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13. ABSTRACT *(Maximum 200 words)*
The combustion of Hypergolic bipropellants in missile systems where the oxidizer and fuel are gelled involve important areas of research: ignition time delay, chemical kinetics, atomization, and liquid phase mixing. Measurements of the total ignition time delay for Hydrazine and Red Fuming Nitric Acid (RFNA) and UDMH/RFNA have been accurately measured using a droplet contact techinque. The results show the effect of fuel/oxidizer ratio and the total ignition time delay involves initial contact, quiescent mixing in the liquid phase, gas evolution and combustion in the vapor phase. The liquid phase mixing and the gas evolution comprise the most significant fraction of the total ignition time delay in comparison to the chemical ignition time delay. In addition, the results of atomization studies of gelled, Non-Newtonian Viscoelastic impinging jet sprays has indicated significant differences between Newtonian and viscoelastic fluids. The breakup and droplet formation of viscoelastic liquid sheets is largely affected by the directional nonhomogeneous nature of viscosity as quantified by the first normal stress difference. Also, Exciplex Flourescence imaging has been implented to visualize diffusion layers which form at the contact interface of mixing liquids.

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COMBUSTION DIAGNOSTICS AND FLOW VISUALIZATION OF HYPERGOLIC COMBUSTION AND GELLED MIXING BEHAVIOR

FINAL TECHNICAL REPORT

by

Dr. Douglas A. Feikema and Dr. James E. Smith

December 19, 1997

U.S. ARMY RESEARCH OFFICE

Grant No: DAAH 04-94-G-0265

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FORWARD

A new method to measure and examine ignition delays for hypergolic bi-propellants has been developed as part of this grant [1, 2, 3]. This technique, as a result of its time resolution, is the first to measure the true chemical performance of hypergolic bi-propellants. The chemical delay time is a fraction of the ignition delay time and represents the minimum time that hypergolic bipropellents need to react after mixing effects have been subtracted. Thus, for the first time, we have a technique that defines a chemical reaction delay time that may serve as a boundary condition for designers of hypergolic engines.

As part of this grant, a review of methods used by previous investigations were researched and critically compared to the new technique. Information obtained by the new laser diagnostic technique were not available from past methods. The results of testing unsymmetrical dimethylhydrazine, hydrazine, and furfuryl alcohol with red fuming nitric acid by this new technique have been published, with several of these papers attached as appendices. The measurements obtained are highly accurate and reproducible at the optimum oxidizer to fuel ratios.

The results of the time resolved ignition time delay measurements have demonstrated that the mixing time of classical hypergolic propellants is a significant fraction of the ignition time delay. This fact accounts for the large discrepancy of reported ignition time delay values reported in the literature. Contrary to conventional thought, the mixing is much greater than the chemical ignition time delay and hence the results of this study demonstrate that hypergolic bipropellant combustion is mixing limited. This concept is a significant result of this investigation and demonstrates that hypergolicity is not synomous with "spontaneous ignition upon contact."

As a parallel effort, liquid phase mixing as well as techniques for determining mixing have been investigated as part of this grant [4, 5, 6, 7]. In connection with the fluid mechanics effort, well characterized Newtonian and Non-Newtonian (i.e. Gels) fluids were characterized with respect to several parameters affecting the atomization and mixing of liquids. Two experimental configurations were investigated because of their relevance to practical considerations: 1) Impinging liquid jets and 2) Single droplets impacting a liquid pool. Several techniques have been implemented and developed as a result of this effort. Among these techniques the most noteworthy involves a unique application of Exciplex fluorescence for visualization of diffusion layers formed between mixing liquids. Time resolved images of Exciplex fluorescence have been obtained for the first time which provide new information on the duration of a mixing event as well as the length and time scales of the diffusion process. A linear stability analysis for viscoelastic, psuedoplastic Non-Newtonian fluids has been developed to predict atomization characteristics of gelled liquids.

In future studies, the chemical delay time period will be used to study the vaporization and combustion of hypergolic bipropellants free of the mixing regime. The

laser diagnostic system, equipped with both visible and near IR equipment will be used to obtain Raman spectra which will be triggered by the lead edge of the chemical time delay region of the ignition time delay period.

