Thiokol Propulsion

JOCG Continuous Mixer Extruder Users Group Meeting
Thiokol Conference Center (TCC)

May 10-12, 1999

ATK
ALLIANT TECHSYSTEMS

Final Agenda for JOCG Continuous Mixer Extruder Users Group Meeting
Cordant Technologies Conference Center (CTCC)

Monday, May 10, 1999

5:00 – 7:00 PM Early Meeting Registration at Radisson Suite Hotel

Tuesday, May 11, 1999

7:30 – 8:30 AM Meeting Registration and Continental Breakfast at CTCC

8:30 AM **Introductory Comments**. Mr. Andrew Haaland, Thiokol Propulsion

8:35 AM **Formal Welcome**. Mr. Robert Crippen, President, Thiokol Propulsion

8:45 AM **JOCG Historical Perspective, Meeting Call to Order, and Keynote Speaker Introduction**. Mr. Kirk Newman, NSWCIHD

9:00 AM **Keynote Speaker**. Mrs. Mary Lacey, Technical Director, NSWCIHD

Morning Session, Tuesday, May 11, 1999
Mr. Andrew Haaland, Thiokol, Session Chairman

9:30 AM New Techniques for the Simulation of the Die and Twin Screw Extrusion Processing of Energetic Materials by D.M. Kalyon, S. Railkar, and A. Lawal (SIT/HfMI)

10:00 AM Simulation Based Design and Manufacture of Screw and Dies for the Energetics Industry by James E. Kowalczyk (Shear Tool, Inc.)

10:30 AM Break

11:00 AM On-line and Off-line Analysis of the Extruded and Cast Energetic Materials for Micro-structural Distributions by R. Yazici and D.M. Kalyon (SIT/HfMI). Presented by Dr. S. Railkar

11:30 AM Developing an On-line Quality and Process Control System for the Continuous Production of Energetics using Near Infared (NIR) Spectroscopy by Robert Thompson, Mark Michienzi (NSWCIHD) Presented by Mr. Mitch Gallant

12:00 PM Lunch at CTCC

Afternoon Session, Tuesday, May 11, 1999
Mr. Kirk Newman, U.S. Navy NSWCIHD, Session Chairman

1:00 PM A New Purpose-Built Remote-Controlled Capillary Extrusion Rheometer Facility for the Study of Highly Filled Energetic Materials by: R. Clift, M. Gough (DERA Ft. Halstead) and R. Carter, G. Privett (Magna Projects & Instruments Ltd)

1:30 PM A New Squeeze Flow Rheometer for the Rheological Characterization of Energetic Materials by D.M. Kalyon, B. Karuv, J. Garrow (SIT/HfMI)

2:00 PM Break

2:30 PM Novel Slit Rheometer for Energetic Extrusion Applications at TACOM-ARDEC, by Fevci Zeren (Zeren Research) and Sam Moy (TACOM-ARDEC)

3:00 PM Processing of Energetic Materials at Thiokol's 19mm Twin Screw Extrusion Facility by Andy Haaland and Quinn Barker (Thiokol)

3:30 PM Break

Tuesday, May 11, 1998 (Continued)

4:00 PM	Processing of PAX-2A Using a Twin Screw Mixer/Extruder, by Kristin Jasinkiewicz (TACOM-ARDEC)
4:30 PM	**Invited Guest Speaker** Gary Smith, Manager, Process Development, Autoliv ASP, Inc. Twin Screw Processing of Gas Generants for Air Bag Applications at Autoliv
5:30 PM	Cocktail Reception at CTCC
6:00 PM	Dinner at CTCC

Wednesday, May 12, 1998

7:30 – 8:00 AM	Meeting Registration and Continental Breakfast at CTCC

Morning Session, Wednesday, May 12, 1999
Mr. David Fair, U.S. Army ARDEC, Session Chairman

8:00 AM	Achievements of the Continuous Processing of Composite Propellants (CPOCP) Project by Eric Giraud (SNPE)
8:30 AM	Continuous Processing of Green Energetic Materials (GEM) Gun Propellant by Mitch Gallant (NSWCIHD)
9:00 AM	Processing Energetic Materials Through the Totally Integrated Munitions Enterprise (TIME) Network by Dave Gilson (TACOM-ARDEC)
9:30 AM	Break
10:00 AM	Explosion in Propellant Production Plant by Per Lagerkvist (NEXPLO Bofors AB)
10:30 AM	A Method of Isolating Energetic Material Feed Systems from Extrusion Processes Processing Results at Thiokol's 58mm Twin Screw Extrusion Facility by Mike Rose (Thiokol)
11:00 AM	Development of Processes Suitable for Manufacturing of Molding Powders to be used as Extrusion Raw Material Feed Streams by Jeff Widener (Thiokol)
11:30 AM	Injection Loading Technology: An Automated Unit Operation for the Automated Agile Manufacturing Facility by Kirk Newman (NSWCIHD)
12:00 AM	Lunch at CTCC
1:00 PM	Travel to Thiokol Plant
2:00 PM	Start Tour of Extrusion Facilities
4:30 PM	End Tour of Thiokol Plant

Processing Equipment Vendors Invited to JOCG Conference
5 May 99

1.) Extruder Vendors

Mr. Jan Levi
B&P Process Equipment
1000 Hess Street
Saginaw, MI 48601
Phone: 517-757-1391

Mr. Mike Starer
Krupp Werner & Pfliederer
663 E Cresent Avenue
Ramsey, NJ 07446
Phone: 201-825-6414
E-Mail: Mstarer@WP-Corp.com

2.) Feeder Vendors

Mr. Andy Kovats
Brabender Technologie Inc
6500 Kestrel Road
Mississauga, Ontario
Canada L5T 1Z6
Phone: 905-670-2933

Ms. Kathy Hunter
Ktron America
Route 55 & Route 553
Pitman, NJ 08071
Phone: 609-589-0500
E-Mail: Khunter@Ktron.com

Mr. Scott Campbell
Acrisson Area Representative
Camtec Industrial Sales
P.O. Box 1700
Sandy, Utah 84091
Phone: 801-566-6000

Ms. Gina Donnelly
Accurate
P.O. Box 208
Whitewater, WI 53190
Phone: 800-558-0184
E-Mail: Donnelly@SARInc.com

3.) Post Processsing Equipment

Mr. Frank Romeo
Romeo Engineering
4217 Hahn Blvd
Ft. Worth, TX 76117
Phone: 817-656-0048

NAVAL SEA SYSTEMS COMMAND

NSWC

Naval Surface Warfare Center

Indian Head Division

Historical Perspective

JOCG Continuous Mixer & Extruder Users' Group

Presented at the 10th Extruder Users' Group Meeting

Ogden, UT, 11-12 May 99

by

Kirk Newman

Navy Co-chair, JOCG CMEUG

NSWC Indian Head, Code 930

Yorktown, VA 23691-0160

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NSWC
Naval Surface Warfare Center
Indian Head Division

Chronological Record

of the Joint Army / Navy Continuous Mixer & Extruder Users' Group

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This is the 10th meeting of the CMEUG. These meeting have been held in hotel conference rooms and usually feature a tour of the host facility. The 5th was the first to be a JOCG event. The meetings were as follows:

[1] NSWC White Oak, Silver Spring, MD, 30 Nov - 2 Dec 87 (Ottmar Dengel)
[2] Radford AAP, Radford, VA, 6-8 Dec 88 (Carl Chandler)
[3] NOS Indian Head, Oxon Hill, MD, 5-7 Dec 89 (Tony Dunay)
[4] LAAP and LHAAP, Shreveport, LA, 4-6 Dec 90 (Lee Estabrook & David Dillehay)
[5] NOSIHDET @ NWS Yorktown, Williamsburg, VA, 2-4 Dec 91 (Kirk Newman)
[6] UPCO, Phoenix, AZ, 16-18 Nov 92 (Hugh McSpadden)
[7] LHAAP, Marshall, TX, 15-17 Mar 94 (David Dillehay)
[8] NSWCIHD, Arlington, VA, 20-22 Sep 95 (Connie Murphy)
[9] HfMI/SIT, Lincoln Harbor, NJ, 2-4 Jun 97 (Dilhan Kalyon)
[10] Thiokol Propulsion, Ogden, UT, 11-12 May 99 (Andy Haaland)

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JOCG Charter

of the Continuous Mixer & Extruder Users' Group

GOAL = Understand the technology of continuous mixers and extruders sufficiently for their implementation into an automated and agile facility for the manufacture of energetic materials

- Who? - Technical managers, engineers, and scientists
- What? - Discuss facility design, equipment, processing, and safety
- Why? - Technology is interdisciplinary, and implementation requires cooperation
- How? - Open meetings (papers cleared for public release)
- When? - Annually, if appropriate
- Where? - DOD co-chair identifies a host and coordinates a meeting site that includes a tour

"Good news! We know one
more thing that won't work!"

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Evolution of Technology

for Continuous Mixers & Extruders

Depends upon our ability to solve problems

- *Materials*
  - Insensitive Munitions (IM)
  - Resource Reclamation & Reuse (R^3)
- *Applications*
  - Performance Requirements
  - Geometry Requirements
  - Operational & Storage Requirements
- *Economics*
  - Minimize Unit Cost
  - Batch or Continuous Unit Operations ?
  - How Flexible is an Automated Facility ?

