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Fabrication of Graded Energetic Materials Using Twin Screw Extrusion Processing

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ABSTRACT

A new *Materials by Design* approach to creating energetic materials using Functionally Graded Materials (FGMs) concepts has recently been developed in a joint collaboration between the University of Maryland (UMD) and Indian Head-Naval Surface Warfare Center (IH-NSWC) through the Center for Energetic Concepts Development (CECD). This approach has been facilitated by previous efforts at IH-NSWC to apply a new process, known as Twin Screw Extrusion (TSE), for continuously manufacturing energetic polymer composites. It takes advantage of the continuous nature and superior mixing characteristics of the TSE process to manufacture a new concept for propellants and explosives: Functionally Graded Energetic Materials (FGEMs). For example, conventional geometrically-complex homogeneous grains for solid rocket motors can be replaced by a geometrically-simpler cylindrical FGEM configuration with an axial gradient in the energetic material formulation. The simpler geometry does not have the undesirable stress concentrations present in a conventional grain that end up reducing the reliability of the grain due to the formation of cracks, and the gradient in the energetic polymer composite formulation offers new possibilities to the designers of advanced energetics concepts that are not possible with the geometrically-complex homogeneous grains.

Since the fabrication of graded polymer composites with either inert or energetic ingredients has never before been investigated, a research program was initiated to understand the fundamental scientific and technical issues involved in the processing of these novel materials focusing on graded polymer composite propellants. Of primary concern is characterizing and modeling the relationship between the extruder screw geometry, transient processing conditions, and the gradient architecture that evolves in the extruder. Recent interpretations of the Residence Time Distributions (RTDs) and Residence Volume Distributions (RVDs) for polymer composites in the TSE are used to develop new convolution process models for predicting gradient architectures in the direction of extrusion. An approach was also developed for characterizing the sections of the extrudate using optical and combustion analysis to determine the gradient architectures and associated burning rates. The new process models and burning rate properties that have been characterized in this research effort will be the basis for future development of an inverse design procedure that is capable of determining gradient architectures for grains in solid rocket motors that possess tailored burning rate distributions that conform to user-defined performance specifications. It also serves as the basis for a new combinatorial materials science approach to formulating energetic materials.

INTRODUCTION

FGMs represent one of the latest developments in the *Materials by Design* revolution that is defining materials science in the 21^{st} century. They are being considered for employment in a wide variety of applications where conventional homogeneous composites are compromised against competing physical or chemical requirements. FGMs are microscopically inhomogeneous composites by design, in which the mechanical and other physical properties of the material are continuously or discretely graded from one surface to another. This is typically achieved in a single direction within a component by a continuously or gradually changing the composition of the materials. An often-cited examples are cutting tools and thermal barrier coating that possess gradients transitioning from a 100 percent ceramic surface to a 100 percent metallic interior to improve the wear or thermal resistance while maintaining the fracture toughness of the component (1). To adequately design, study, and optimize FGMS, materials scientists must be able to understand the gradient architectures that can be created in manufacturing processes, as well as the response of the gradient architecture to external stimuli, such as mechanical loadings and thermal shocks. Modern and future applications for these novel materials that are already under consideration include corrosion and radiation-resistant pipes for chemical plants and nuclear reactors, and thermally resistant superstructures for transatmospheric vehicles.

Recently, FGM concepts have become of interest to DoD scientists to improve the performance of energetic systems such as propellants, by replacing geometrically complex features of the energetic portion of a gun or rocket motor, known as a *grain*, with simpler geometries that improve reliability while meeting the desired performance requirements using a gradient in the energetic formulation. For example, a rocket motor with a functionally graded propellant offers new possibilities to rocket designers such as, simplifying motor systems by incorporating boost and sustain in a single stage, increasing kinetic energy on target impact, tailoring ballistic flight, etc. These new energetic material concepts that have gradients in formulation can be referred to as Functionally Graded Energetic Materials (FGEMs)

The collaborative research has shown that the volume fraction of 30 and 200-micron ammonium perchlorate (AP) particles can be varied along the length of the grain to produce a corresponding variation in burning rate properties. It is important to realize that the burning rate is related not only to the volume fraction of AP particles, but the particle size distribution as well. Both of these parameters, among others, are available to the motor designer/propellant formulator for compositional grading.

