosives, as well as in gun and missile propulsion. Over the last century new chemicals have been discovered or designed for rapid release of energy in either relatively simple compositions, like that used in certain warheads, or in more complex formulations like the advanced composites used as propellants. Currently, a class of energetic materials known as nitramines are actually used for both explosives and propulsion applications in various weapons systems. Some major considerations for successful weaponization of energetic materials include performance (e.g. energy density, rate of energy release), long-term storage stability, and sensitivity to unwanted initiation.

As demands on munitions increase with regards to improved performance (i.e. increased lethality and survivability as well as the development of emerging high precision weapons concepts), the challenge on the R&D community is ever increasing. Additional drivers and concerns for the US military come from the continuing development of new munitions (including new types of energetics) by foreign nations. Munitions development is also fundamentally impacted by the approaching limit of the amount of improvement that is possible for the traditional and now rather mature C, H, N, and O energetic chemistries.

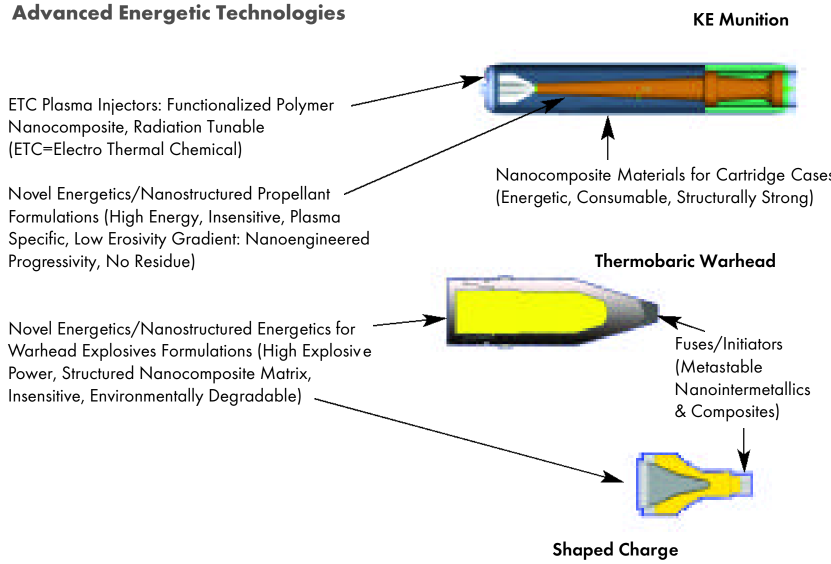
In recent years researchers have found that energetic materials/ingredients that are produced on the nanoscale have the promise of increased performance in a variety of ways including sensitivity, stability, energy release, and mechanical properties. As such, they represent a completely new frontier for energetic material research and development with the potential for major payoffs in weapons systems. Very simply, nanoenergetics can store higher amounts of energy than conventional energetic materials and one can use them in unprecedented ways to tailor the release of this energy so as to maximize the lethality of the weapons. The field of nanoenergetics R&D is quite young, but is already undergoing rapid growth. The goal of this article is to give the reader a sense for the physical and chemical characteristics and properties that make these materials so promising. This will be accomplished through a discussion of a few selected examples of current research.

Background

The 221st National Meeting of the American Chemical Society held during April 2001 in San Diego featured a symposium on *Defense Applications of Nanomaterials*. One of the 4 sessions was titled Nanoenergetics. This session featured speakers from government labs (DOD and DOE) and academia (for further information about this symposium, please contact the author). This session provided a good representation of the breadth of work ongoing in this field, which is roughly 10 years old. A number of topics were covered, including a few that will be discussed in detail below, namely Metastable Intermolecular Composites (MICs), sol-gels, and structural nanomaterials. The presentations given at this symposium largely form the basis for this report.

At this point in time, all of the military services and some DOE and academic laboratories have active R&D programs aimed at exploiting the unique properties of nanomaterials that have potential to be used in energetic formulations for advanced explosives and propellant applications. Figure 1 represents some concepts of how nanomaterials, especially