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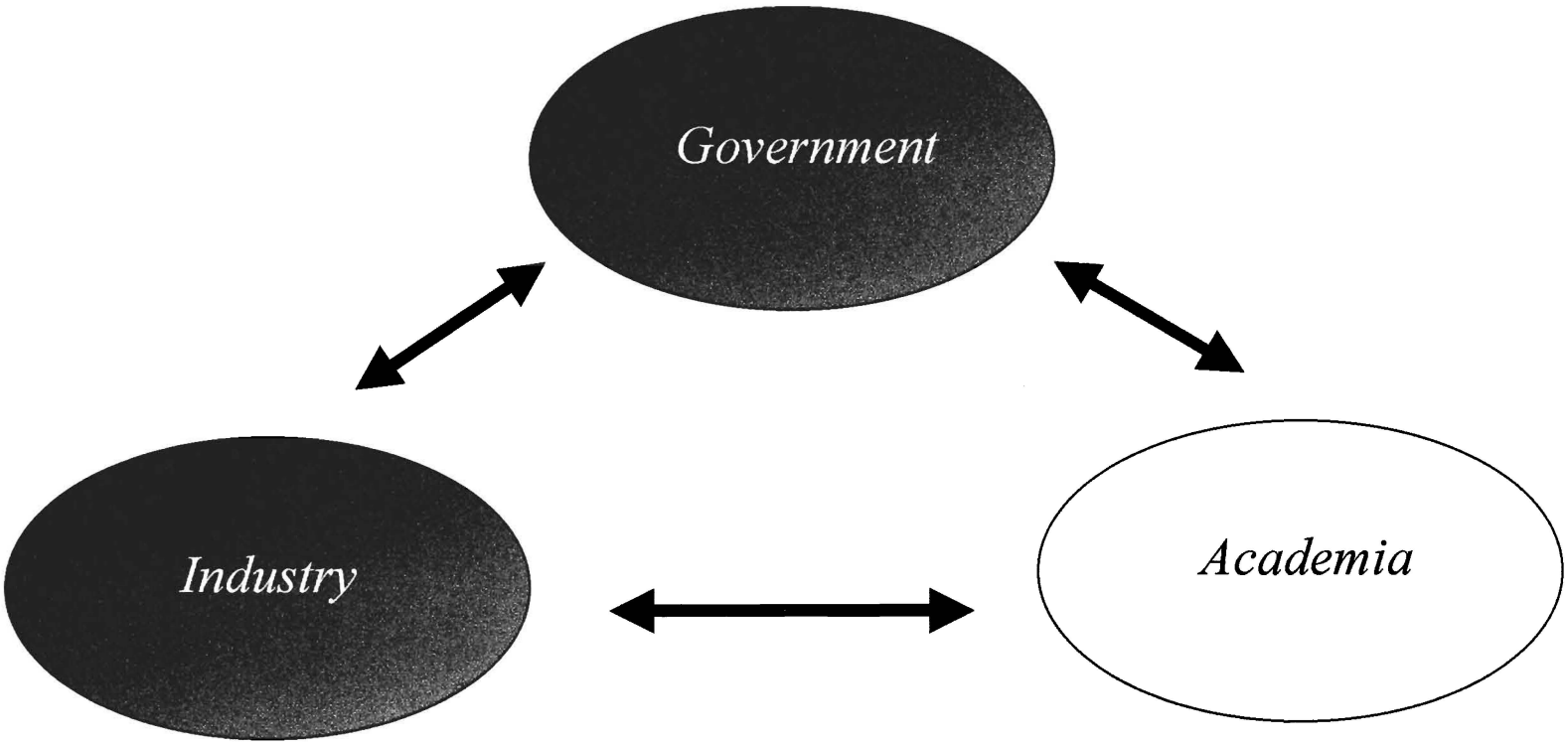
Technology Development

A cooperative relationship between Government, Academia, and Industry



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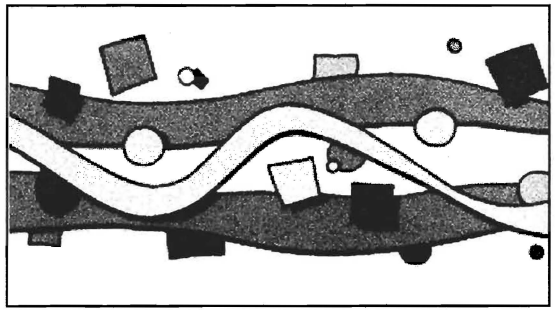
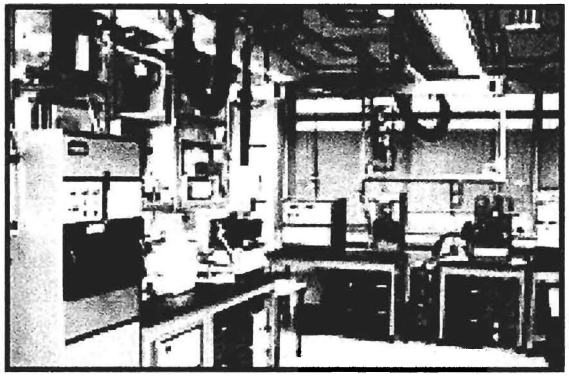
Material Development

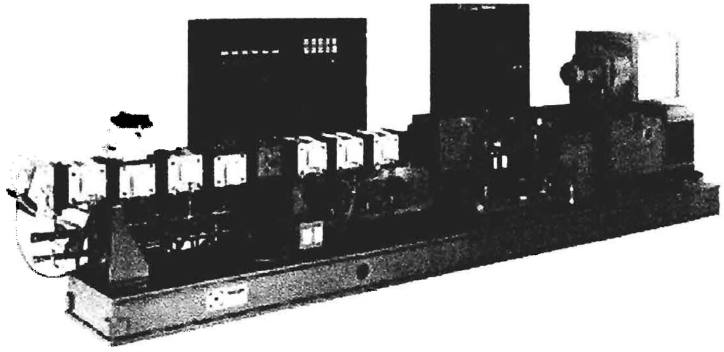
An iterative process of formulation, processing, and characterization



formulation



characterization

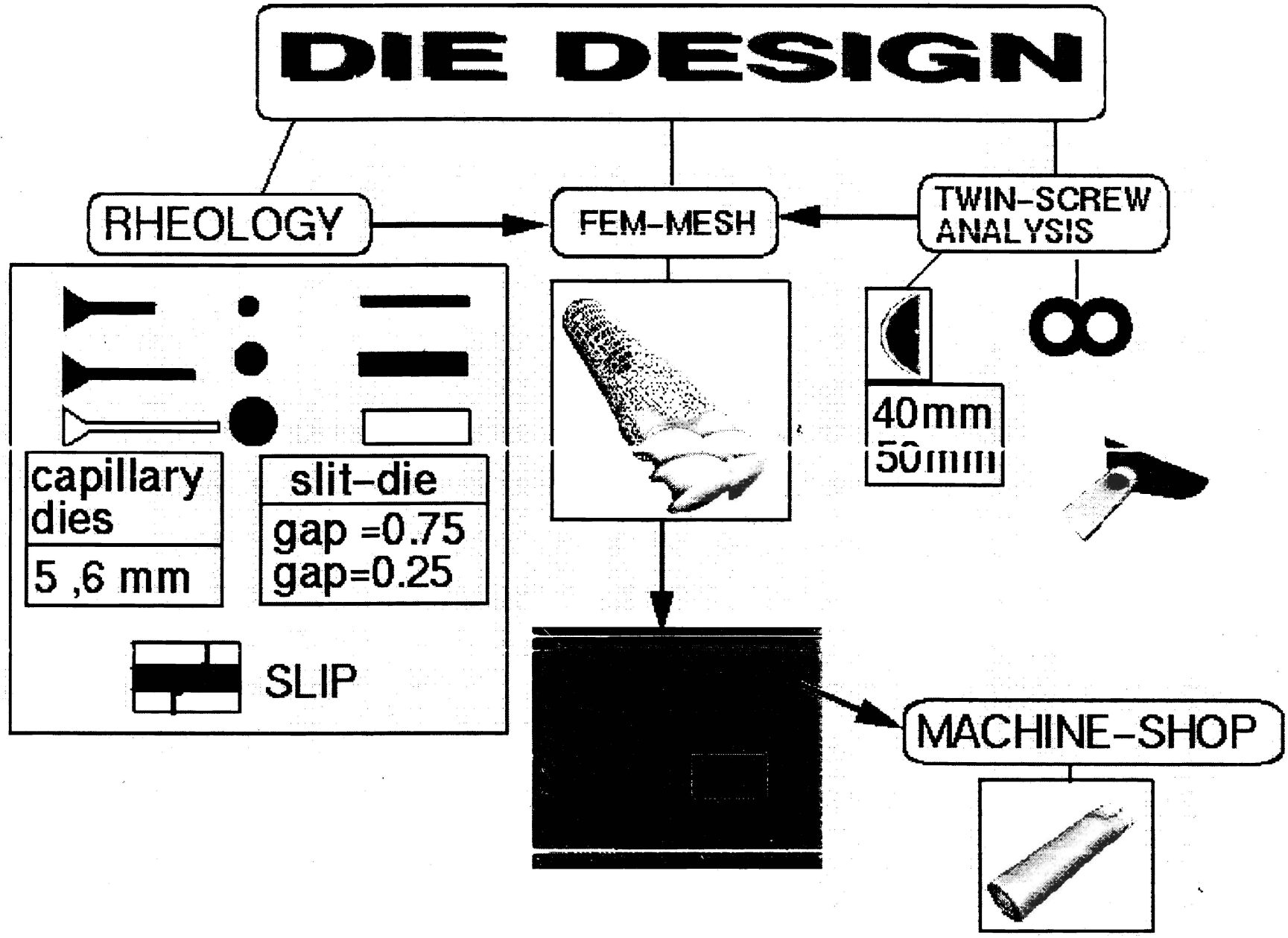


processing

New Techniques for the Simulation of the Die and Twin-Screw Extrusion Processing of Energetic Materials

Dr. Dilhan M. Kalyon
Dr. Sudhir B. Railkar
Dr. Adeniyi Lawal

HfMI, Stevens Institute of Technology
Hoboken, NJ


DIE DESIGN
RHEOLOGY
FEM-MESH
TWIN-SCREW ANALYSIS
capillary dies
5 ,6 mm
slit-die
gap =0.75
gap=0.25
SLIP
40mm
50mm
MACHINE-SHOP