There is a great deal of interest in tailoring structures so the functional requirements can vary with location. In most cases, this will involve varying the materials that are used at specific locations within the structure resulting in discrete interfaces throughout. A number of manufacturing technologies have been proposed for the processing of FGMs. They can be categorized as either transport-based or constructive processes (1). Constructive manufacturing processes that have been currently used to manufacture FGMs include: powder densification, coating, and lamination. When this is accomplished in a batch-wise method, the result is discrete interfaces. These discrete interfaces are often weaker than the surrounding materials and also act as stress concentrators, a dangerous combination that can lead to structural failure. Transport-based processes include: mass transport, thermal diffusion, centrifugal separation, and melt infiltration. Generally speaking these methods result in continuous interfaces yielding a smooth gradient in material composition and microstructure. The current challenge for manufacturing FGMs is to develop scalable processes that can easily control the evolution of the gradient architecture within a structure in order to optimize structural performance. A description of the gradient architecture for composite materials can be seen in Figure 1 (2).

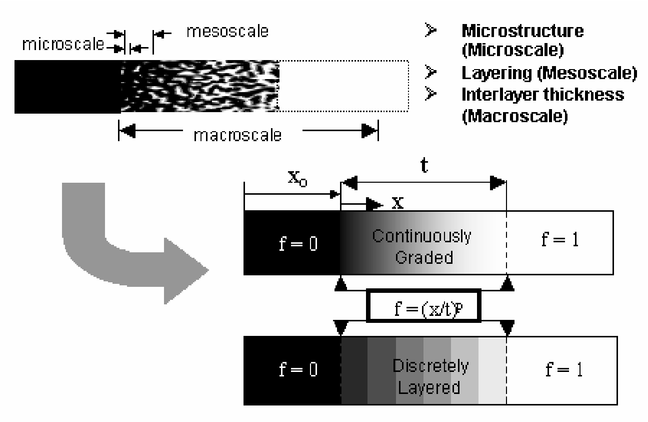

Figure 1. Description of gradient architectures for graded composites (2).

Prior to this research, Twin Screw Extrusion (TSE) processing is a manufacturing technology that had yet to be used for producing FGMs. TSE processing combines mixing and extrusion processes to enable the continuous manufacturing of finished products from raw ingredients in a one-step process. It has been primarily used to make polymers, polymer composites, and foods. Unlike a batch mixing process, where a fixed quantity of material is reconfigured in a transport-based process, raw ingredients are continuously fed into the TSE process where they are reconfigured into a finished product in a constructive manner. The raw ingredients can be varied continuously, or multiple extruders with different raw ingredients can be used to build up products with material variations. It is the former case that is of the greatest interest in manufacturing materials that possess continuous gradient architectures.

The research described herein serves as the basis for developing an inverse design procedure that is capable of determining gradient architectures for grains in solid rocket motors that possess tailored burning rate distributions that conform to user-defined performance specifications. The contributions made by this program to the inverse design procedure are highlighted by red in Figure 2 (COMMENT: where is this figure?). The research approach that was taken is multidisciplinary, involving both materials science (structure/property characterization) and manufacturing science (process characterization and modeling), and was primarily sponsored by an Office of Naval Research Young Investigator's Program (ONR YIP) granted to Professor Hugh A. Bruck with additional in-house research funding and support from the Indian Head Division of the Naval Surface Warfare Center (IHDIV/NSWC) for the energetic materials portion of the program. This research program was facilitated by a collaborative research agreement between UMD and IHDIV/NSWC that was administered through the Center of Energetic Concepts Development (CECD). The research was conducted at both the College Park campus of UMD and in the unique processing facilities of IHDIV/NSWC with the inert/energetic ingredients demarcating the two facilities. The research was conducted in three areas: processing science, materials characterization, and property /performance modeling.