Figure 1. Weaponization of Advanced Energetic Technologies



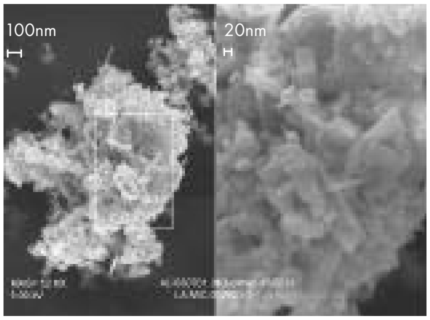


Figure 2. **SEM of MIC (Al/MoO_3 Mixture)**

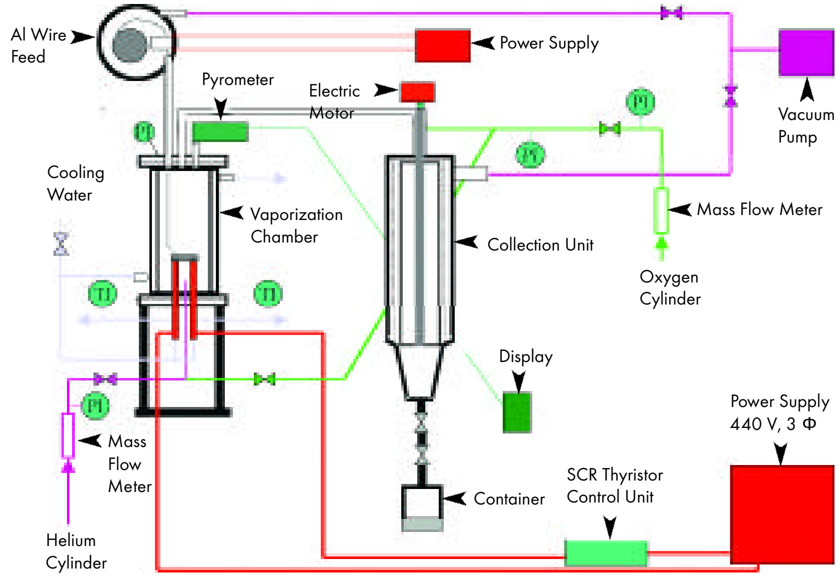


Figure 3. **The Process Schematic for the Production of Nanoscale Al for MIC Applications at NSWC/Indian Head**

nanoenergetics could be used for improving components of munitions. The figure shows that nanoenergetic composites and ingredients can be used in the ignition, propulsion, as well as the warhead part of the weapon. With regards to the latter application, nanoenergetics hold promise as useful ingredients for the thermobaric (TBX) and TBX-like weapons, particularly due to their high degree of tailorability with regards to energy release and impulse management.

Metastable Intermolecular Composites (MICs)

Metastable Intermolecular Composites (MICs) are one of the first examples of a category of nanoscale energetic materials which have been studied and evaluated to a considerable degree. MIC formulations are mixtures of nanoscale powders of reactants that exhibit thermite (high exothermicity) behavior. As such, they differ fundamentally from more traditional energetics where the reactivity is based on intramolecular (not intermolecular) properties. The MIC formulations are based on intimate mixing of the reactants on the nanometer length scale, with typical particle sizes in the tens of nanometers range (e.g. 30 nm). One important characteristic of MICs is the fact that the rate of energy release can be tailored by varying the size of the components. Three specific MIC formulations have received considerable attention to date; Al/MoO_3, Al/Teflon, and Al/CuO.

Research and development on MIC formulations is being performed in laboratories within all military services, as well as at Los Alamos National Laboratory (LANL). LANL researchers Drs. Wayne Danen and Steve Son, along with their colleagues, have not only pioneered the dynamic gas condensation method for the production of nanoscale aluminum powders (also known as Ultra Fine Grain [UFG]), but they have also conducted numerous studies on physical and chemical properties. As an example, Figure 2 shows a scanning electron microscope (SEM) image of a nanoscale MIC (Al/MoO_3 mixture) produced by the dynamic gas condensation process at LANL. One critical aspect of producing successful MIC formulations is the ability to produce nanoscale aluminum particles of small particle sizes in the tens of nanometer range, as well as with reasonably narrow size distribution. And, of course, the production process needs to be reproducible batch to batch. The current state of UFG aluminum production is that this is an area that still requires considerable effort. Even though there are commercial sources for UFG aluminum (such as the ALEX process originated in Russia, or commercial sources in other nations such as Japan), the need for reliable non-government sources of ingredient materials for uses in MIC applications is still there. Progress in this area is being made by companies such as Technanogy and Nanotechnology.