A CO-ROTATING TWIN-SCREW EXTRUDER

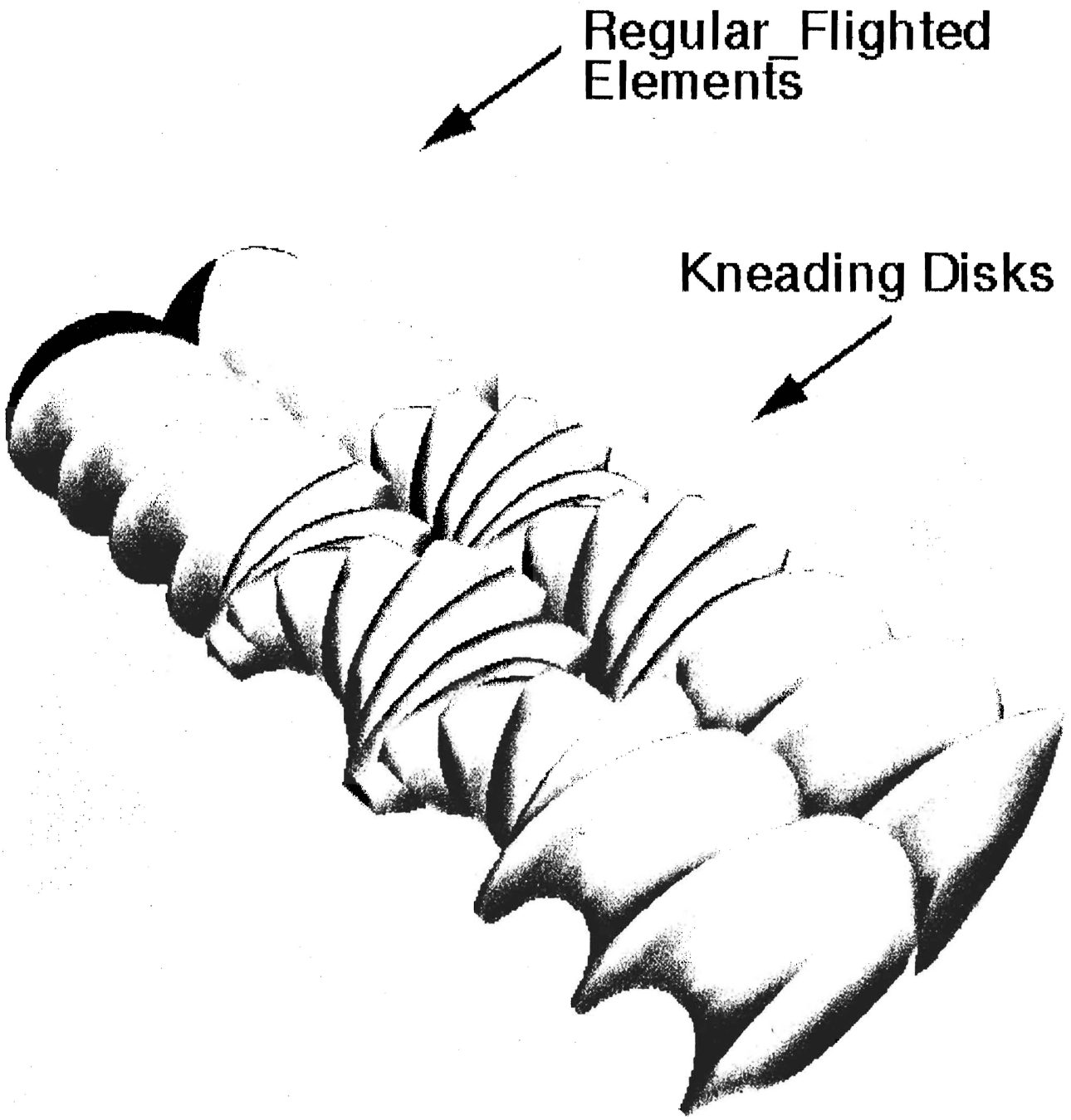
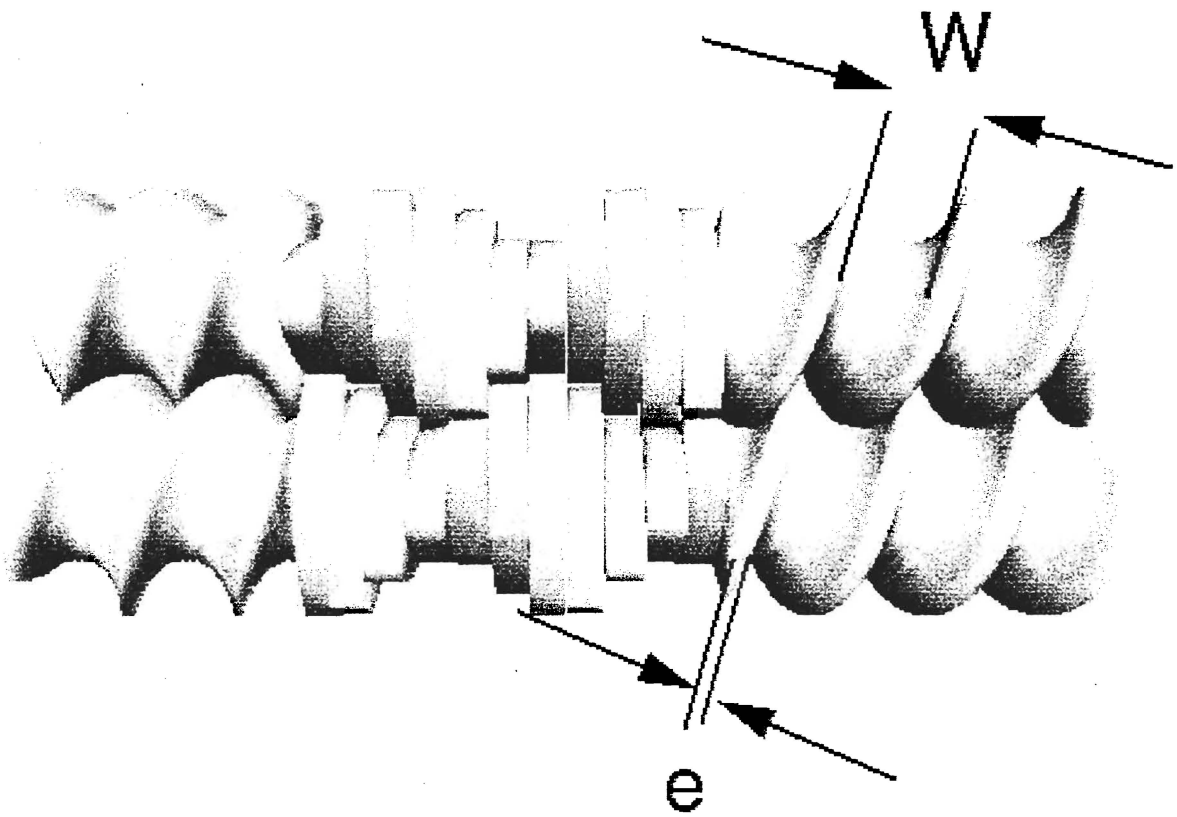