CONTINUOUS PROCESSING OF POLYMER COMPOSITES USING TWIN SCREW EXTRUSION

TSE processes are utilized to manufacture a number of consumer and industrial goods from snack foods and medical tubing to plastic pellets and military propellants. The process is unique in that it combines advanced mixing characteristics and high extrusion pressures to continuously produce products with superior quality as long as the ingredient supply is maintained. Because this type of process has many advantages over batch type, it has found widespread utility across diverse industries. For most, however, the advantages are universal: economy, quality, and flexibility. These characteristics make TSE processing a very promising technology for producing functionally graded materials.

To understand the TSE process, a description of the equipment is necessary. The extruder consists of two screws, typically fully intermeshing, which run through temperature-controlled barrels as shown in Figures 2 and 3.

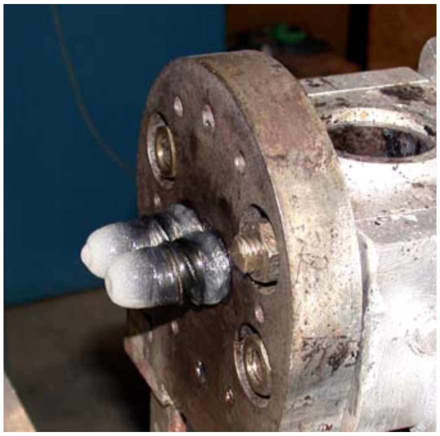

Figure 2. The ZDSK-28 (screw diameter in mm) in the University of Maryland Polymer Processing Laboratory shown without an extrusion die. Note the presence of polymer-melt on the screws and screw tips.

The barrels are modular in design and specialized for feeding solid and liquid ingredients, vacuum, or other functions. They are interchangeable, allowing for the screw elements to be configured so as to optimize mixing and transport for a particular material system. Furthermore the screws consist of various segmented elements that slide onto the screw shafts allowing for customizable screw designs. Like the barrel sections, various screw geometries are available which are utilized for conveying ingredients, gentle and high shear mixing, devolatilization, and many others. Hence the mixer is highly configurable and thus very flexible, allowing for the optimization of mixing and extrusion for a many different types of materials. In less than a work shift, the mixer can be reconfigured for a completely different product. This is especially important for energetic materials considering the wide range of new formulations that are being developed from new microscale and nanoscale ingredients (e.g., nanoaluminum, nanoRDX, composite particles) for advanced solid propellants and plastically-bonded explosives (PBXs).

This flexibility lends itself to facility expenditure savings for the processing of energetic materials. In alternative discrete batch mixing processes, a number of individual process steps are required, each with its own facility, equipment and operators. The continuous process with a twin screw extruder eliminates some steps and allows the rest to be combined. The result is a great reduction in the number of facilities required. Furthermore, the continuous process permits the process to be controlled in a manner that can yield a more consistent product, thus improving overall quality. For example, screw speeds and ingredient feed rates can be continuously maintained in a steady state operating condition to achieve significantly less variation in material, and the mixing quality can be enhanced over batch methods. The process also lends itself to on-line analysis using novel techniques like Near Infrared Spectroscopy (NIR) allowing for the quick detection of anomalous material properties and automatically diverting it to waste or altering the operating conditions to eliminate the anomaly (REFERENCE THOMPSON'S TR). In the batch process this is impossible until after the material has been completely processed and a large quantity of potentially bad product has been made.

Figure 3. One of three twin screw extruders at Indian Head Division, the pilot-scale ZSK-40 (mm) produced functionally graded solid rocket propellant at a conservative rate of 30 pounds/hr [change to grains/hr ??].