Another example of a significant effort at producing MIC compounds is found at the Indian Head Division of the Naval Surface Warfare Center (NSWC/IH). This work is being performed by Dr. Magdy Bichay, Pam Carpenter, and Tom Devendorf along with other co-workers. The Indian Head process for producing UFG aluminum is also based on the dynamic gas condensation process with some changes, such as the use of resistance heating instead of RF coils. Figure 3 shows

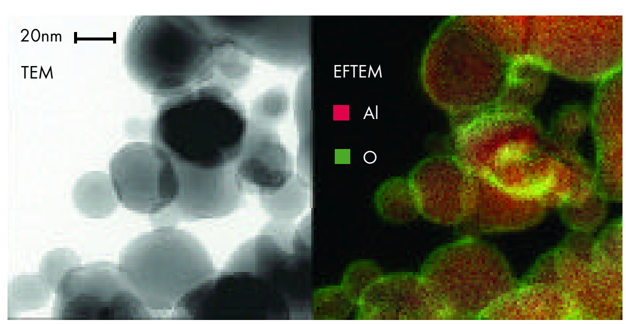


Figure 4. **Al Nanoparticles with a Passivation Layer of Al_2O_3**

the process schematic as developed by Professor Jan Puszynski of the South Dakota School of Mines, working in concert with Indian Head. Design goals included the use of continuous aluminum wire feed, a continuous collection system, as well as a production rate of approximately 10 grams/hour. It was found that one major limitation of the process was the 12 hour life of the titanium diboride/boron nitride ceramic resistance boat used for heating the material.

Figure 4 shows an example of the typical UFG aluminum that is produced by the Indian Head process. A transmission electron microscope (TEM) image of the Al nanoparticles is shown on the left while an EFTEM (energy filtered TEM) is shown on the right, clearly indicating a thin passivation layer of Al_2O_3. These images were taken at Lawrence Livermore National Laboratory (LLNL). In summary, much more research and development needs to be done in the production and characterization of these and new types of MIC formulations. Issues of MIC ignition and safety characteristics (such as impact, friction, and electrostatic initiation) are promising, but need to be fully explored. Overall though, certain key MIC characteristics are very attractive and quite promising for practical applications. These include energy output that is 2x that of typical high explosives, the ability to tune the reactive power (10 KW/cc to 10 GW/cc), tunable reaction front velocities of 0.1-1500 meters/sec, and reaction zone temperature exceeding 3000K. Specific areas of possible applications include use in environmentally clean primers and detonators, chem/bio agent neutralization, improved rocket propellants, IR flares/decoys, thermal batteries, and others.

Sol-Gels

Researchers at LLNL, Drs. Randall Simpson, Alexander Gash, et al., have pioneered the use of the sol-gel method as a new way of making nanostructured composite energetic materials. The advantages of making energetics on the nanoscale are shown in Figure 5 which provides a comparison between conventional energetic compounds (micron scale) and those which are composed of nanoscale ingredients. The sol-gel chemistry involves the reactions of chemicals in solution to produce primary nanoparticles, called "sols", which can be linked in a 3-dimensional solid network, called a "gel", with the open pores being occupied by the remaining solution. There are typically two types of sol-gels. "Xerogels" are the result of a controlled evaporation of the remaining solution/liquid phase, yielding a dense, porous solid. On the other hand, "aerogels" can be formed by supercritical extraction (SCE), which eliminates the liquid surface tension and thus alters the capillary forces of the egressing liquid that normally would lead to pore collapse. Since the pores have been largely kept intact through the use of the SCE method, the resulting solid is highly porous and lightweight, with excellent uniformity given that the particles and the pores are both in the nanometer range. Figure 6 illustrates the sol-gel methodology.

The sol-gel approach is fundamentally different than most approaches to energetic material production in that it is a relatively simple methodology (e.g. chemistry in a beaker) performed at low temperatures. It can also be relatively inexpensive