Also, future research should be directed toward further understanding the role of mixing in hypergolic combustion and the influence on ignition and chemical time delay. Better understanding of these mechanisms could lead to a better understanding of the toxicity associated with hypergolic bipropellants. Towards these ends, a new diagnostic technique, which enables visualization of liquid phase contact layers, has been developed.

The ARO funding, that was used to partially support three full time graduate students, and several undergraduate students. To date one Ph.D. and one Master's degree have been conferred with an additional Masters degree expected in May 1998. Four journal publications, ten conference proceedings and three interim technical reports were prepared documenting the above accomplishments. Additional publications are in preparation and pending publication at this time.

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STATEMENT OF THE PROBLEM STUDIED:

Gelled bipropellant propulsion offers several advantages of both liquid and solid propulsion. It offers the energy management of liquid propulsion and the storability and high density impulse of solid propulsion. Additionally, gelling a liquid propellant reduces the sloshing associated with the movement of the propellant in the tank. Previous research has noted the benefits of gelling a liquid propellant for military requirements for future propulsion systems; namely: safety, storability, and energy management [8]. Additionally, gelled bipropellants have been considered for Earth-to-orbit applications [9, 10, 11] and for use in space exploration missions [12, 13] where propellant density plays a crucial role.

Gelled bipropellants are formed by adding a gelling agent to a liquid propellant. The gelling agent is typically in the form of a swellable polymer or a particulate. The resulting fluid behaves in a non-Newtonian manner, i.e., the viscosity of the fluid is a function of the shear rate. Many of these fluids also have a yield stress which must be overcome before the fluid will flow. When a large enough shear rate is applied to overcome the yield stress, the propellants are injected and burned similar to neat liquid propellants.

A common method of injection for hypergolic propellants is the unlike doublet [14]. In this scheme, a jet of oxidizer impinges a jet of fuel thereby using the fluid momentum to atomize and mix the propellants. The mixing process is important in achieving maximum combustion efficiency and stable combustion. Previous researchers have noted the link between injector characteristics and subsequent combustion processes [15, 16, 17, 18]. However, there is still a basic lack of understanding regarding the processes controlling atomization, mixing and combustion [19].

The general problem encountered for designers of gelled bipropellant engines include issues of safety, combustion stability, storability, combustion efficiency, method of injection and mixing efficiency, and ignition time delay. In this investigation the research addressed the atomization and mixing issues as well as the ignition time delay.

OBJECTIVES:

Combustion of hypergolic propellants where the oxidizer and fuel are gelled, involve two important areas of research: combustion kinetic and mechanistic studies and atomization, mixing and flow visualization. The objective of the former is principally concerned with the reaction rate and ignition time delay of the oxidizer and fuel and represents a microscopic problem. Reactant contact area, atomization and flow visualization is concerned with the macroscopic fluid dynamic effects of the process important to combustion stability. The long range goal is to improve the overall

understanding of reaction mechanisms and the dynamic mixing needed to initiate and propagate hypergolic bipropellant gel combustion. This effort is significant for future improvements in efficiency, for understanding the combustion instability phenomenon and for reduced levels of toxicity.

APPROACH:

The approach taken in this research was to develop experimental techniques to consider both the microscopic and macroscopic effects in hypergolic combustion. Experiments and techniques were implemented to determine the ignition time delay of classical hypergolic propellants. In addition, atomization and mixing experiments were conducted with Newtonian and Non-Newtonian fluids in order to assess the atomization of gelled liquids. The two approaches complemented each other.

The results for two classical hypergolic reactions are shown in Figures 1 and 2 to illustrate the combustion diagnostics approach. Various reference points have been added to typical oscilloscope traces as shown in Figure 1 and 2 to identify key features. In these Figures the upper oscilloscope trace (channel 1) is the signal collected from a photodiode which determines the time of contact between liquids. The lower trace (channel 2) is the signal collected from a detector which measures flame luminosity and hence initiation and intensity of combustion. Point A on channel 1 represents the instance of contact between the fuel and oxidizer. At point B the falling droplet has completely entered into the other. Thus, the time between point A to point B represents the time from initial contact of the fuel drop to the time of final entry into the oxidizer. The response of the upper trace in the region from point B to C is representative of a still liquid/air interface. This is the region in which mixing is taking place between fuel and oxidizer by a combination of convection and diffusion between the two reactants. Region B-C, therefore, represents the time period when mass transfer and fluid mechanical mixing are occurring. In other words, the rate of reaction is limited by mass transfer and mixing. All previous reported techniques for measuring ignition time delay in the literature include this fluid mechanic mixing and mass transfer as part of the ignition time delay measurement. At point C the rate of chemical reaction increases rapidly. At point D the flame luminosity begins to increase rapidly. The region C-D represents the true chemical delay while region A–D represents total ignition time delay commonly referred to in the literature.