FIGURE 1.1

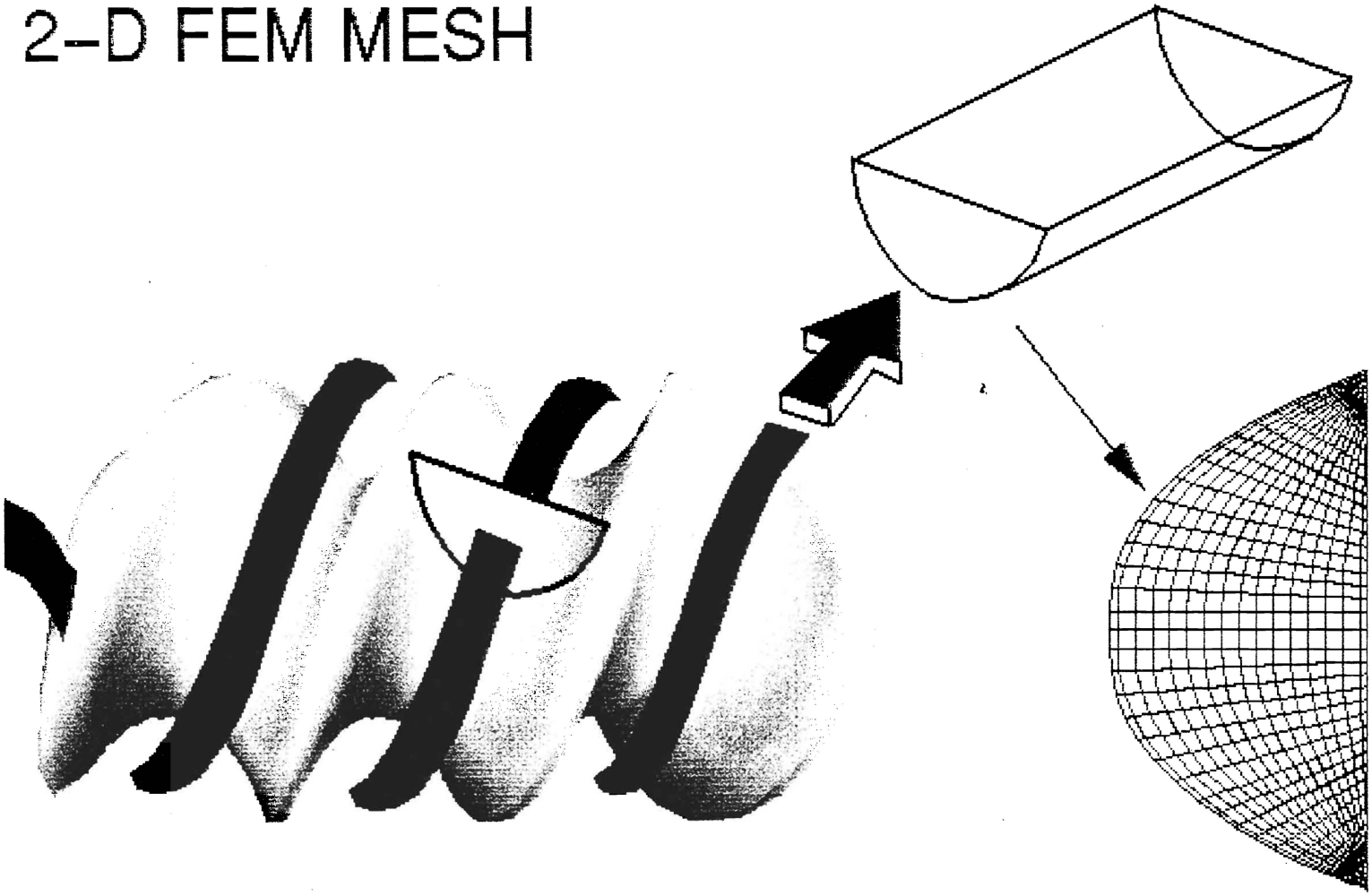
HIMI, STEVENS TECH

UNWRAPPING OF REGULAR_FLIGHTED ELEMENTS

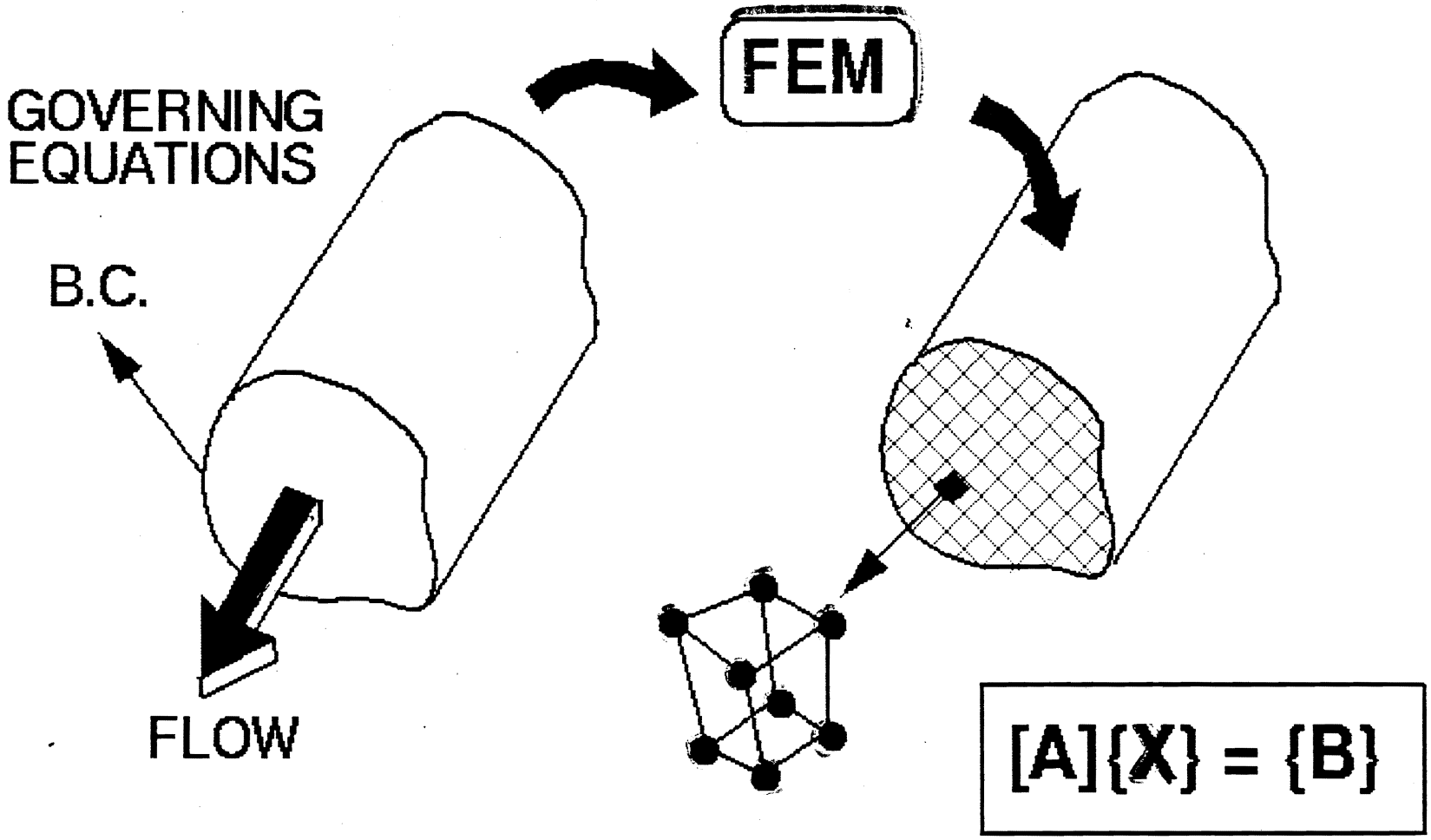


$W >> e$

2-D FEM MESH



COMPUTATIONAL MODELING



MASS-MOMENTUM EQUATIONS

$$\frac{\partial u_x}{\partial x} + \frac{\partial u_y}{\partial y} + \frac{\partial u_z}{\partial z} = 0$$

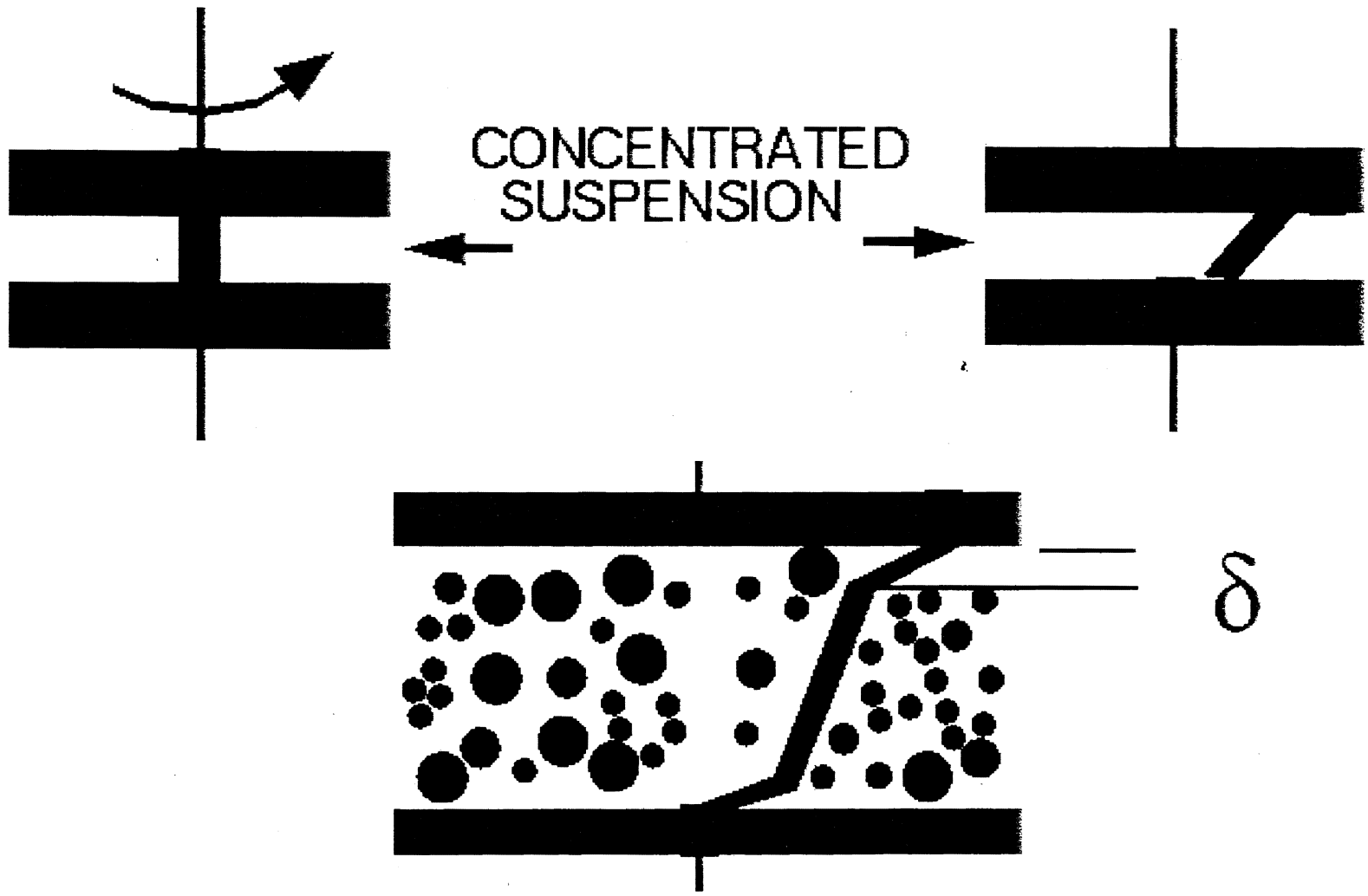
$$\frac{\partial \overline{p}}{\partial x} = \frac{\partial}{\partial x}\left(2\eta \frac{\partial u_x}{\partial x}\right) + \frac{\partial}{\partial y}\left\{\eta\left(\frac{\partial u_x}{\partial y} + \frac{\partial u_y}{\partial x}\right)\right\}$$