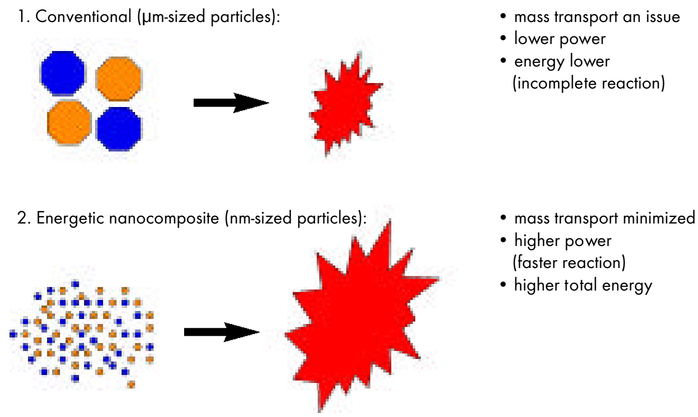


Figure 5. Composite Energetic Materials: Conventional vs Nanosized

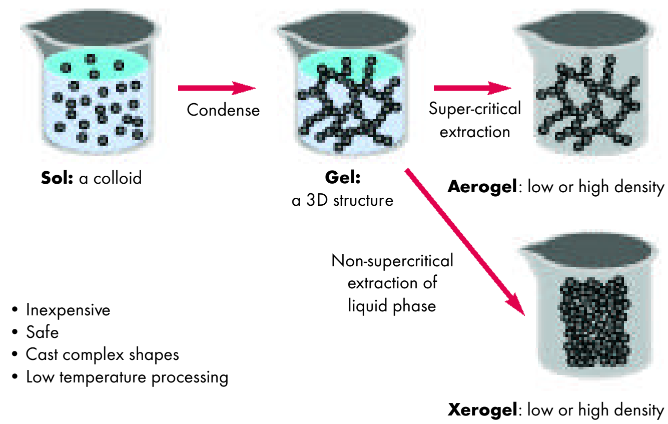


Figure 6. The Sol-Gel Methodology

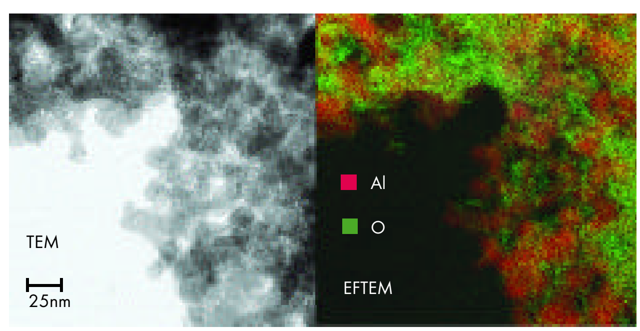


Figure 7. Sol-Gel Fe_2O_3/Al Nanocomposite

and has the promise of creating entirely new energetic materials with desirable properties.

One current promising nanocomposite being pursued by the researchers at LLNL involves the use of Fe_2O_3 which is generated using the sol-gel method. The reason that Fe_2O_3 is chosen is because its thermite reaction with UFG aluminum is very exothermic (with only CuO and MoO_3 yielding greater energy of reaction). An example of the high degree of mixing and uni-

Figure 8. **The Coiled Graphitic Structure of Carbon Nanotubes**

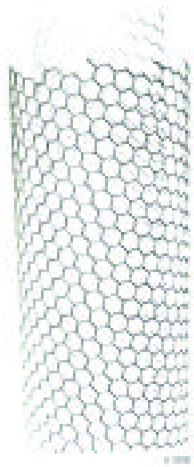
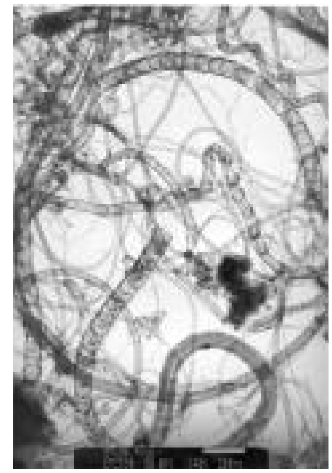


Figure 9. **Micrograph of Amine-Terminated Carbon Nanotubes**



formity between two nanophases is found in Figure 7, which indicates the excellent dispersion of Al and Fe on the nanoscale domain. The Fe_2O_3 was prepared by the use of an organic epoxide which was added to an Fe(III) salt solution resulting in the formation of nanoscale crystalline and amorphous Fe_2O_3. The reaction to produce Fe_2O_3 was done in solution which already contained the UFG aluminum. In this case, the nanoparticle aluminum was sonnicated (suspended in isopropanol and placed in an ultrasonic bath to break up any aluminum aggregates) before mixing with the Fe(III) salt solution. For this work, the UFG aluminum was supplied by the NSWC researchers at Indian Head using the dynamic gas-phase condensation method (discussed above), which yielded an average aluminum particle size of approximately 35 nanometers.

As sol-gel materials and methodology advances, there are a number of possible application areas that are envisioned. These include: (1) high temperature stable, non-detonable gas generators, (2) adaptable flares, (3) primers, and (4) high-power, high-energy composite explosives. In addition, the sol-gel chemistry may have advantages of being more environmentally acceptable compared to some other methods of producing energetics.