The extruder is only the heart of the process (Figure 4). Various ingredient feeders support the process. The quality of the extruded product is directly influenced by the accuracy of the ingredient addition. For this reason only the most accurate feeding technologies are utilized. These commonly include loss-in-weight control for solid ingredients and flow metering control for liquid. Other support equipment includes product collection. For the example process shown in Figure 4, the extruded strand is conveyed to a cutting blade where it can be separated into a finished product. Not shown are the temperature control units for the extruder barrels, on-line quality analysis instrumentation, and the process control system for the facility.

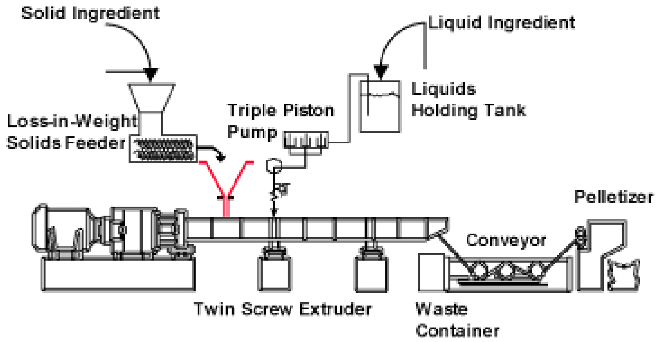

Figure 4. Typical facility components for a twin-screw extrusion facility.

TWIN SCREW EXTRUSION PROCESSING OF GRADIENT ARCHITECTURES

The TSE process is naturally suited to producing continuous composition gradients. Operating a twin screw extruder at one steady condition and introducing a step or ramp in the ingredients' feeding rates to produce a new formulation will result in the extrudate changing from the original composition to a second one. Because of the inherent backmixing in a twin screw extruder, an abrupt change in ingredients results in a more gradual change in the composition of the extruded product. There are several ways to achieve this for a polymer composite. It is possible to decrease the volume fraction of particle reinforcement, or to change the ratio of coarse to fine sized particles for a bimodal particle distribution. In this way, the continuous process is especially well suited to producing functionally graded materials. In contrast, batch mixing processes are incapable of producing a smooth continuous transition.

In addition to variations in the ingredients, the operating conditions of the TSE can also be varied. This can be used to control the extent and composition profile of the graded microstructure. It will also be possible to vary the screw configuration of the extruder to alter the characteristics of the graded microstructure. The effects of dynamic variations in ingredients and operating conditions for a given screw configuration must be modeled in order to predict the evolution of graded microstructures in the TSE process. Examples of graded polymer composite fabricated by changing the ingredients in the TSE process can be seen in Figure 5.

Figure 5. Graded polymer composites manufactured using the TSE process

RESIDENCE DISTRIBUTION (RD) MODELING OF TSE PROCESS

There are a number of research issues to understand the nature of the process that will ultimately lead to a predictive model. These are: (a) quantitative material characterization, (b) process modeling, and (c) a study of the coupling between the processing conditions and the microstructure that evolves. Material transport through a fully intermeshing twin-screw extruder is accomplished by screw geometry and screw motion (3). In other words the material flow is due to a combination of drag flow and pressure flow. The actual breakdown between each is highly dependent upon material properties, the geometry of screw elements, and the rotational speed of the screws.

In the case of polymer composites, the TSE is used as a mixer (or compounder) of ingredients, deaerator, and extruder in the same process step as material travels through the barrels. This aspect alone can result in material properties that are difficult to estimate and are dependent upon time and location in the process. Therefore calculations of drag flow and degree of fill are subject to approximation. For these reasons, the quantitative residence time of material in the system, characterized by the Residence Time Distribution (RTD), becomes a convenient way to express the cumulative effect of all processing and material parameters have on material transport. The RTD is typically normalized in order to describe the probability, *e(t)*, that a given quantity of material will reside in the extruder for a time, *t*, as follows:

$$e(t) = \frac{c(t)}{\int_0^\infty |c(t')| dt'} \qquad [1]$$

where *c(t)* is a filtered probe response obtained from the extruder (4). The RTD can be used to quantify various characteristics of the TSE process, such as the dampening that occurs as a result of backmixing in the extruder.