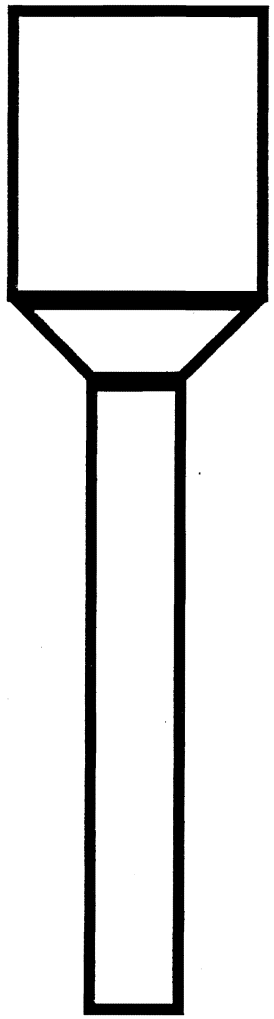
$$\frac{\partial \overline{p}}{\partial y} = \frac{\partial}{\partial y}\left(2\eta \frac{\partial u_y}{\partial y}\right) + \frac{\partial}{\partial x}\left\{\eta\left(\frac{\partial u_x}{\partial y} + \frac{\partial u_y}{\partial x}\right)\right\}$$

$$\frac{\partial p_m}{\partial z} = \frac{\partial}{\partial x}\left(\eta \frac{\partial u_z}{\partial x}\right) + \frac{\partial}{\partial y}\left(\eta \frac{\partial u_z}{\partial y}\right)$$

$$\frac{dp_m}{dz} = \frac{dp_m}{dz}(z) \qquad \overline{p} = \overline{p}(x,y)$$

WALL SLIP MARKER TECHNIQUE



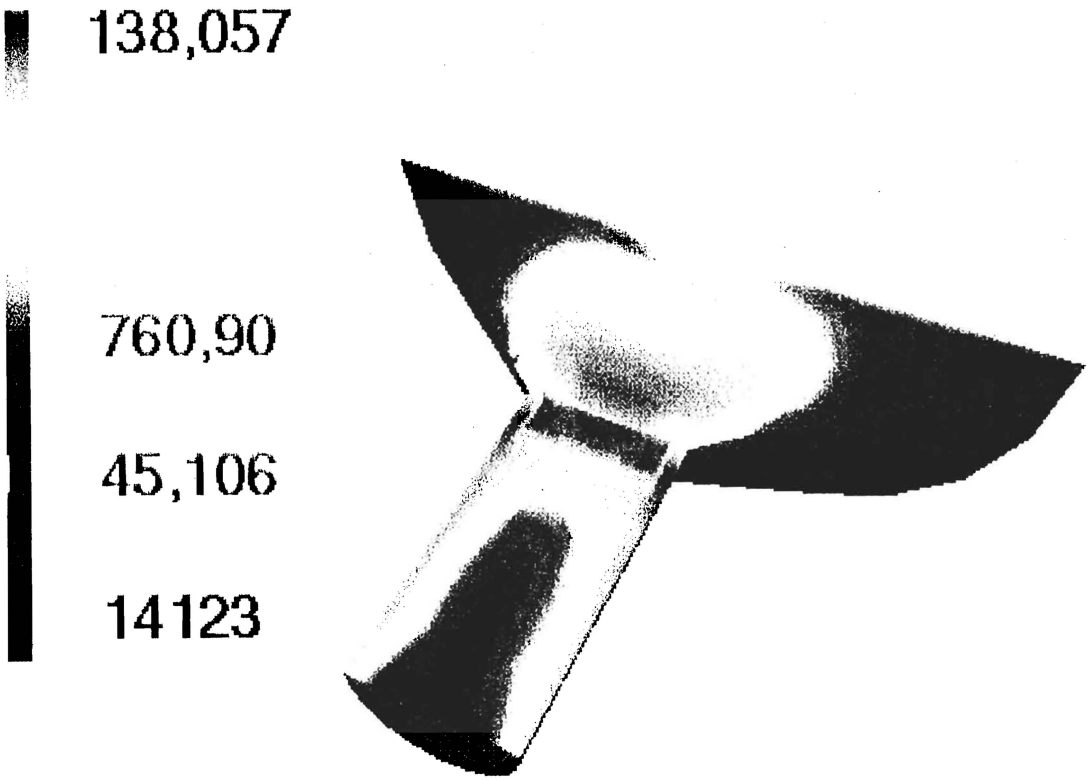


MODELING CAPILLARY FLOW

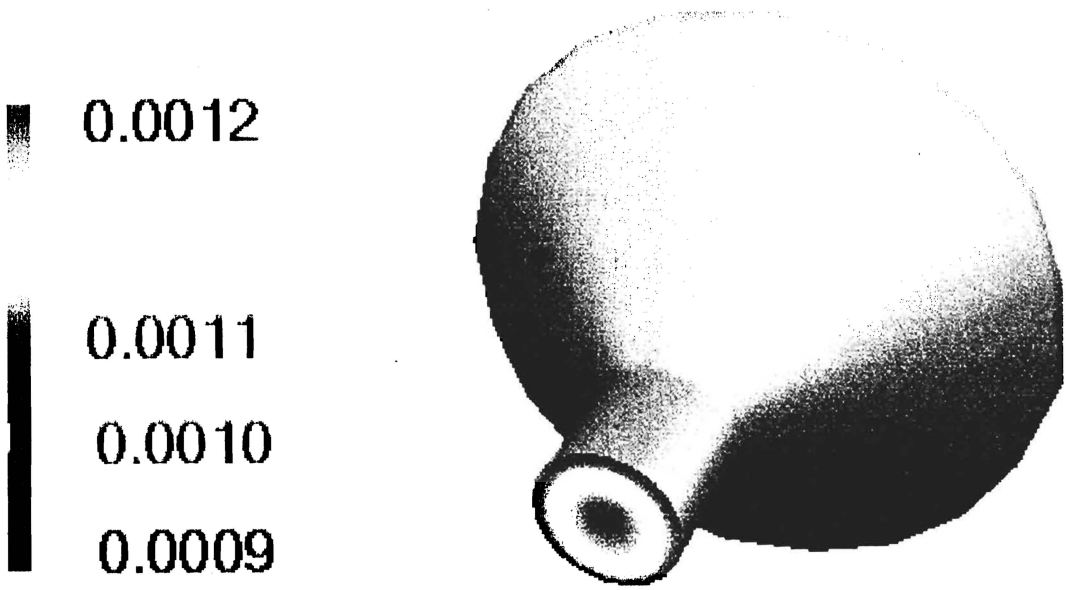
Shear Viscosity, Pa.s
Shear Rate, s^{-1}
0.001 0.01 0.1 1 10 100 1000 10000
100
1000
10000
T = 40 C
ϕ = 60 % by volume
n = 0.43
m = 6888
τ_o = 20 Pa
Tracer Method
L/D=60
D=2.0 mm
Capillary Data
(Adiabatic)
Parallel Plate Data
Slit Die Data

Shear Viscosity, Pa.s
Shear Rate, s^{-1}
0.001 0.01 0.1 1 10 100 1000 10000
100
1000
10000
T = 40 C
ϕ = 60 % by volume
λ = 7.72
m = 21779.5
n = 0.43
Tracer Method
L/D=60
D=2.0 mm
Capillary Data
(Adiabatic)
Parallel Plate Data
Slit Die Data

