

# Highly Filled Materials Institute at Stevens

# news

Spring 2004

## 15<sup>th</sup> ANNIVERSARY ISSUE

### From the Director

Dear Friends:

**W**e are celebrating the 15th year anniversary of the establishment of our Research Center. We were founded in May of 1989 with the unwavering support of our President Harold J. Raveché as a Center of Excellence at Stevens.

I am very pleased to note that in the interim we have received over 120 research grants and contracts and the number of our Government and corporate sponsors has reached 70. The total count of students and staff who have worked at our Center has surpassed the 100 mark.



Unusual for an academic research center, our research has spanned multiple industries including energetics, ceramics, polymers and filled polymers, electronic materials, materials for magnetic shielding, conductive composites, personal care, automotive, foodstuffs, inks, batteries, diapers, toners, environmental remediation, rubbers and elastomers, encapsulation materials for chips and nanoparticles.

At this important junction and on behalf of the entire Center staff, I would like to express our gratitude to all of you who have supported our Center with your funding and input. We could not have done it without your contributions and we are counting on your support in the years to come.

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## New Capabilities

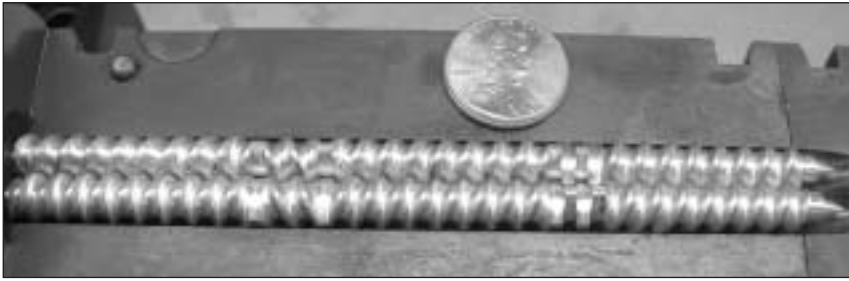
### Universal Extrusion Platform

The novel aspect of the platform is its ability to be converted into single screw or twin screw, co-rotating or counter-rotating, and fully intermeshing or tangential in both co and the counter. The unit has a splittable barrel with hydraulic quick release opening as well as consisting of segmented barrel sections to allow the length over the diameter ratio of the extruder to be tailored. This is an important capability that will allow us to test and recommend the correct technology given a new processing application. All of the modules of the Universal Extruder will also be accessible to mathematical modeling. This is probably the most versatile extrusion platform in the world and will allow us to rigorously compare various extrusion configurations, validate mathematical modeling results and will allow us to provide important scale-up criteria and manufacturing data for the processing of complex fluids for many industries. The Universal Extrusion platform is run remotely either using wireless technology or using Internet connections.



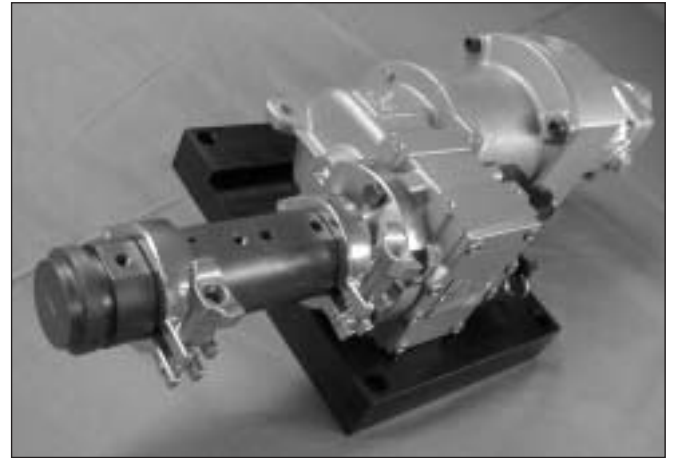
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## New Capabilities continued on from page 1



### **Smallest twin screw extruder in the World for Nanocomposites and Nanoenergetics**

This is a 7.5 mm twin screw extruder manufactured by MPR Inc. of Hackensack for ARDEC Picatinny, NJ. The unit is used for the manufacturing of nanoenergetics and nanocomposites (formulations containing nanoparticles). The capability is especially important for the characterization of the rheology and processability and generating samples when the ingredients are only available in small quantities. Some nanoparticles cost thousands of dollars per pound or are only available in multi gram quantities. The extruder is fully mathematically modeled using 3-D FEM and is used to generate the basic understanding of the extrusion of nanoparticle based formulations in partnership with ARDEC Picatinny.



