High Energy Density Explosives

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A JASON summer study was performed to assess the status of ongoing research programs in the area of energetic materials. The goal of this study was to examine the current status of work in the area of energetic materials in order to provide DARPA with a technical evaluation of which, if any, research approaches have the potential to produce significant advances in the state of the art of this area.

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Contents

1 NEW APPROACHES TO HIGHLY ENERGETIC MATERIALS

Energetic materials play an important role as high explosives in nuclear weapons primaries and as key ingredients in nuclear weapon firing and delivery systems. Energetic materials also are important constituents of conventional weapons, playing a key role as propellants in weapon delivery vehicles and providing the explosive components of many conventional warheads. Research programs to develop new generations of energetic materials therefore exist in both the DoE and the DoD.

For any new energetic material, a delicate tradeoff exists between feasibility of synthesis and large scale manufacture, explosive performance, and safety. Materials with extremely high stored energy densities are of course the most desirable on a performance basis. However, materials with very high energy densities are often unstable thermally or present possible explosion hazards in response to shock. Such materials can not be safely made in the laboratory or deployed safely into the stockpile. The key to obtaining a new generation of energetic materials is to strike a balance between these competing criteria of performance versus manufacturability and safety.

The JASONs involved in this study heard briefings on a variety of topics. The topics presented included:

- the use of nanometer sized particulates to exert control over rates of thermite reactions;
- quantum chemistry-guided exploration of new high energy density materials;
- organic chemistry efforts targeted towards synthesis of specific molecular reagents hypothesized to represent the next generation of CL-20 high explosive;

- a proposal to use nuclear isomerization processes to obtain high energy release rates from existing radioisotopes;

- use of ultrahigh pressures to prepare metastable materials having high energy densities.

The remainder of this report provides feedback to DARPA on the challenges faced, and potential for important advances in energetic materials that are offered by each of these avenues of research, as deduced from the information presented to the members of this JASON study in July 1997.

2 METASTABLE INTERSTITIAL COMPOSITES: SUPER THERMITES

These materials are misnamed, and are more appropriately designated as controlled particle size thermite composites. The key to this approach is an innovative preparation method developed by Los Alamos, designated as dynamic gas condensation. This process allows synthesis of ultrasmall particles of Al. The Al particles are obtained by evaporation under controlled conditions of the liquid metal, and particles as small as 20 nm can be obtained under the proper conditions. The freshly formed particles are passed through an oxygenated gas stream in order to form a passivating Al oxide film on the surface of the particles. This passivation step allows the particles to be handled in air and subsequently mixed with an oxidizer such as MoO_3, potassium chlorate or potassium perchlorate. The composite exists in a stable, inert form until the mixture is ignited by heat, impact or spark.

Through control over the particle size, various different dispersions of metal and oxidizer can be formed, each possessing a characteristic reaction velocity. The reaction velocity is determined by the time required for the reactants to diffuse through the initial particles to the reacting surface, and is therefore approximately D/r, where D is the diffusion coefficient and r the particle size. This dependence is consistent with the experimental data, although there is also a material-dependent coefficient, perhaps the result of kinetic barriers to reaction. Los Alamos data on the reaction of Al with MoO_3 (to yield Al_2O_3 and Mo) show that the reaction velocity can be varied from < 1 m/s, i.e., in the conventional thermite regime, to $> 10^3$ m/s, i.e., approaching the conventional high explosive regime, through variation in the particle size of the thermite composite.

These results are extremely impressive and seem to represent a very promising approach to the design of new energetic materials. The question is what are these materials good for? Several applications have been suggested:

1. *Percussion primers for small caliber ammunition.* MIC materials can be ignited by percussion and can in turn ignite gunpowder. The mechanism of ignition has been suggested to be deformation of Al particles in regions of high stress, resulting in fracture of the passivating Al_2O_3 layer and contact between the pyrophoric fresh Al surface and either the oxidizing agent or oxygen in the air. Although not directly demonstrated, we consider this suggestion plausible. This application has been shown to be feasible. The advantage of MIC materials is that they produce no toxic products, in contrast to the lead-containing primers presently used. Potential concerns with MIC are the sensitivity of some compositions (Al/MoO_3, for example) but not all (not Al/teflon, for example) to friction and sparks. These may not be significant problems within a metal cartridge case, but for reasons of safety this needs to be investigated carefully.

2. *Additives to explosives and propellants.* MIC materials can increase the energy release, much as the conventional powered Al added to solid rocket propellant. It is unclear that it is, in addition, advantageous to add the oxidizing components of MIC, which are comparatively heavy. It is also unclear that ultrafine particles offer any advantage in this application over conventional powders, because the rate of burning is controlled by the matrix in which the MIC or ultrafine powders are embedded.