2nd Invariant of the stress tensor, Pa
5 mm, L/D=12



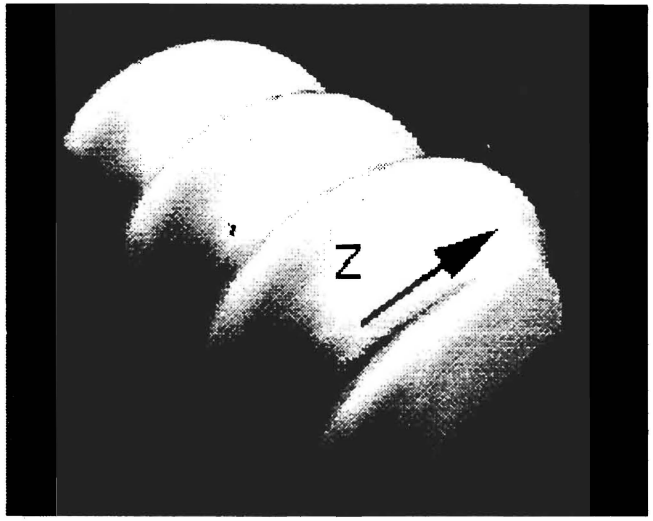
z–velocity, m/s

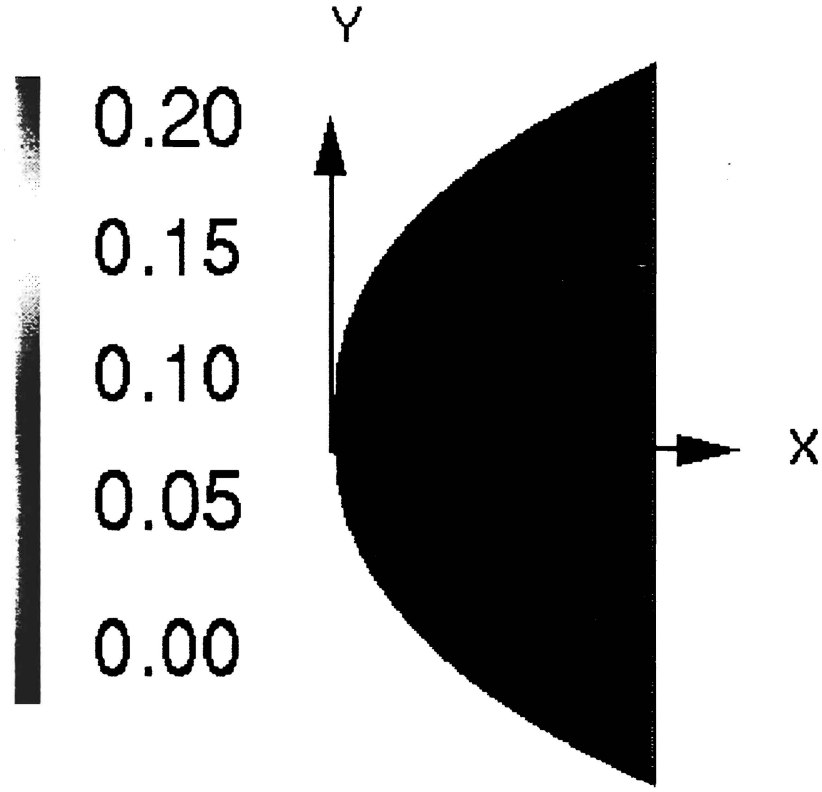


RPM = 100, Q = 33 lbs/hr

LEAD 1

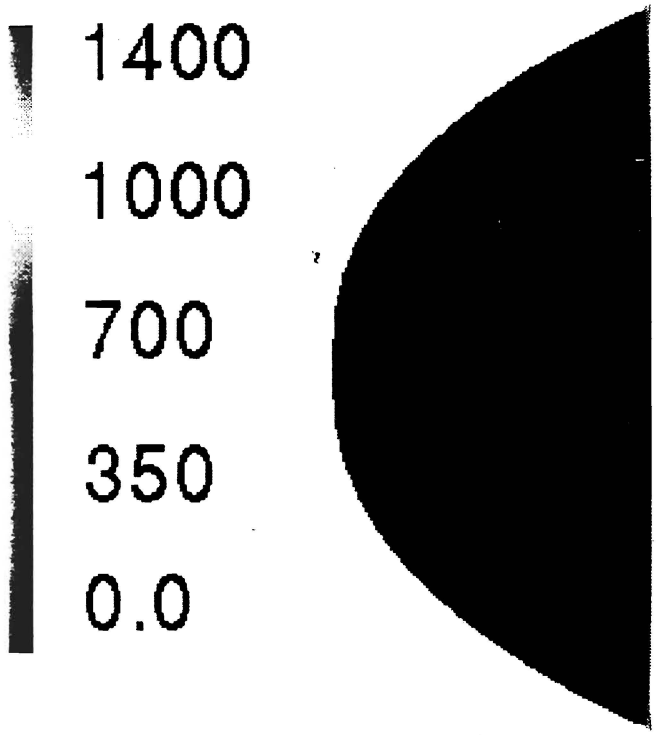
Z –VELOCITY m / s



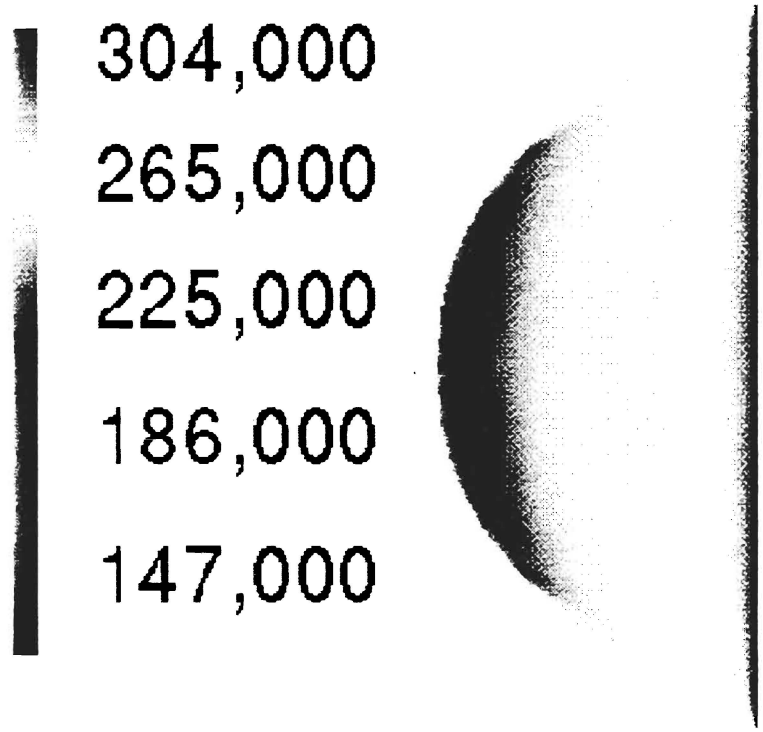


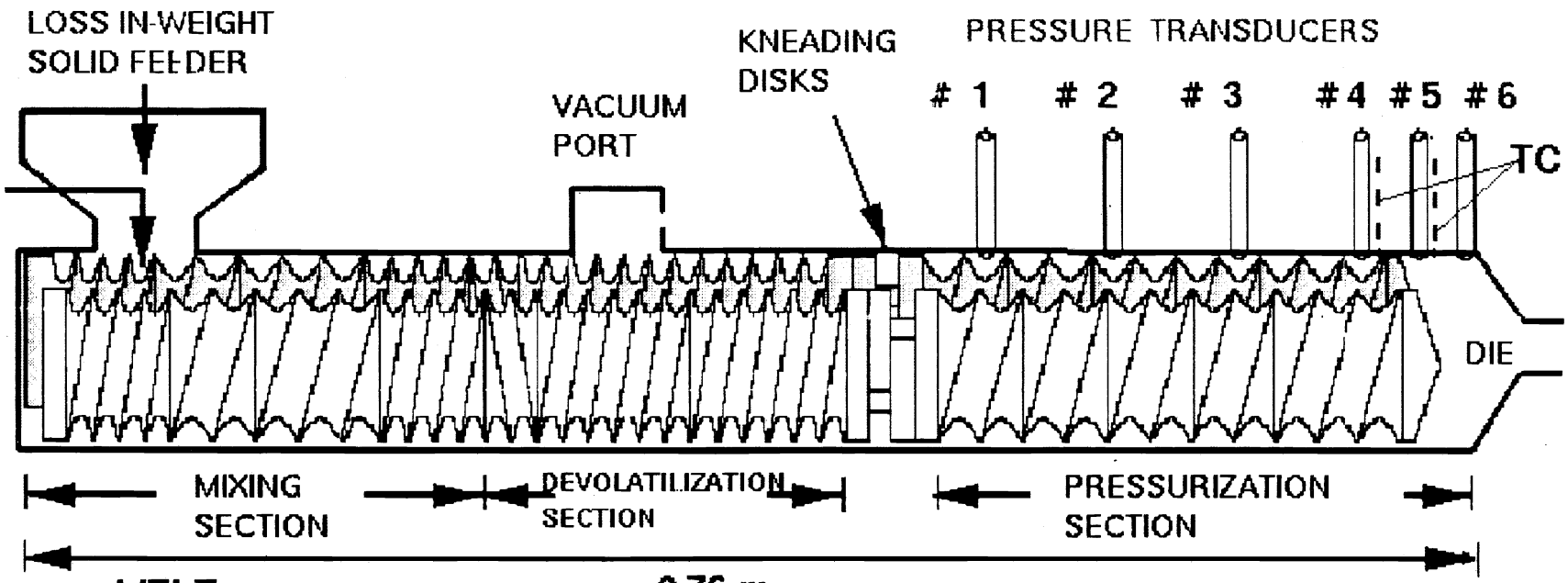
RPM = 100, Q = 33 lbs/hr

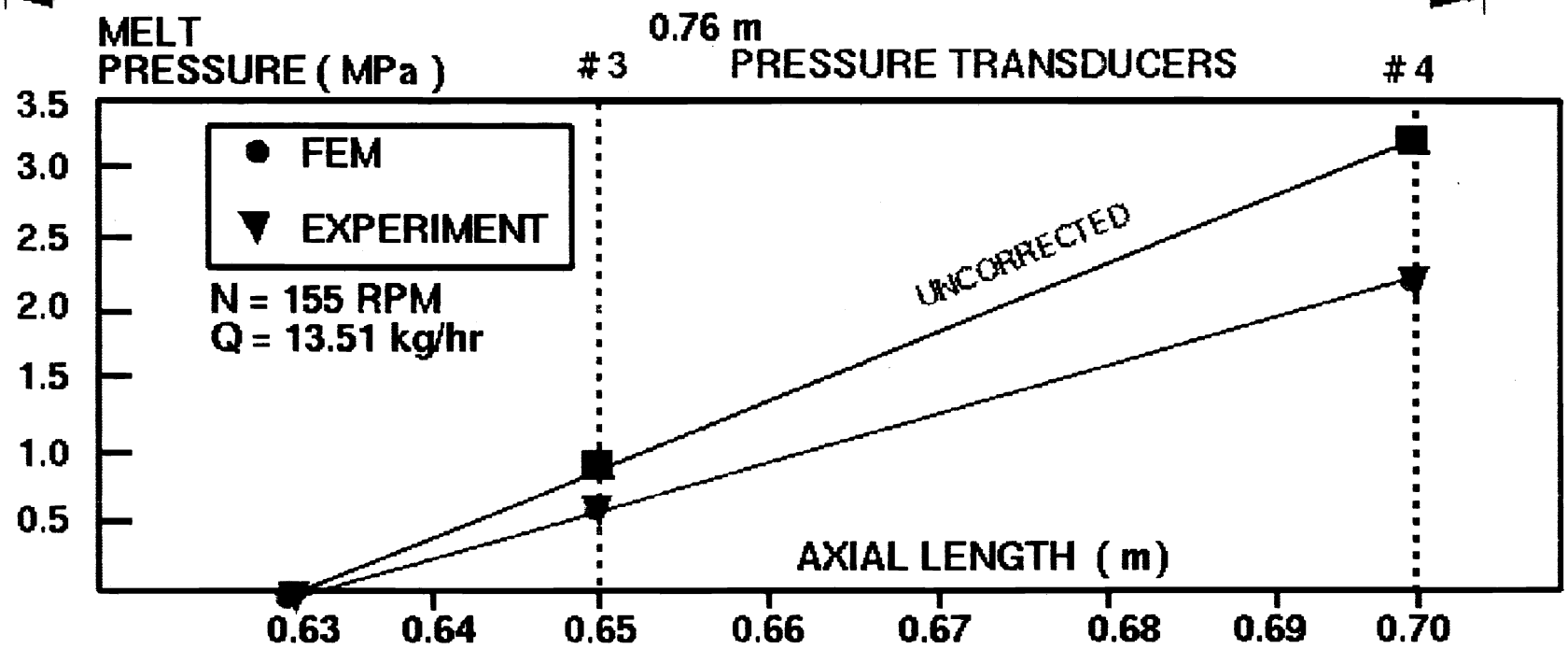
SHEAR RATE $\left|\dot{\bar{\gamma}}\right|$ 1/s