Functionalized Carbon Nanotubes for Energetic Applications

Dr. Lalitha Ramaswamy and her colleagues at the University of Maryland, along with Army Research Laboratory scientists (Drs. Matt Bratcher, Pamela Kaste, and Sam Trevino) are exploring the use of carbon nanotube structures (Figure 8) as starting material for various possible energetics applications. One concept involves the functionalization of carbon nanotubes with the notion of incorporating or binding this material into propellant matrices. The hope is that by doing so there will be significant performance enhancement in the areas of initiation, overall propellant performance, safety, as well as in mechanical properties. One specific goal that is being pursued is the assessment of carbon nanotube-based ingredients for the improvement of propellant initiation for either an advanced plasma-based initiator (which is under development), or for use with more conventional electrical initiators. Here the optical as well as electrical properties of nanotubes may be important in generating an improved propellant formulation. In particular, the high electron density that characterizes the nanotube structure as well as the high conductance along the tube wall may lead to more robust and reliable ignition behavior.

Long-term storage stability is one of the key elements of a successful propellant formulation. In this regard, there is hope that carbon nanotubes could be used to encapsulate nanoscale energetic ingredients, perhaps even the nitro-organic energetic compounds themselves (e.g. HMX, RDX), to yield a propellant that not only has the same (or better) performance for energy release, but also much improved performance for handling and long-term storage. An example of progress in the effort to functionalize carbon nanotubes is given in Figure 9 which shows a micrograph of some amine-terminated carbon nanotubes. In addition to the synthesis of functionalized carbon nanotubes this effort also involves chemical analyses of the products as well as the use of characterization techniques such as Prompt Gamma Activation Analysis for elemental ratio analyses.

Center for NanoEnergetics Research at the University of Minnesota

One of the major challenges that will be faced by the Department of Defense sometime in the near future (with regards to the utilization and implementation of nanoenergetic materials and ingredients) will be the ability to produce such materials in not only large quantities, but also in controlled sizes, size distributions, and chemical compositions. To address this eventuality, the DOD has recently funded a university-based research program in a competitive process through the Defense University Research Initiative on NanoTechnology (DURINT) program. This research program is headed by the University of Minnesota, which established the Center for NanoEnergetics Research (CNER) (see www.me.umn.edu/~mrz/cner.html). The primary goal of the CNER is to conduct a comprehensive and multidisciplinary study of the high rate production and behavior of nanoenergetics. Professor Michael Zachariah is the Director of the CNER and he has expertise in the synthesis and in-situ characterization of nanoparticles, as well as molecular dynamics simulations of particle growth and production. A significant aspect of this DURINT-funded program is the close scientific collaboration with a DOD research organization, namely the Army Research Laboratory. ARL researchers Dr. Barrie Homan and the author of this article are working closely with Professors Zachariah and Steven Girshick and their research groups in developing virtually identical nanoenergetics production and characterization facilities. Figure 10 shows a schematic of this research facility which represents the centerpiece of the CNER. It is based primarily on the use of a thermal plasma arc reactor for processing of a large number of possible precursors in the solid, liquid and vapor phases. In addition to the plasma, provisions have been made for the use of a furnace, as well as a diffusion flame apparatus, to serve as alternate techniques for producing nanoscale ingredients. In addition to the production of single or multicomponent (e.g. metals or multimetals) nanoscale particles, this facility allows for the in-situ coating of the particles using a number of different approaches (e.g. condensation or uv curing). This coating could be chosen to be an inert shield to keep the core nanoingredient from further reacting with air (oxygen or moisture), or it could be chosen to be energetic to dramatically increase the energy density of the final energetic formulation.