Figure 1 shows the oscilloscope trace for Hydrazine and Red Fuming Nitric Acid (RFNA). The total ignition time delay for is 22 msec, while for UDMH and RFNA as shown in Figure 2 the delay was 16 msec. This indicated that UDMA/RFNA reacts about 27% faster that Hydrazine/RFNA. If the chemical time delays are compared, namely 1.8 msec for UDMH/RFNA and 6 msec for Hydrazine/RFNA, then UDMH/RFNA reacts 70% faster. Both of these results are for oxidizer/fuel ratios of two. Thus, the chemical delay time, enables direct comparison of chemical performance of hypergolic acid-organic reactions.

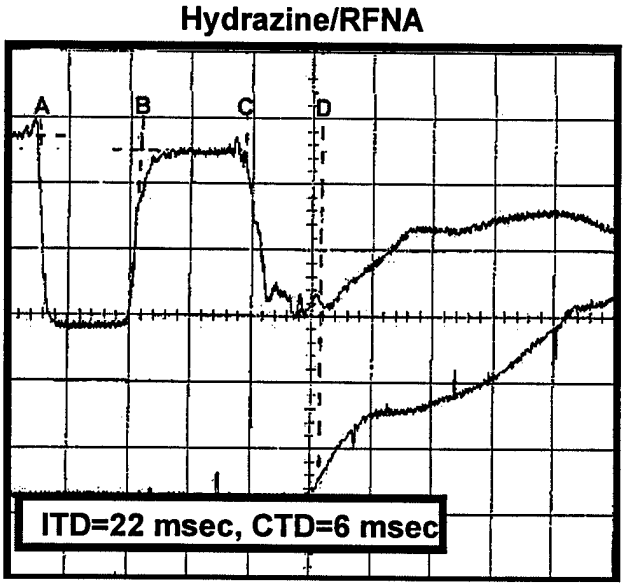

Figure 1: Oscilloscope Trace for Hydrazine and Red Fuming Nitric Acid
A: Droplets Initiate Contact; B: Falling Droplet Penetrates Fully;
C: Rate of Chemical Reaction Increases Rapidly; D: Flame Luminosity Begins.
Total Ignition Time Delay (ITD) = 22 ms; Chemical Ignition Time Delay (CTD) = 6 ms

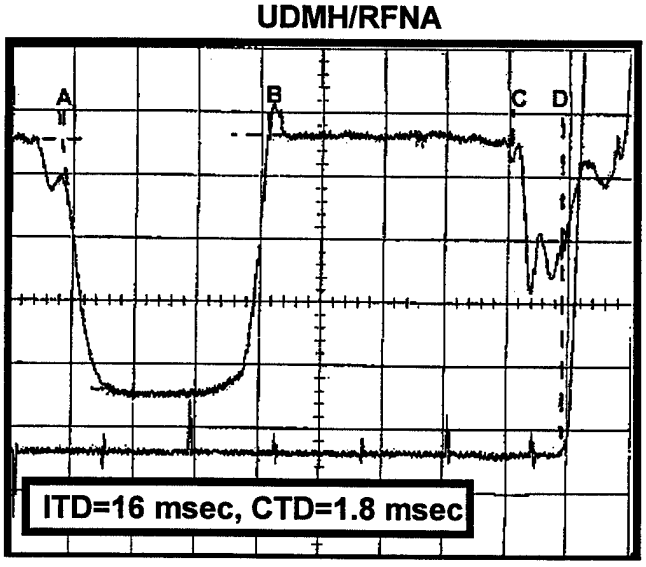

Figure 2: Oscilloscope Trace for UDMH and Red Fuming Nitric Acid
A: Droplets Initiate Contact; B: Falling Droplet Penetrates Fully;
C: Rate of Chemical Reaction Increases Rapidly; D: Flame Luminosity Begins.
Total Ignition Time Delay (ITD) = 16 ms; Chemical Ignition Time Delay (CTD) = 1.8 ms