Under steady operating conditions there is a continuous supply of material conveyed to the mixing zones in the twin screw extruder, and an equal amount conveyed away. Much of the literature for the twin-screw process is characterization of the extruder operating in this steady state. The RTD therefore becomes the basis for characterizing the steady state transport of material within a twin-screw extruder.

In an excellent review of this body of work, Gao reports that the basis of RTD studies is to characterize the ability of the process to dampen transient disturbances, such as ingredient variations, feeder upsets, etc. (5). The answer is dependent upon the time scale of the disturbance. To characterize these time scales, an impulse disturbance is created by directly injecting a concentrated amount of a tracer material in solid or fluid form onto the rotating screws. This process results in the spreading and dilution of the tracer material in the extrudate that is a function of the residence time in the extruder. The diluted structure has a limited compositional variation that reflects one of the gradient architectures that can be achieved in the extrudate. However, to achieve more diverse gradient architectures, the processing of graded polymer composites will also require step or ramp changes in ingredient addition.

For the production of graded polymer composites, it is more important to understand how disturbances due to transient operating conditions will affect the composition within a specific particular volume of the extrudate rather than understanding their temporal effects. Gasner *et al* expressed the Residence Distribution (RD) in different domains beyond time, e.g., as screw revolutions and material volumes (6). Gasner used published data to illustrate these domain transformations. In a more complete study, Gao developed general forms of the RDs and conducted experiments to illustrate their applicability. The following function was used to describe the shape of normalized RTDs, *f(t),* as follows:

$$f(t) = \frac{a^n}{(n-1)!}\left(t - t_d\right)^{n-1} e^{-a(t-t_d)} \qquad [2]$$

The function consists of two parameters, the delay time, t_d, and a shape factor, a_x. Gao showed how these parameters can be estimated from knowledge of the screw geometry and a set of constants determined experimentally. The order of the function, given by n, represents the number of ideal mixers in series to model the response of the system to a disturbance. The function was extended and generalized during this research for $n > 2$.

A characterized RTD from one set of conditions can be used to predict the residence time at various other conditions given the same screw geometry profile. The power in this is that a certain desired microstructure in a graded polymer composite could be directly related to a specific RTD. Then the conditions to achieve that RTD could be estimated using Gao's set of relationships as defined by the parameters t_d and a_x.

Gao showed that in the volume domain [Equation 3], the residence volume distributions (RVDs) are independent of screw RPMs and volumetric throughputs and only dependent upon screw geometry. In this manner screw designs indeed could be uniquely identified (i.e., fingerprinted). This being true then it is possible to predict the RTD for different conditions once the RVD is established. These treatments were an important aspect of this project.

$$g(v) = \frac{c\left(\frac{v}{Q}\right)}{\int_0^\infty \left| c\left(\frac{v}{Q}\right)\right| dv} = \frac{e\left(\frac{v}{Q}\right)}{Q} \qquad [3]$$

$$g(v) = \frac{a_v^n}{(n-1)!}\left(v - v_d\right)^{n-1} e^{-a_v(v-v_d)} \qquad [4]$$

The delay volume, v_d, is given by,

$$v_d = t_d \times Q \qquad [5]$$

and the RVD curve shape parameter, a_v, shown below.

$$a_v = \frac{a}{Q} \qquad [6]$$

By converting the residence distribution model from the time to the volume domain, it is possible to directly describe the output of a specific volume of material from the extruder. This was essential to describing and predicting the spatial distributions of material in the gradient architecture.