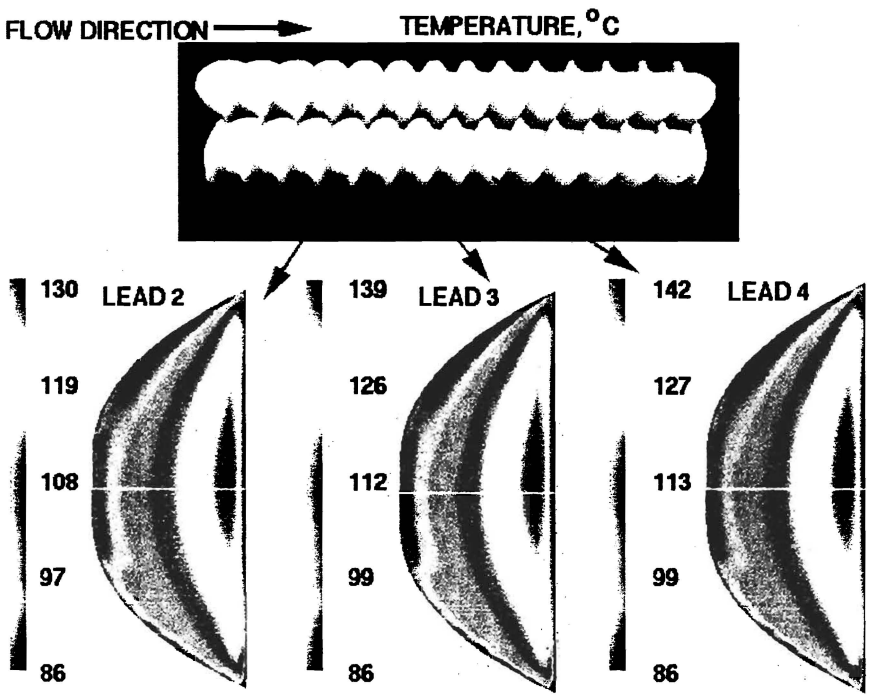
$\left|\bar{\tau}\right|$ Pa



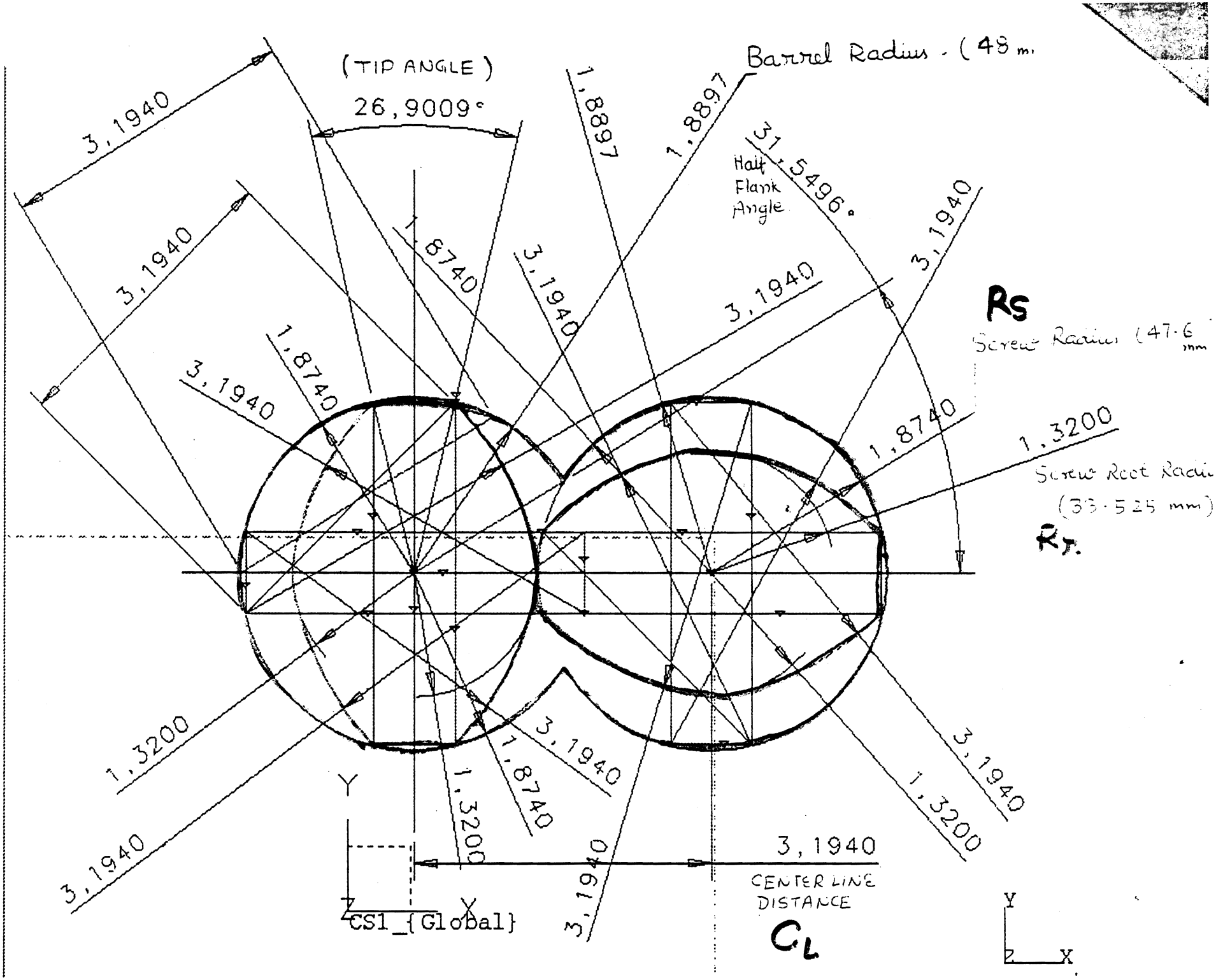

LOSS IN-WEIGHT
SOLID FEEDER
VACUUM
PORT
KNEADING
DISKS
PRESSURE TRANSDUCERS
1
2
3
4
5
6
TC
DIE
MIXING
SECTION
DEVOLATILIZATION
SECTION
PRESSURIZATION
SECTION
0.76 m


MELT
PRESSURE (MPa)
3
PRESSURE TRANSDUCERS
4
3.5
3.0
2.5
2.0
1.5
1.0
0.5
FEM
EXPERIMENT
N = 155 RPM
Q = 13.51 kg/hr
UNCORRECTED
AXIAL LENGTH (m)
0.63
0.64
0.65
0.66
0.67
0.68
0.69
0.70

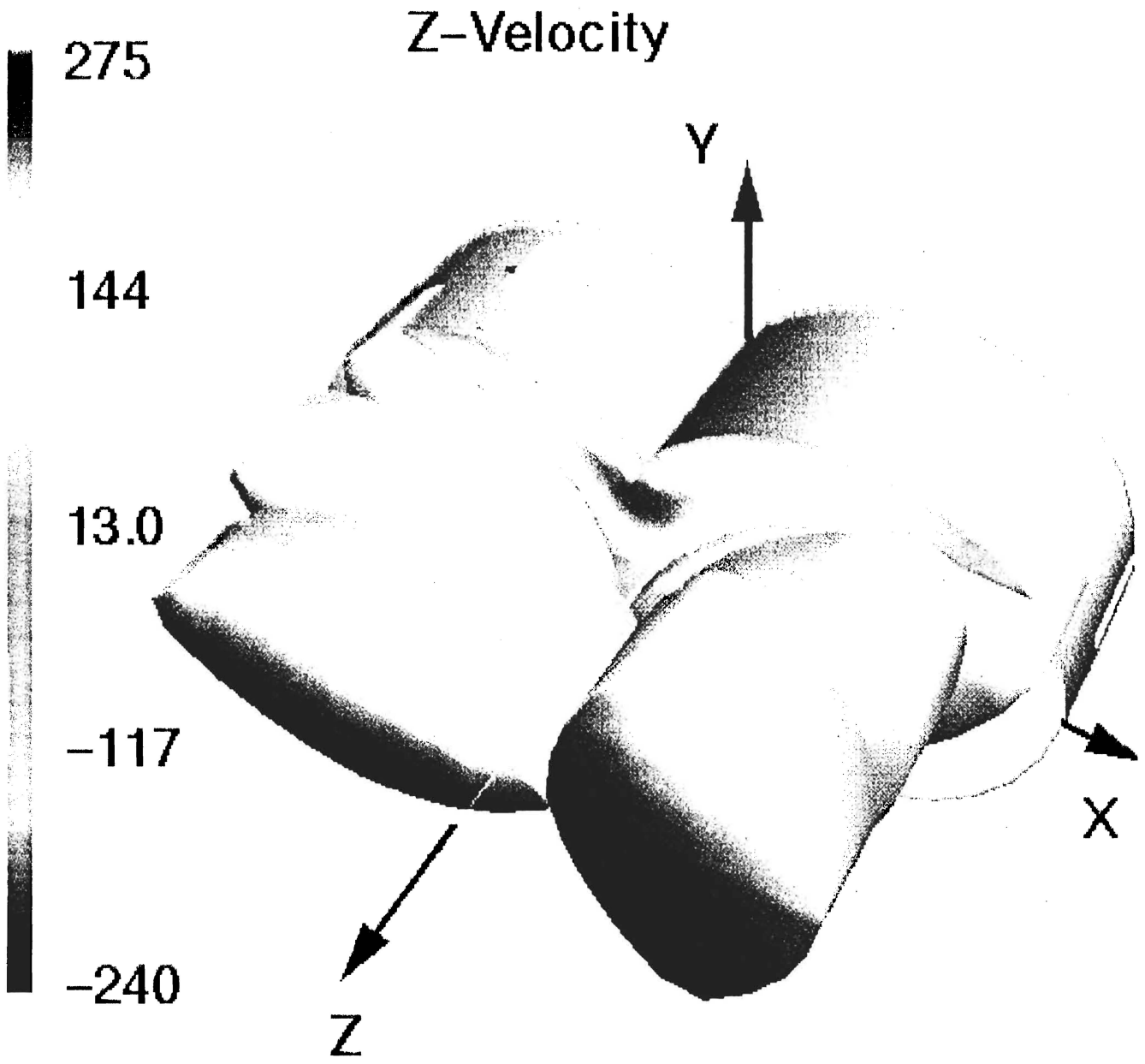
Simulation identifies and eliminates safety concerns without experimentation