3. *Warheads and projectiles.* MIC materials have enthalpies of reaction per unit volume which are two to three times those of conventional high explosives, and enthalpies of reaction per unit mass which are 1.5 to two times greater. These numbers are impressive, but in contrast to high explosives, MIC materials do not produce great overpressure because they do not produce gaseous products. For this reason, although MIC materials burn rapidly they do not detonate; the burning front remains subsonic. If a nonreactive gas or vaporizable material is added, then a high overpressure may be produced, but the addition of the nonreactive material reduces the mean specific enthalpy of reaction. One application of MIC materials may be in incendiary munitions, in which a comparatively slow-burning MIC can serve as a source of ignition. It is unclear if there is any advantage over existing incendiaries.

4. *Low density materials.* Loose MIC powders have a remarkably low density (values of 2–5% of solid density have been observed, in contrast to values 20–50% for most conventional powders). This is likely to be a general property of ultrafine powders, resulting from the forces between small particles, and not specific to powders made of energetic materials. It is possible that such low density powders, made of non-energetic materials, may have novel applications. For example, these may include catalysis, filtration, as substrates for other surface chemistry and as shock-absorbing packing material.

Another key concern is whether the preparation of these materials can be scaled up to produce quantities sufficient for large scale use. The practical concern involves the reactivity of Al heated to its gas condensation operating temperature of 1600 °C, because at that temperature Al reacts chemically with most refractory metals and with many ceramics. Methods such as rf induction are now being explored, as are new materials for use in confinement of the molten Al. It seems reasonable to assume that these engineering obstacles could be overcome with a dedicated manufacturability program in place. In addition, it is useful to note that the field of colloid science is undergoing a modern renaissance and that chemically-based synthetic techniques are now becoming available for producing controlled particle size, nanometer-scale dispersions of metals, semiconductors, and insulators using straightforward wet chemical processes. It ought to be possible to utilize similar methods to produce controlled size, passivated particles of aluminum (or even of lithium possibly), and this strategy possibly offers an alternative, inexpensive approach to the formation of the ultra fine grain Al that is needed for these very interesting thermite composite-based high energy density materials.

3 NEW CHEMICAL SYSTEMS FOR HIGH ENERGY MATERIALS

The field of energetic materials from organic chemical syntheses is often considered to be mature. In fact, over the last 20 years, only incremental progress has been made in the performance of energetic materials that are actually deployed into the military stockpile.

Two different approaches were briefed to the JASON study group in addressing this issue. An ONR-led program is focused on developing the next generation of HMX/CL-20 materials, and is attempting to introduce relatively subtle molecular variations into existing explosives in order to improve their bulk energy density by 10-20%. The claim is that even such modest gains can produce significant gains in important performance characteristics such as penetrating power of a projectile. In an unrelated effort, quantum chemistry calculations by workers at Livermore Laboratory have been used to predict new classes of materials that might be highly energetic relative to existing materials. These materials are targets for synthetic efforts by bench chemists at LLNL.

In general, the research is being performed appropriately in that the workers realize that breakthroughs in this area will require a strong interplay between modeling and experimental efforts. Quantum chemistry models can, in principle, provide useful suggestions towards classes of compounds that can be made by a synthetic chemist which would possibly have a high stored energy per molecular unit. A further requirement, however, is that the compound must crystallize in a desired polymorph with a high gravimetric density, in order to have a high stored energy density in the bulk material. At present, quantum chemistry codes are not typically capable of predicting the unit cells into which compounds will crystallize in the laboratory. Thus, this aspect of materials design is often problematic. Additionally, quantum chemistry codes are generally unable to predict macroscopic properties that will be important to achieving a practically useful energetic material, such as

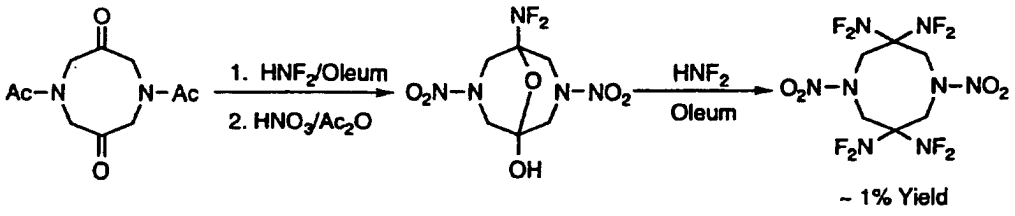
thermal stability or shock sensitivity. Computations can therefore provide a guide to classes of compounds that might be interesting energetic materials, but cannot be relied upon to predict which specific targets should be focused on in great detail. The latter requires a significant experimental synthesis program in order to evaluate numerous target materials in an expedient fashion.