PREDICTION OF 1-D COMPOSITION GRADIENTS IN TSE PROCESS

There were a number of research issues that were investigated as part of this research program to understand the fundamental nature of the TSE process that has led to a model capable of predicting gradient architectures. These issues included quantitative material characterization to study the coupling between the processing conditions and the gradient architectures that evolve in order to develop appropriate process models. For this research, it was demonstrated that the 1-D composition gradient, *f[z(v)]*, can be predicted by convolving the feed input conditions, *h(v),* for the extruder with the RVD model as follows:

$$f[z(v)] = \int_o^v g(v - v')h(v')dv' \qquad [7]$$

A series of experiments were conducted to explore the relationship between the evolution of the gradient architecture and characteristics of the TSE process. These experiments were conducted using two polymer-based composite formulations, an ammonium perchlorate (AP)-based formulation with an energetic thermoplastic elastomeric (ETPE) matrix material known as Hytemp that was processed at IH-NSWC and an inert analog that was processed at UMD using KCl in place of the AP and Engage$^{\text{TM}}$ in place of the Hytemp. The process response corresponding to the RTDs for the extruders were obtained at both locations using fiber-optic probes that were similar only in that they quantitatively measured light reflected from the tracer material. The results from these experiments are now discussed.

Using the RVDs (Figure 6) for the propellant containing 79 and 87 weight percent AP processed at screw speeds of 45 and 85 rpm, the parameters for the third order (n = 3) distribution function [Eq. 2] are 43.23 ±0.59 and 0.235 for a_v and v_d respectively.

$$g(v) = \frac{43.23^3}{(2)!}\left(v - 0.235\right)^2 e^{-43.23(v-0.235)} \qquad [8]$$

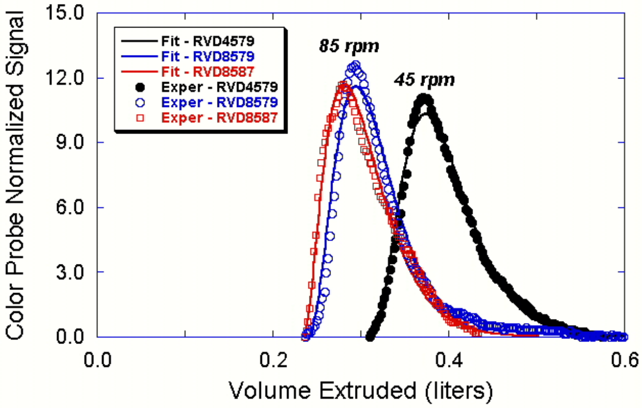

Figure 6. Residence volume distributions for composite rocket propellant as a function of AP concentration (79 vs. 87 percent by weight) and extruder screw speed.

From these RVDs, it was concluded that the parameters were insensitive to transience in the operating conditions (i.e., changes in the material properties did not affect the mixing characteristics of the extruder). Using these parameters, a fifth order convolution [Eq. 2 and 7] of the

RVD was found to best predict the gradient microstructure of the extruded propellant.

$$G(v) = 1 - \left(1 + \frac{a_v(v - v_d)}{(n-1)!}\right)^{n-1} e^{-a_v v} \qquad [9]$$

$$G(v) = 1 - \left(1 + \frac{43.23(v - v_d)}{24}\right)^{4} e^{-43.23v} \qquad [10]$$

To test the hypothesis, axially oriented strands, Figure 7, were submitted for determining the burning rate using the acoustic strand burner at IH-NSWC. An approach for characterizing burn rate was developed using strand lengths much less than the standard were tested to increase the spatial resolution of the measurement. The sample identification and location for each strand was painstakingly measured and recorded so that the results could be correlated to the original structure.

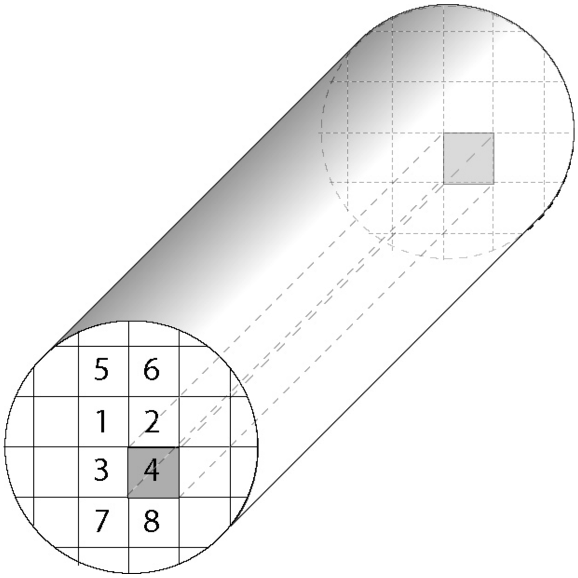

Figure 7. The four central axial strands (#1 – 4) were used to experimentally measure the burning rate along the direction of the gradient in the solid propellant.