### **Adjustable gap slit rheometer (off-line) with remote loading capability**

In 1980s we had developed our patented adjustable gap slit die rheometer as an on-line rheometer to characterize the shear viscosity and wall slip behavior of highly filled suspensions. An explosion proof version of the on-line rheometer is currently in service at NSWC/IH. This capability is now also transferred into our rheology laboratory as an off-line technology. The impetus for the new rheometer was the requirement to load samples into the barrel of the rheometer under vacuum, keep the sample sealed as they are brought to the test temperature and the shear viscosity and the wall slip behavior to be characterized simultaneously. The ability to load disposable cartridges on a remote basis under vacuum simplifies and renders safer the characterization of energetic materials and eliminates the air entrainment effect (a major structuring factor).

### **Squeeze flow rheometer**

We have expanded the mathematical models of the squeeze flow to allow the parameters of wall slip and shear viscosity material function of viscoplastic materials to be determined upon the solution of the inverse problem. Our current capability uses a touch-screen based control screen and can be run remotely either wireless or through the Internet. An explosion proof version of the rheometer was built and is being used at ATK Inc. at Radford, VA for gun propellants.

### **Enterprise modeling**

We have expanded our mathematical modeling capabilities by completely modifying (we have rewritten every FEM source code) and integrating our source codes into a form, which allows user-friendly implementation. Our simulation now cover tangential as well as fully intermeshing twin screw extruders and many die shapes.

## List of projects carried out since the last newsletter

2003 continued to be another excellent year for our research effort. Our research docket was again relatively heavy, as will be seen from our funding, the lists of completed PhD theses, grants and contracts and our publications and presentations. Currently, HfMI employs a total of 16 staff and research assistants. Here are some specifics of the accomplishments and happenings:

- During the last year we have completed our ARDEC Picatinny funded investigation of the shear roll mill process, using experimental and mathematical modeling means. We have understood the fundamentals of the process and designed and built an 85 mm prototype (first of its kind in the US). We have merged this processor with state-of-the-art thermal imaging and mathematical modeling capabilities. We are especially proud for identifying the mechanisms of the detachment of the melt from the roll as affected by its wall slip behavior.
- We have completed our work for ARDEC Picatinny in the area of the coating of the CL-20 particles. A new WAXD based technique was developed to characterize the coating thickness distributions. Furthermore, the techniques revealed uncoated areas in various particulates suggesting a means for the increased sensitivity of such CL-20 products.
- We have completed a major effort for ARDEC Picatinny in the area of process control of crystallization reactors, focusing specifically on the crystallization of RDX at BAE's manufacturing site in Holston, TN.

- We had earlier developed a new technology to allow the recycling of large quantities of Composition B explosive being discarded at various Army sites. Our invention disclosure had received the approval of the Patent Committee. However, the demand of such recycling appears to be decreasing and we have not pursued this technology.
- In the area of crystallization from solution we have received a two-year project from the Office of Naval Research for HNFx, a new energetic molecule HNFx. This project aims at the determination of the proper conditions for solution crystallization of HNFx. It also strives for generating the right polymorph and morphology upon molecular modeling based selection of the solvents and antisolvents used in crystallization.
- We have developed with ARDEC Picatinny a continuous process and appropriate extrudable formulations for the manufacturing of novel black powder formulations. Projects in the area of extrusion of an energetic thermoplastic elastomer binder and simulant development were also undertaken.
- We have also initiated or completed projects funded by Henkel-Loctite, IBM, a major battery manufacturer, and ink manufacturer, a toner manufacturer, and MPR, Inc. I.. Projects in the area of co-extrusion are also currently being carried out.