The Livermore approach targets fused ring-based systems, such as furazan triazoles. The goal of this work is to identify radically different classes of materials that might display 150-200% increases in stored energies relative to conventional materials. This specific set of target molecules was arrived at based on achieving oxygen balance during the decomposition of the explosive, on packing considerations, and on bond strength calculations. The claim was made that the quantum chemistry calculation programs can reliably predict <10% differences in molecular enthalpies of formation, and this is probably correct given current state-of-the-art codes. Similarly, packing densities can probably be predicted to within 5%, provided that the desired polymorph is formed and provided that solvent molecules are not included into the crystal lattice. Flat molecules such as the fused ring systems are therefore expected to pack at a higher density than nonplanar ones, producing increased energy density for the same molecular enthalpy of formation. On the other hand, the claim is made that computations predict that these new molecules will be stable because their lowest bond dissociation energy is larger than that of TNT or HMX. This conclusion is suspect, because it is not clear that the reaction coordinate which is responsible for shock or thermal sensitivity is understood sufficiently to allow such a conclusion to be formulated. In fact, inspection of the briefing materials indicates that the bond dissociation energies calculated for these materials fall into the "very unstable" category that was obtained from empirical observations of the behavior of a variety of explosives. Also, given the bond strain of the five membered rings in the proposed molecules, combined with the degree of nitration in the final target molecules, it seems very reasonable to predict that the compounds will be on the borderline of thermal stability if they indeed are stable at all. Unfortunately, synthesis of any of the target molecules in this family of compounds

has not yet been achieved, so it is not possible to evaluate whether any of these predictions are borne out by experiment.

The ONR-led effort is focused on synthesis of *gem*-difluoroaminonitramines. These compounds are derivatives of the well-known HMX, RDX, and CL-20 compounds. Quantum chemistry codes indicate these new derivatives might have 10-20% higher energy densities, due to increased molecular energies of reaction as well as higher theoretical molecular packing densities. In addition, the NF_2 metal oxide fluxing ability is expected to result in complete combustion/detonation of Al and B-containing formulations, and some preliminary results have been obtained on linear organic compounds in which replacement of $-NO_2$ groups by $-NF_2$ groups did indeed produce an increase in propellant performance characteristics.

There are many possible NF_2 derivatives that one might target in order to validate the use of this functional group to obtain new energetic materials. The ONR program has logically targeted derivatives of HMX and RDX for this purpose. The target molecules have proven to be difficult to prepare in a straightforward fashion in high yield. One synthesis, described in scheme (1), has produced the desired compound in approximately 1% yield:



Various other schemes, including sequential introduction of gem-difluoroamino groups, and other precursor routes, are also being explored at the present time.

To our knowledge, despite the availability of small quantities of these

compounds, no data on the density or explosive properties of this material are yet available. At present, the compounds are entrained with solvent so that accurate density and enthalpy measurements are precluded. Such data can be collected at the earliest possible date. Only if the data are in agreement with the theoretical predictions should synthetic efforts should be increased to find higher yield, less expensive methods to make this target molecule.

In addition, it seems fruitful to pursue alternate strategies that do not involve the use of oleum (mixtures of H_2SO_4 and SO_3) to synthesize these compounds. Modern dehydrating anhydride/acid mixtures such as triflic acid/triflic anhydride etc. seem appropriate to force the protonation of the ketone oxygens in the starting materials of scheme 1 while also scavenging the released water and thereby forcing the desired fluoraminting process to proceed. This should be performed under a variety of forcing conditions in order to explore the range of opportunities presented by these relatively new synthetic methods to achieve the desired chemical transformations.

In general, it seems that the synthetic programs used to make these molecules are progressing relatively slowly. One of the reasons for this is that the community of researchers involved in the research is relatively small and is exploring reaction conditions in a serial fashion using a limited set of fairly well-proven synthetic protocols. For some functional groups of interest, there are few general methods available to workers in the area. Given the power of modern organic chemistry to make almost any stable small molecule target of interest, with the proper effort it ought to be possible to obtain samples of the desired possible high energy density materials within 12 months, and to perform a serious evaluation of the potential for these materials, or modifications thereof, to actually yield a breakthrough in new energetic materials. To do this most efficiently, if there is to be a focused effort in this field, the sponsoring agency should capture the attention of leading synthetic organic chemistry groups in the U.S., which would include researchers at Scripps, Columbia, Harvard, Berkeley, Chicago, Caltech, and Stanford, for example. These researchers should be stimulated to use the desired molecules as a target for developing new synthetic methodologies in