Figures 3, 4, and 5 have been included to illustrate the approach taken to study the atomization and mixing effects. Figure 3 shows a liquid sheet produced by impinging jets with an impingement angle 2θ of 60° and jet velocity of 12 m/s. Some important points are to be noted concerning the structure of the liquid sheet generated. Two types of wave structures are observed which appear to be symmetric about the central axis. High frequency circular waves are generated at the point of impingement which have a wavelength of 6 to 9 mm. Also, cardiod waves are produced which are lower in frequency. Huang [20] and Taylor [21] also observed the presence of these waves in liquid sheets of water. The liquid sheet begins to breakup far from the impingement point. Ligaments are formed where holes in the sheet appear. The ligaments or liquid strings are not observed to form readily into drops; however, the liquid sheet is similar in structure to photographs of Dombrowski and Fraser [22]. When the sheet lifetime is long relative to the gel relaxation rate, which occurs for low speed phenomena, the fluid at locations far from the impingement region have a lower shear rate and hence higher apparent viscosity than at the jet exit and impingement regions. This effect would cause the secondary atomization effects to be slower than for Newtonian liquids. However, at high jet velocities, the gel relaxation rate is longer than the sheet lifetime and the liquid sheet would breakup and atomize much like a constant viscosity or Newtonian fluid.

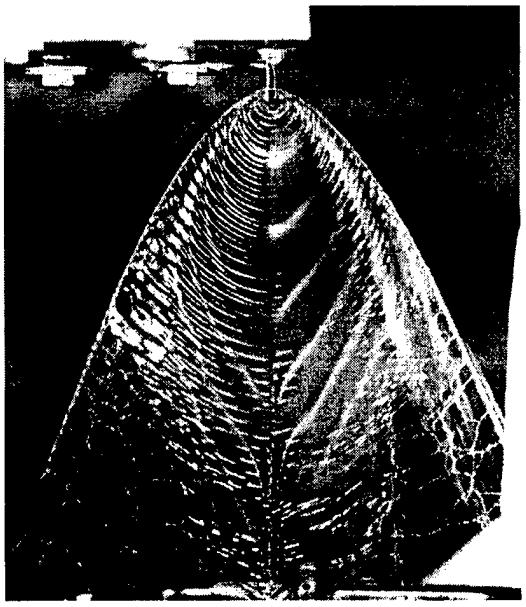

Figure 3: Gelled liquid sheet produced by impinging jets with an impingement angle 2θ of 60° and jet velocity of 12 m/s.

In order to study mixing with the B-C region defined above, an Exciplex fluorescence imaging technique was developed to investigate the mixing of a drop with a liquid pool. The experimental design for this set of experiments is illustrated in Figure 4. A pulsed Nd:YAG laser operating on the fourth harmonic at 266 nm and 40 mJ/pulse

illuminates liquid inside a quartz cuvette. A liquid pool is contained in a rectangular quartz cuvette with dimensions of 40 mm height, and 12 mm by 40 mm rectangular cross section. The cuvette was orientated such that the laser absorption path through the cuvette was 40 mm. The laser sheet optics consist of a spherical lens with a focal length of 500 mm and two cylindrical lenses with focal lengths of 50 mm and 127.1 mm. The resulting sheet has a thickness of approximately 100 μm and a height of 25 mm. The drops are generated by an eyedropper with an exit diameter of 1 mm and have a diameter of approximately 2 mm. The rectangular experimental arrangement inside the cuvette produces images which contain no index of refraction gradients except near the surface of the liquid and hence the images were not corrected for optical distortion. During the laser pulse, monomer excitation occurs inside the drop. Where the excited monomer in the drop and reaction partner in the pool bind, Exciplex fluorescence is detected by a Princeton Instruments Intensified CCD camera. An Oriel filter, Model No. 59355, with a 10 nm bandpass filter centered at 490 nm was positioned in front of a Nikon Micro 105 f 2.8 auto-focus lens to filter light outside of the range for Exciplex fluorescence. An area of approximately 9 mm by 6 mm was imaged onto the intensified CCD. The camera was gated with the pulsed laser to give a 100 ns intensity integration time on each pixel which began with the 5 ns laser pulse.