## New Staff

We are very pleased to announce that the following staff members recently joined our group.

**Dr. Nebahat Degirmenbasi** – received her PhD from Gazi University in Ankara/Turkey. She is a chemist and will be working in the areas of crystallization, characterization and molecular modeling.



**Dr. Zenaida Peralta-Inga** –

an expert in analytical chemistry and computational modeling, has received her PhD from the University of New Orleans. Prior to joining HfMI, she worked at the Advanced Materials Institute in New Orleans and at Louisiana State University Health Sciences Center.



## HfMI's Industry Day

An industry day to introduce the capabilities of our Research Center to the industry was held on May 20, 2003. The event involved presentations of our capabilities and research and incorporated the tours of our simulation, characterization and processing laboratories. The Industry Day was attended by over 50 participants from the industry representing over 15 companies covering the pharmaceutical, food, ceramic, environmental, machine design and manufacturing, energetic and other highly filled material processing industries. The opening presentation of the day was made by our President Dr Harold J. Raveché, who also introduced other happenings at SIT.



# Recent Publications and Presentations

## Publications in 2003

H. Tang and D. Kalyon, "Estimation of the Parameters of Herschel-Bulkley Fluid under Wall Slip Using a Combination of Capillary and Squeeze Flow Viscometers," *Rheologica Acta*, 43, 1 80-88 (2004).

D. Kalyon and H. Gevgilili, "Wall slip and extrudate distortion of three polymer melts," *J. Rheology*, 47, 3, 683-699 (2003).

D. Kalyon, "Letter to the Editor: Comments on "A new method of processing capillary viscometry data in the presence of wall slip" [*J. Rheol.* 47, 337-348 (2003)]," *J. Rheology*, 47, 4, 187-1088 (2003).

D. Kalyon, H. Gevgilili and A. Shah, "Detachment of the polymer melt from the roll surface: data from a shear roll extruder," accepted to appear in *International Polymer Processing* (2004).

D. Kalyon, "Twin Screw Extrusion Processing of Energetic Materials," *AICHE 2003 Annual Meeting Conference Proceedings CD-ROM AIChE-831*, Paper #90c (2003).

D. Kalyon, E. Birinci and H. Gevgilili, "Development of Extrusion Instabilities and Surface Irregularities of Extrudates of Polymer Melts and Filled Polymers," *AICHE 2003 Annual Meeting Conference Proceedings CD-ROM AIChE -831*, Paper #59c (2003).

G. Lu, D.M. Kalyon, I. Yilgor and E. Yilgor, "Coextrusion of TPU and BaSO<sub>4</sub> filled Medical-Grade TPU," *Society of Plastics Engineers ANTEC Technical Papers*, Volume 49 (2003).

G. Lu, D. Kalyon, I. Yilgor and E. Yilgor, "Extrusion of BaSO<sub>4</sub> filled medical-grade thermoplastic polyurethane," *Society of Plastics Engineers ANTEC Technical Papers*, Volume 49 (2003).

B. L. Greenberg, D. M. Kalyon, M. Erol, M. Mezger, K. Lee and S. Lusk, "Analysis of Slurry-Coating Effectiveness of CL-20 using a Novel Grazing Incidence X-ray Diffraction Method," *Journal of Energetic Materials*, 21, 185-199 (2003).

H. Tang and D. Kalyon, "Inverse Problem Solution for Parameters of Shear Viscosity and Wall Slip Using Squeeze Flow," submitted to *SIAM Scientific Computing Journal*, April (2003).

A. Wagner, D. Kalyon, R. Yazici, and T. Fiske, "Uniaxial Extensional Flow Behavior of a Glass Fiber-Filled Engineering Plastic," *Journal of Reinforced Plastics and Composites*, 22, 4, 327-337 (2003).

D. Kalyon, H. Gevgilili, R. Yazici, A. Post and G. McFann, "Flow and structure development behavior of bar soaps containing synthetic detergent," accepted for publication in *Rheologica Acta*, December (2003).