All experimental results for grains produced by a positive step input are plotted in Figure 8. A positive gradient is made by beginning with the process in a steady state extruding composite with 79 percent by weight AP and changing the upstream feeding rate of AP to correspond to a final concentration of 87 percent. The resulting extrudate consists of a lower concentration of AP at the leading end and a higher concentration at the opposite end with a continuous gradient between the two homogeneous material. The data in Figure 8 show this change in AP concentration as a function of location in the grain.

There are two very important conclusions that can be made from the data. First, the convolution of the RVD function does an excellent job of relating a process model characteristic, i.e., the residence distribution, to the resulting material gradient produced by a step input to the feeding system. Second, the response of the extruder to the step change is highly reproducible. The 24 data points in the table represent samples sectioned from 12-inch strands taken from 4 different graded grains that were processed using both a positive direction (increasing solids loading) and negative direction (decreasing solids loading) for the step changes in ingredient addition. This has important implications on the manufacturing of graded composites, since it implies that there is no need to return to the original feed rate first before a gradient architecture can be continuously reproduced, simplifying the production of the graded composites and minimizing waste.

Another important conclusion that can be drawn from this data is obtained by comparing the burn rates obtained at various compositions in the graded grains with burn rates obtained from homogeneous compositions manufactured in the TSE process using a Design of Experiments (DOE) approach (Figure 10) (INCLUDE A REFERENCE TO OUR COMPOSITE MATERIALS PAPER WE ARE REVISING). From this comparison, the graded grain can also be looked at as a new combinatorial materials science approach for the developed of advanced energetic materials formulations using microscale or nanoscale ingredients (e.g., nanoaluminum, nanoRDX, composite particles). This is an exciting new capability for the formulations community is a simpler, faster, and more cost effective approach to developing advanced energetic materials than can be achieved using more conventional batch processes with a DOE approach.

CONCLUSIONS

A new *Materials by Design* approach to creating energetic materials using Functionally Graded Materials (FGMs) concepts has recently been developed in a joint collaboration between the University of Maryland (UMD) and Indian Head-Naval Surface Warfare Center (IH-NSWC) through the Center for Energetic Concepts Development (CECD). This approach has been facilitated by previous efforts at IH-NSWC to apply a new process, known as Twin Screw Extrusion (TSE), for continuously manufacturing energetic materials. It takes advantage of the continuous nature and superior mixing characteristics of the TSE process to manufacture new concepts for propellants and explosives..

Using this approach, an entirely new concept in energetic materials has been developed known as Functionally Graded Energetic Materials (FGEMS). These materials are fabricated by using transient operating conditions in the TSE process to achieve gradient architectures. Through this research effort, fundamental scientific and technical issues were addressed for the TSE process that enabled the process to be characterized under transient operating conditions, and also enabled the resulting gradient architectures to be predicted. The results of this research effort serve as the basis for the development of an inverse design procedure that is capable of determining gradient architectures for grains in solid rocket motors that possess tailored burning rate distributions that conform to user-defined performance specifications.

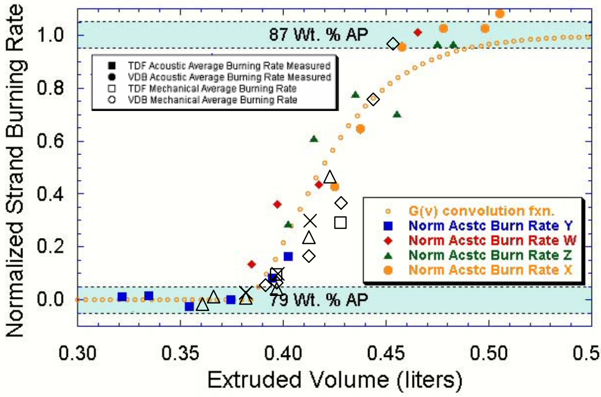

Figure 8. The individually measured burning rates were determined as a function of location (given by volume) along the gradient. The predicted response as given by the convoluted function is plotted for comparison.