Figure 5 shows a drop of approximately 2 mm diameter which has penetrated a liquid surface. In the image Exciplex photoemission was collected for a total of 100 ns beginning with the 5 ns laser pulse. The intensity level is indicated with a gray scale. White in the images show regions of highest intensity where Exciplex fluorescence occurs. At these locations the binding of M* and G occur resulting in the Exciplex photoemission reaction. Black designates regions where no Exciplex fluorescence has occurred. The Figure demonstrates that the droplet is mixing with the liquid pool both at the diffusion interface and on the surface of the liquid pool. At the instant of the image collection, the interior of the droplet has not mixed with the liquid pool. Since the laser light sheet was propagating from left to right, the highest intensities (white) are typically on the left side of the images due to the absorption. This image illustrates that this method can be used to determine the location and spatial extent of the diffusion layers with the liquids. Such mixing zones are important in enabling understanding of liquid phase mixing phenomena which is particularly important for hypergolic bipropellant combustion.

Although not discussed here, the Exciplex technique described has been extended as part of this grant to collect time resolved images of the droplet mixing event. It was shown that the method can quantify the duration of a mixing event as well as the length and time scales of a diffusion process.

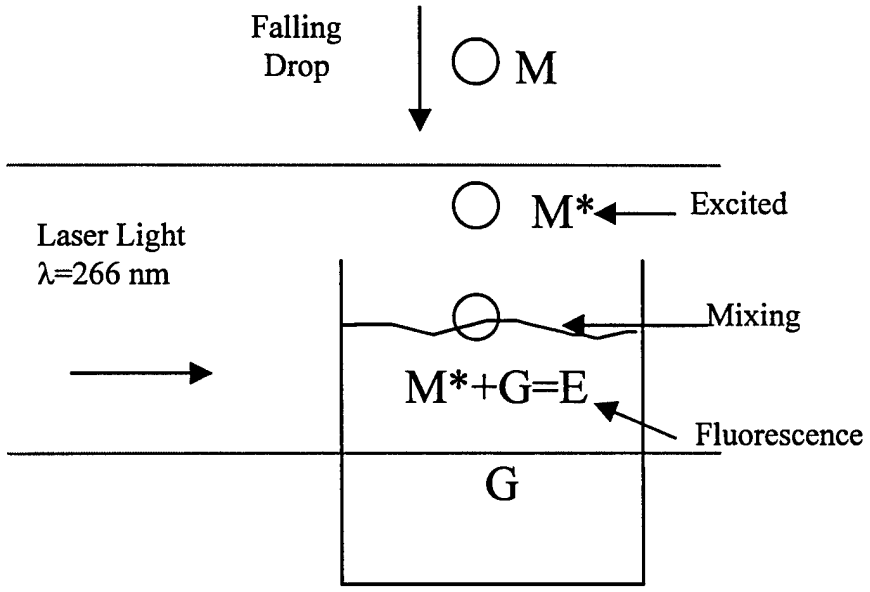

Figure 4: Schematic illustrating the implementation of Exciplex Fluorescence to study the mixing of a drop with a liquid pool and visualize the diffusion layers at the contact interface between the mixing liquids.

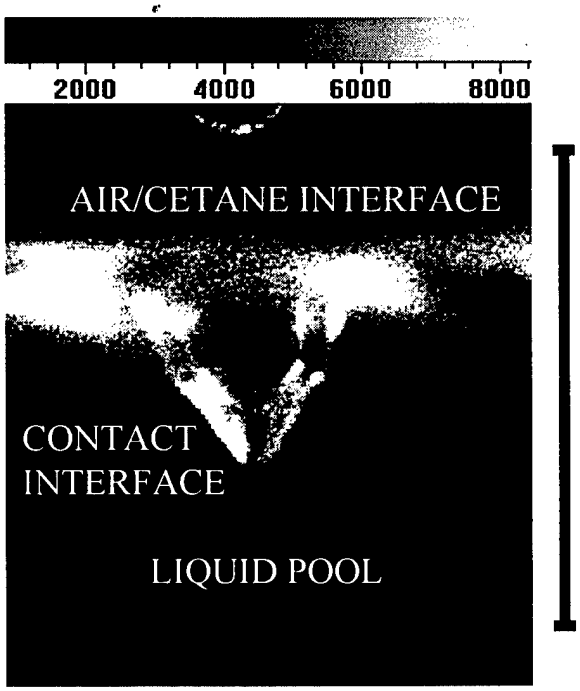

Figure 5: Exciplex Fluorescence Image Collected demonstrating the mixing and diffusional processes present between a drop and a liquid pool. The bar on the left side of the image has a true length of 8 mm.