B. Greenberg, D. Kalyon, M. Erol, M. Mezger, P. Redner, K. Lee and S. Lusk, "Structural analysis of slurry coated CL-20 (PAX 12 granules) using a novel grazing incidence x-ray diffraction method," *Proceedings of Joint Army, Navy, NASA, Air Force Propellant Development and Characterization Meeting*, Charlottesville, VA, March 26 (2003).

S. E. Prickett, C Murphy, W. Thomas and D. Kalyon, "Shear viscosity, extrudate swell and wall slip behavior of a double base propellant," *Proceedings of Joint Army, Navy, NASA, Air Force Propellant Development and Characterization Meeting*, Charlottesville, VA, March 26 (2003).

D. Kalyon, G. Lu, I. Yilgor and E. Yilgor, "Rheology and Extrusion of Medical-grade Thermoplastic Polyurethane," *Polymer and Engineering Science*, 43, 12, 1863-1877 (2003).

G. Lu, D. Kalyon, I. Yilgor and E. Yilgor, "Rheology and processing of BaSO<sub>4</sub> filled medical-grade thermoplastic polyurethane" accepted to appear in *Polymer Engineering and Science*, January 2004.

## Patent

R. Yazici and D. M. Kalyon, "Method and Apparatus for x-ray Analysis of Particle Size" provisional application, U.S. Patent and Trademark Office, accepted December (2003).

## Presentations in 2003

D. Kalyon, E. Birinci and H. Gevgilili, "Development of extrudate distortions as affected by wall slip behavior of polymers and filled polymers," 2003 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 17, 2003.

D. Kalyon, "Twin screw extrusion of solid rocket fuels and propellants," 2003 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 20, 2003.

H. Gocmez, B. Greenberg, R. Schefflan and D. Kalyon, "Development of Methodologies for the Control of the Polymorph and Morphology of Crystalline Energetics," Second Advanced Energetics Technical Exchange, Aberdeen Proving Ground, MD, September 22, 2003.

G. Lu, D. Kalyon, I. Yilgor and E. Yilgor, "Extrusion of BaSO<sub>4</sub> filled medical-grade thermoplastic polyurethane," Society of Plastics Engineers Annual Technical Conference, Knoxville, Tennessee, May 6 (2003).

G. Lu, D.M. Kalyon, I. Yilgor and E. Yilgor, "Coextrusion of TPU and BaSO<sub>4</sub> filled Medical-Grade TPU," Society of Plastics Engineers Annual Technical Conference, Knoxville, Tennessee, May 5 (2003).

B. Greenberg, D. Kalyon, M. Erol, M. Mezger, P. Redner, K. Lee and S. Lusk, "Structural analysis of slurry coated CL-20 (PAX 12 granules) using a novel grazing incidence x-ray diffraction method," Joint Army, Navy, NASA, Air Force Propellant Development and Characterization Meeting, Charlottesville, VA, March 25 (2003).

S. E. Prickett, C Murphy, W. Thomas and D. Kalyon, "Shear viscosity, extrudate swell and wall slip behavior of a double base propellant," Joint Army, Navy, NASA, Air Force Propellant Development and Characterization Meeting, Charlottesville, VA, March 26 (2003).

D. Kalyon, "Rheology of concentrated suspensions," Corning Incorporated seminar, Corning, New York, June 26, 2003.

D. Kalyon, "Development of wall slip and flow instabilities," University of Akron, M. R. Kamal Symposium, April 25, 2003.

D. Kalyon, "Rheology and processing of highly filled suspensions," Levich Institute seminar, March 18, 2003.

## PhD Theses

Two of our graduate students completed their PhD theses under the supervision of Professor Kalyon in 2003. We congratulate Dr. Guangyu Lu (PhD thesis in the area of rheology and extrusion of filled and virgin medical grade polymers) and Dr. Halil Gevgilili (PhD thesis in the area of rheology and extrusion of complex fluids including filled polymers and soap dispersions).