much the same way that these world-class researchers target natural products and potential drugs that are of interest to the pharmaceutical industry to develop new synthetic protocols of interest to the chemical community. These workers should then, after preparation of the initial target molecules, engage in feedback with the theoretical chemists in order to adequately benchmark the abilities of the quantum chemistry codes to predict the stored energies of various molecules of interest. In general, there might be an opportunity to obtain an interesting, and potentially significant, advance in this area, but assessing this possibility in a timely fashion will require a strong team and a well-managed collaboration between state-of-the-art synthetic chemists and theoretical chemists who are engaged in vigorously attacking the problem from a variety of synthetic angles using state-of-the-art tools of organic chemistry.

4 ENERGERIC MATERIALS FROM RADIOSOTOPE REACTIONS

A proposal was presented to the members of this JASON study to use nuclear isotope processes to prepare energetic materials. In brief, the notion is to couple nuclear transitions to electronic transitions in isomers such as ^{178m}Hf, and, in some ill-defined fashion, to then achieve a resonance condition through which electronic processes could be stimulated with high yield and with a rapid ($< 10^6$ sec) energy release rate.

There was a disturbing absence of a reality check in this proposal. There was no scientific justification of how such a process might possibly be obtained with high enough efficiencies to be useful in any practical process. The electron would likely be injected into the continuum, and even if the nuclear process were in resonance with an atomic electronic process, it is not clear how the energy release rate will be increased to any practically useful value. Before committing resources to such an experimental effort, there must be an adequate existence proof in the form of approximate, order-of-magnitude, estimates to justify investigating this effect. If such calculations have been done, they were not presented to the JASON study members, nor were the briefers aware of the existence of such estimates. Without such a defendable order-of-magnitude estimate of how the reaction rate will be increased to useful values, this approach seems to have no merit at the present time.

5 USE OF ULTRAHIGH PRESSURES TO CREATE METASTABLE, HIGHLY ENERGETIC, MATERIALS

The use of high pressures to synthesize novel, high energy-density materials is intuitively appealing. In addition to composition and temperature, the two variables traditionally emphasized in materials synthesis, pressure has the potential of leading to dense materials that contain high-energy bond configurations. Pressure is thus not only an added experimental dimension to explore, but can in principle lead to some of the specific properties being sought. The underlying concept is to use high pressure to achieve a transformation into a metastable material which will be highly energetic. The metastable material would, in this scenario, act as a storage medium for the pressure-volume work that was used to produce it. A simple existence proof of this possibility, albeit of a small magnitude, is found in the carbon allotrope transformation from graphite to diamond.

For such an effort to be worthwhile, however, three questions must have affirmative answers:

1. Can interesting (high energy-density) materials be produced at ultrahigh pressures?
2. Can they be retained at ambient conditions? and
3. Can the bond energies be accessed or released in a useful manner?

These issues are addressed in the discussion below.

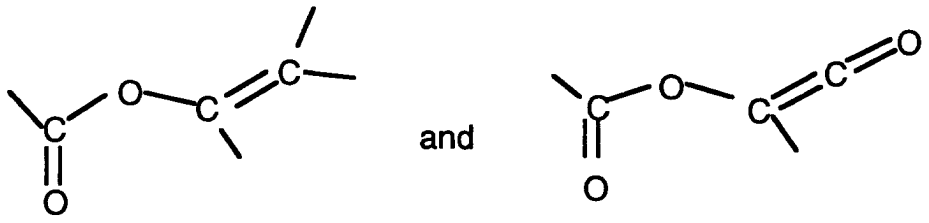
Although some theoretical work suggests that novel forms of nitrogen might be produced at high pressures, there is no assurance that such states are thermodynamically stable or, if metastable, that they are experimentally accessible. Instead, experiments yield evidence of phases different from those

predicted theoretically, including the new phases "1" and "2" of nitrogen. These and previous experiments provide no confirmation for the existence of the predicted novel states, and it is not advisable pursuing an extensive research program until (or unless) credible experimental evidence can be found for these states.