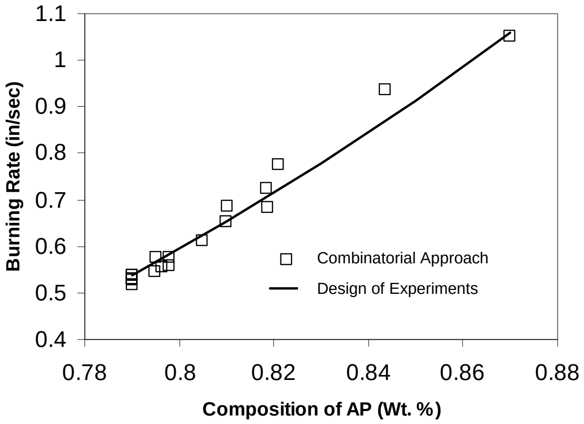

Figure 10. Comparison of burning rate versus composition from new combinatorial approach with variation determined by conventional Design of Experiments.

The effects of transience in the operating conditions for the TSE process were characterized using Residence Distribution (RD) models. These models were then convolved with feeder input conditions to predict the 1-D composition gradients that evolve in the TSE process. Experimental studies were conducted using a experimental composite rocket formulation consisting of ammonium perchlorate (AP) in an energetic thermoplastic elastomeric (ETPE) matrix material. In-situ optical measurements were used to characterize the RTDs and RVDs that evolved from a step change in ingredient addition. An approach was also developed for characterizing the variation of burn rate in the gradient architecture. The burn rate measurements were quantitatively similar to those predicted by using the RVDs in the convolution model, indicating that the convolution model has excellent potential for predicting gradient architectures in TSE-processed polymer composites.

For the extruded energetic material, the gradient architecture was characterized a posteriori using mechanical and acoustic strand burning tests. The results from these tests spanned the range of burn rates that were anticipated from the homogeneous tests, and correlated well with each other. Furthermore, it was determined that the gradient architecture was highly reproducible, evolving independently of the direction of the step change for the feed rate. This has important implications on the manufacturing of graded composites, since it implies that there is no need to return to the original feed rate first before a gradient architecture can be continuously reproduced, simplifying the production of the graded composites and minimizing waste.

Another important conclusion that was drawn from this work is the graded grains manufactured in the TSE process using can be used in a combinatorial materials science approach to the formulation of advanced energetic materials using microscale and nanoscale ingredients. This an exciting new capability for the formulations community that is a simpler, faster, and more cost effective approach to developing advanced energetic materials than can be achieved using more conventional batch processes with a DOE approach.

ACKNOWLEDGEMENTS

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REFERENCES

1. **Suresh S, Mortensen A. 1998. *Fundamentals of Functionally Graded Materials: Processing and Thermomechanical Behavior of Graded Metals and Metal-Ceramic Composites*. London: IOM Communications**
2. **Bruck HA, Evans JJ, Peterson ML. 2002. *Experimental Mechanics* 42: 361-71**
3. **Rauwendaal C. 1986. *Polymer Extrusion*. Munich: Hanser. 568 pp.**
4. **Gao J, Walsh GC, Bigio DI, Briber RM, Wetzel MD. 1999. *American Institute of Chemical Engineering Journal* 45: 2541-9**
5. **Gao J, Walsh GC, Bigio DI, Briber RM, Wetzel MD. 2000. *Polymer Engineering and Science* 40: 227-37**
6. **Gasner GE, Bigio DI, Marks C, Magnus F, Kiehl C. 1999. *Polymer Engineering and Science* 39: 286-98**

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