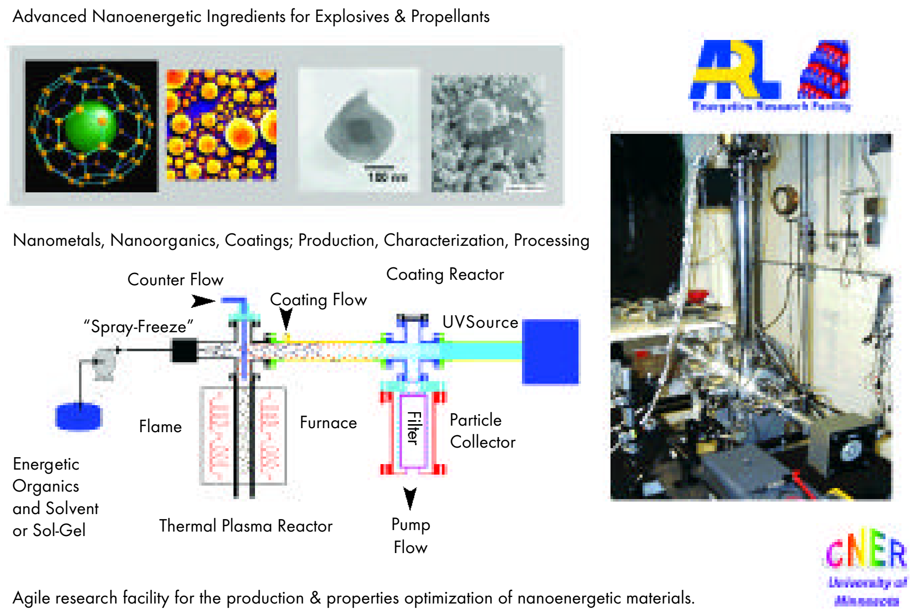


Figure 10. Schematic of Instrument to Produce Nanoscale Energetic Ingredients (Photo inset is the single particle mass spectrometer at the CNER- University of Minnesota)

Characterizing the nanoenergetic particles produced in this facility is accomplished by two major diagnostic approaches. The first involves the use of a single particle mass spectrometer (SPMS, inset photo in Figure 10). Here the SPMS is used to provide size and elemental composition of the particles as they are sampled from the reactive flow. As such, this tool tracks the growth as well as the coating of the targeted nanoenergetic ingredients/composites. Nanoscale particles of different sizes are selected for analysis using aerodynamic focusing. These selected particles can also be analyzed in-situ using spectroscopy. Two techniques in particular are powerful tools for analyzing small particles; Laser Induced Breakdown Spectroscopy (LIBS) and Laser Induced Incandescence (LII). LII has been developed primarily by the combustion research community as a major tool for understanding the formation of combustion-generated particulate matter. This technique utilizes a pulsed laser to rapidly heat the small particles. By monitoring the rate of decay of the resulting incandescent radiation, one can extract particle size information, as the rate is related to the size of the particle. In order to get information on the chemical composition of the particles through the use of spectroscopy, it is necessary to use a different technique. LIBS is an emerging major new tool for the analysis of chemical composition (see http://www.arl.army.mil/wmrd/LIBS).

In LIBS, a pulsed laser is tightly focused on the sample to induce a breakdown (microspark) of the sample material. The spark process leads to the breakup of the sample into its elemental components and simultaneous excitation of the resulting atoms and ions, which subsequently emit light. Thus, by monitoring the emission from this plasma, one can determine the nature of the element by its characteristic emission wavelength and the relative abundance by the intensity of the emitted light at a given wavelength. The LIBS sensor technology is advancing so rapidly that instrumentation has currently become available for the capture of light between 200-940 nm, a region where all elements emit. Thus, researchers are now in a position to simultaneously detect all of the constituent elements of nanoparticles, including the metals, carbides, and organic and/or oxide coatings. Since the LIBS data is generated in real-time (response time 1 second or less), one can keep track of rapid changes in the composition of the particles during the actual production run. Recently Professor David W. Hahn of the University of Florida has demonstrated LIBS analysis of nanoscale particles and has found that the LIBS technique can be very sensitive, having a resolution in the femtogram range (10^{-15} gm), and it is capable of detecting as few as 100 particles per cubic centimeter.

There are a number of other researchers and topics under the CNER umbrella. Professors Steven Girshick and Sean Garrick at the University of Minnesota are working on nucleation theory, aerosol dynamics, and the simulation of reactive flows while their colleague Prof. Alon McCormick, as well as Professor Tom Brill of the University of Delaware, are working on various aspects of sol-gel science. Other principal investigators include Professor Jan Puszynski of the South Dakota School of Mines who is working on solid-state chemistry and kinetics. On the theoretical side, Professors Don Thompson of Oklahoma State University and Don Truhlar of University of

Minnesota are working in the area of fundamental computations aimed towards the simulation of the behavior of nanocomposites. All in all, the CNER represents a major national resource for understanding the science and engineering as they relate to the generation and behavior of nanoenergetic compounds and ingredients. It should be mentioned that the Army Research Office (Drs. David Mann and Robert Shaw) had a major role in the selection of the DURINT topic on nanoenergetics and in the continued administration of the CNER contract which started in 2001 and could run through 2005.

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