High-temperature synthesis does often result in metastable phases being quenched from high pressures to ambient conditions. However, there are numerous examples of phases that are not quenchable, even though they can only be formed at high temperatures and pressures. Therefore, there is no guarantee that high-temperature synthesis will produce a material that can be retained metastably to zero pressure, and one would expect lack of stability to be especially problematic for high energy-density states. It is worth noting that most of the experiments described in the briefing did not involve high-temperature synthesis, and that for the few high-temperature experiments which were described the synthesis conditions were poorly characterized and the state of the sample was not documented during synthesis. The quenched samples are likewise not well characterized, but show no indication of having potential as high energy-density materials. The third issue is complementary to the second, in that any dense phase produced at high pressures must be able to predictably release large amounts of energy upon initiation; it cannot be too (meta)stable. This is traditionally the key focus in developing new materials with the potential of releasing large amounts of energy, yet this issue has received no attention in the ultrahigh pressure research program described in the briefings. This means that even if a dense material with high-energy bond configurations can be created at high pressures, and even if it can be quenched to ambient conditions, there has been no consideration of whether or not this material could release the energy in a useful manner.

One interesting observation by workers at Livermore is the formation of an unidentified black solid upon pressurization of CO to 50 kbar. The claim was made that this is an energetic material, and its vibrational spectrum evidently does not correspond to a known organic compound. The structure

of this material has been tentatively assigned as a mixture of:



Even if correct, there is no reason, however, to believe that these compounds comprise an unusually energetic material. The bonding, as assigned by the investigators from the vibrational spectra, all involves conventional sp^2 and sp^3 hybridization on the atomic orbitals. This compound could almost certainly be prepared by conventional means of organic chemistry (if indeed it is a newly discovered material) or otherwise is unlikely to exist under atmospheric pressure/room temperature conditions. There would appear to be no evidence for unusual bond strains and consequent formation of a highly energetic metastable state at this point in time from the infrared spectra obtained to date. In addition, there is a prominent peak in the vibrational spectrum at 3300 cm^{-1}, suggesting the presence of -OH groups in the compound. This peak implies that neither of the above structures can accurately account for the actual composition of matter in the area being probed by the infrared beam during these experiments.

The focus on second row elements in these high pressure investigations is problematic. In one sense, these elements are appropriate candidates for a high energy transformation because the elements participate in strong covalent bonding in organic chemical compounds. This strong chemical bonding is also highly directional, however, so it will be difficult to maintain metastable materials that are prepared through a pressure-induced transformation to unpreferred atomic bonding geometries. In the case of graphite/diamond, the transformation is from sp^2 to sp^3 hybridization, both of which are preferred bonding geometries for carbon and other second row main group elements.

Linear bonding can of course be obtained from sp hybridization, but such carbon compounds exist in organic chemistry as polyacetylenes. These materials are saturated in their bonding, have conventional bond angles, and are not metastable compounds nor are particularly energetic materials. Forming an energetic network solid from carbon-containing systems would likely require 90 degree bonding angles around each carbon and would likely require unhybridized atomic orbitals, which have yet to be produced in a metastable state by high pressure transformations, to our knowledge.

In addition, any defect present in the material would be a site for detonation and might preclude trapping the metastable state of the material in significant quantities at room temperature/pressure. It is therefore not clear how one would scale up a pressure-induced transformation process to produce significant quantities of material, with unsolved challenges in both the engineering scale-up aspects as well as the domain size and defect density issues. There is thus potential merit in pursuing basic research on high-pressure synthesis, but the burden of proof that this procedure can produce an energetic material that can be trapped at room temperature rests with the researchers at this point in time.

6 SUMMARY AND RECOMMENDATIONS

In summary:

- the use of nanometer sized particulates to exert control over rates of thermite reactions is very interesting, logical, and might well find practical application;

- quantum chemistry-guided exploration of new high energy density materials and organic chemistry efforts targeted towards synthesis of specific molecular reagents hypothesized to represent the next generation of CM-20 high explosive are possibly interesting, but efforts to capture the attention of the state-of-the-art community of synthetic organic chemists in the best academic laboratories is needed in order to fully develop synthetic methodologies to reach these targets and to evaluate the promise, if any, of these classes of molecules;

- a proposal to use nuclear isomerization processes to obtain high energy release rates from existing radioisotopes had no rationale nor credible analysis that it was feasible according to our understanding of the laws of nuclear physics;

- use of ultrahigh pressures to prepare metastable materials having high energy densities is problematic and requires a proof of concept experiment before commanding significant resources towards its further development.

As a final comment on work on advanced energetic materials in general, we note that a possible application of work on developing new high energy density materials is the initiation of pure fusion weapons that are free of fission. Much public attention has been called to the possibility of such new pure fusion devices (Bethe letter, April 27, 1997 to President Clinton). It would be wise to think through and be prepared to discuss the possibilities,

and inherent physical limitations, of such devices as a potential outcome of this work, which as it progresses will inevitably be a lightning rod for political controversy. The DOE and the weapons laboratories should be prepared to face this issue, and the best approach is to develop an understanding of practical parameter ranges as set by known processes and energy conservation, for instance.

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