SUMMARY OF THE MOST IMPORTANT RESULTS:

1. Contrary to conventional thought, the mixing time is much greater than the chemical ignition time delay and hence the results of this study demonstrate that hypergolic bipropellant combustion is mixing limited. This concept is a significant result of this investigation and clearly demonstrates that hypergolicity is not synomous with "spontaneous ignition upon contact."

2. A new method to measure and examine ignition delays for hypergolic bi-propellants has been developed as part of this grant. This technique, as a result of its temporal resolution, is the first to measure the chemical performance of hypergolic bi-propellants independent of the mixing technique employed. The chemical delay time resides within the ignition delay time and represents the minimum time that hypergolic bipropellents need to react after mixing and atomization effects have been eliminated. Thus, for the first time, we have a technique that defines a chemical reaction delay time that may serve as a boundary condition for designers of hypergolic engines.

3. Exciplex fluorescence was demonstrated within the diffusion layers and mixing zones. The technique yielded both spatial and temporal mixing information. Results show that Exciplex fluorescence has the potential to yield valuable new information in connection with the physics of liquid mixing processes. Careful selection of the concentrations of monomer and ground state reaction partner is required for the liquid phase to be considered optically thin. This method can quantify the duration of the mixing event as well as the length and time scales of the diffusion process.

4. Droplet formation, atomization and mixing is much more difficult to achieve for the viscoelastic (i.e. Gels) fluids. The reason for the observed phenomena is attributed to the directional nature of the viscosity in the fluids. The extensional viscosity dominates the breakup process and impedes the formation of droplets. Surface tension appears to play a minor role and therefore the Weber number is not an accurate non-dimensional parameter for correlating the results.

5. Fluid sheets formed by non-Newtonian jets breakup through the same wave structures as liquid sheets formed by Newtonian jets. At lower injection velocities, standing cardioid waves control the breakup process and at higher injection velocities circular waves emanating from the point of impingement dominate the breakup.

6. The linear stability analysis consistently overpredicts the fastest growing wavelength on the fluid sheet when compared to measurements. This can be attributed to the fact that the linear stability theory does not account for the high frequency instability resulting from the impingement process.

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2. Chojnacki, K. and Feikema, D.A., "Exciplex Fluorescence Imaging for Liquid Mixing Studies", accepted pending minor revision, ***Applied Optics***, 1997.

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4. Mays, L., Farmer, M., and Smith, "A Laser Diagnostic Technique to Measure Chemical Delay Time in Hypergolic Combustion", accepted by ***Combustion Science and Technology***, November, 1997.

5. Mays, L., Farmer, M., and Smith, "The Reaction Rate for Hypergolic Propellants using Chemical Delay Time", submitted to the ***AIAA Journal of Propulsion and Power***, May 1997.

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9. Mays, L., Farmer, M., and Smith, Jr., J.E., "A Laser Aided Technique to Measure Ignition Time Delays Between Hypergolic Bi-Propellants", extended abstract and invited presentation, pg. 78-79, 23rd Annual Conference of the National Organization for the Professional Advancement of Black Chemist and Chemical Engineers, Detroit, MI, April 9, 1996.

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LIST OF ALL PARTICIPATING SCIENTIFIC PERSONNEL SHOWING ADVANCED DEGREES:

1. Dr. Kent Chojnacki, Doctorate of Philosophy in Mechanical Engineering, Awarded August, 1997.
2. Mr. Mark Farmer, Earned a Masters of Science degree in Chemical Engineering, Awarded December, 1997.
3. Miss Lynnette Mays, Masters of Science degree in Chemical Engineering, Anticipated May, 1997.
4. Mr. Mathew Warren, Undergraduate MAE Student Assistant
5. Mr. Joel Kennedy, Undergraduate MAE Student Assistant
6. Mrs. Thien VU, Undergraduate MAE Student Assistant
7. Mr. Galen Gunther, Undergraduate MAE Student Assistant
8. Mr. Shawn Willingham, Undergraduate MAE Student Assistant

REPORT OF INVENTIONS:

No inventions or patents have been formally issued or filed at this time, however, the techniques developed as a result of this grant are unique and have never before implemented in the manner described to your knowledge.

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