- Hot spot formation
- Initiation temperature is reached

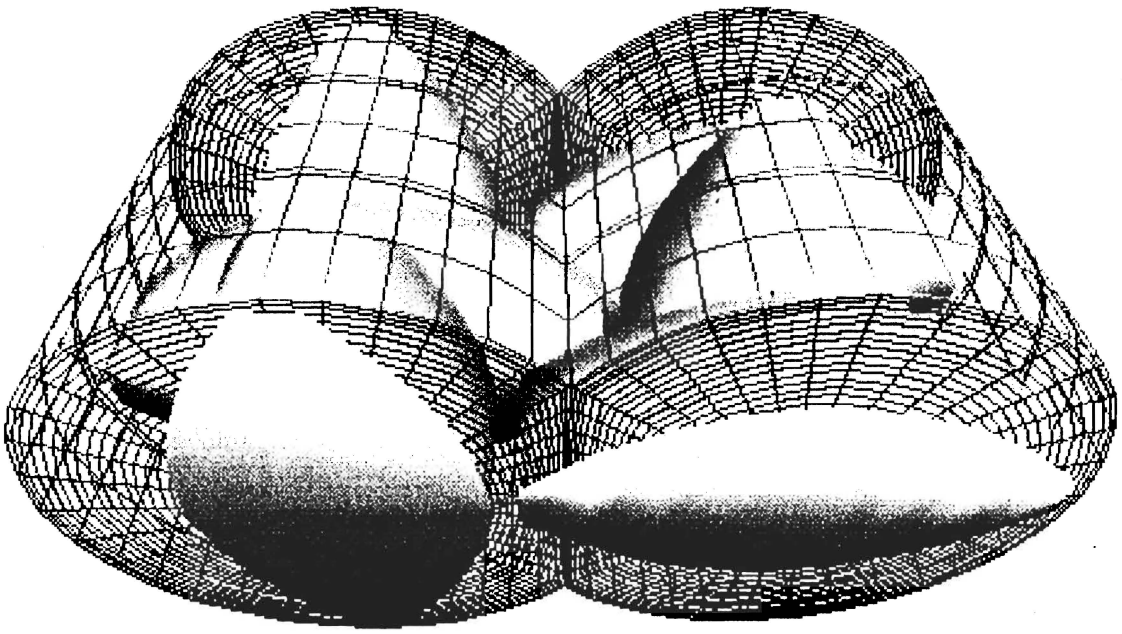

(TIP ANGLE)
26,9009°
Barrel Radius - (48 m
3,1940
3,1940
1,8897
1,8897
Half Flank Angle
31,5496°
1,8740
3,1940
3,1940
3,1940
1,8740
3,1940
1,8740
Rs
Screw Radius (47.6 mm
1,3200
Screw Root Radius
(33.525 mm)
RR
1,3200
3,1940
1,8740
3,1940
1,3200
3,1940
1,3200
3,1940
3,1940
3,1940
CENTER LINE DISTANCE
CL
Y
Z
X
CS1_{Global}
Y
Z
X

KNEADING DISK SECTION OF A CO-ROTATING TWIN-SCREW EXTRUDER

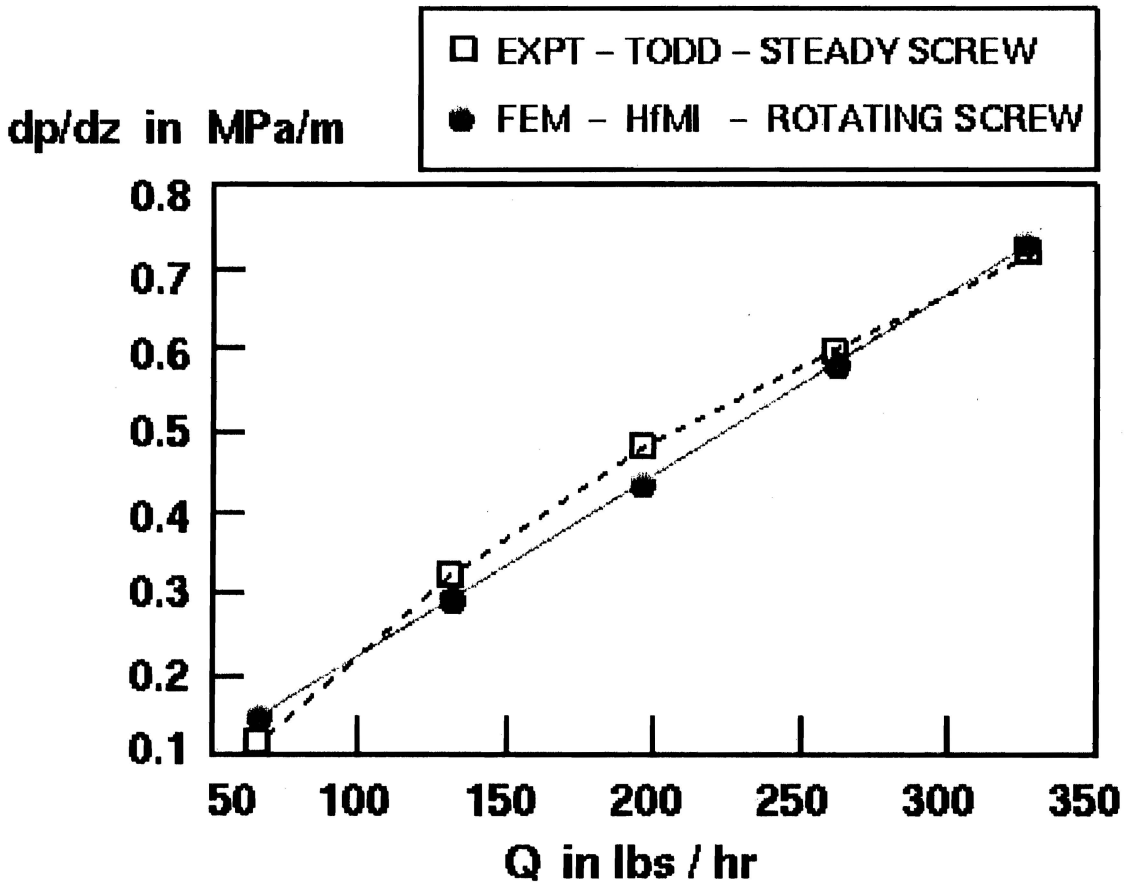


HMI, STEVENS TECH

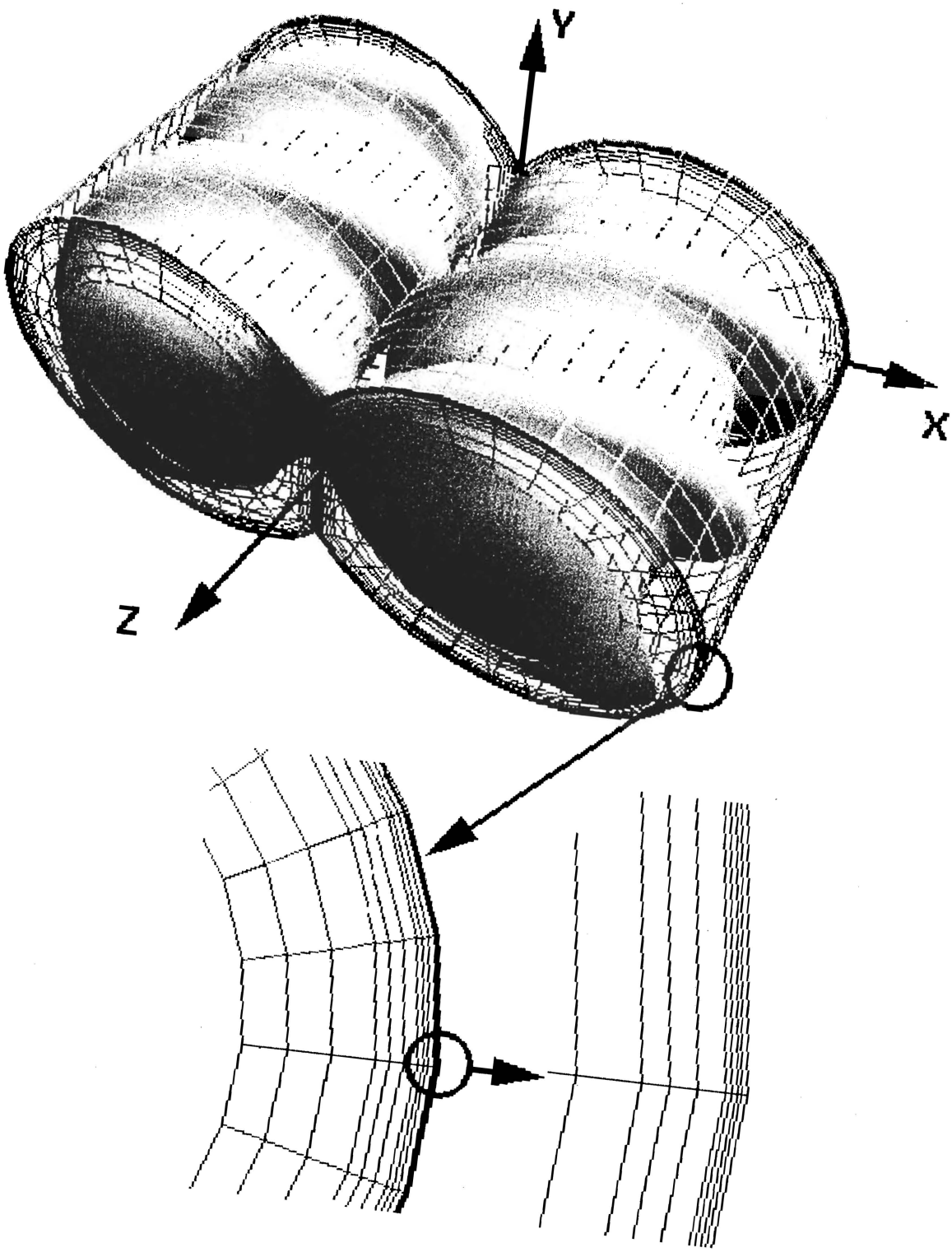
NEWTONIAN FLUID IN 90° STAGGER KNEADING BLOCKS

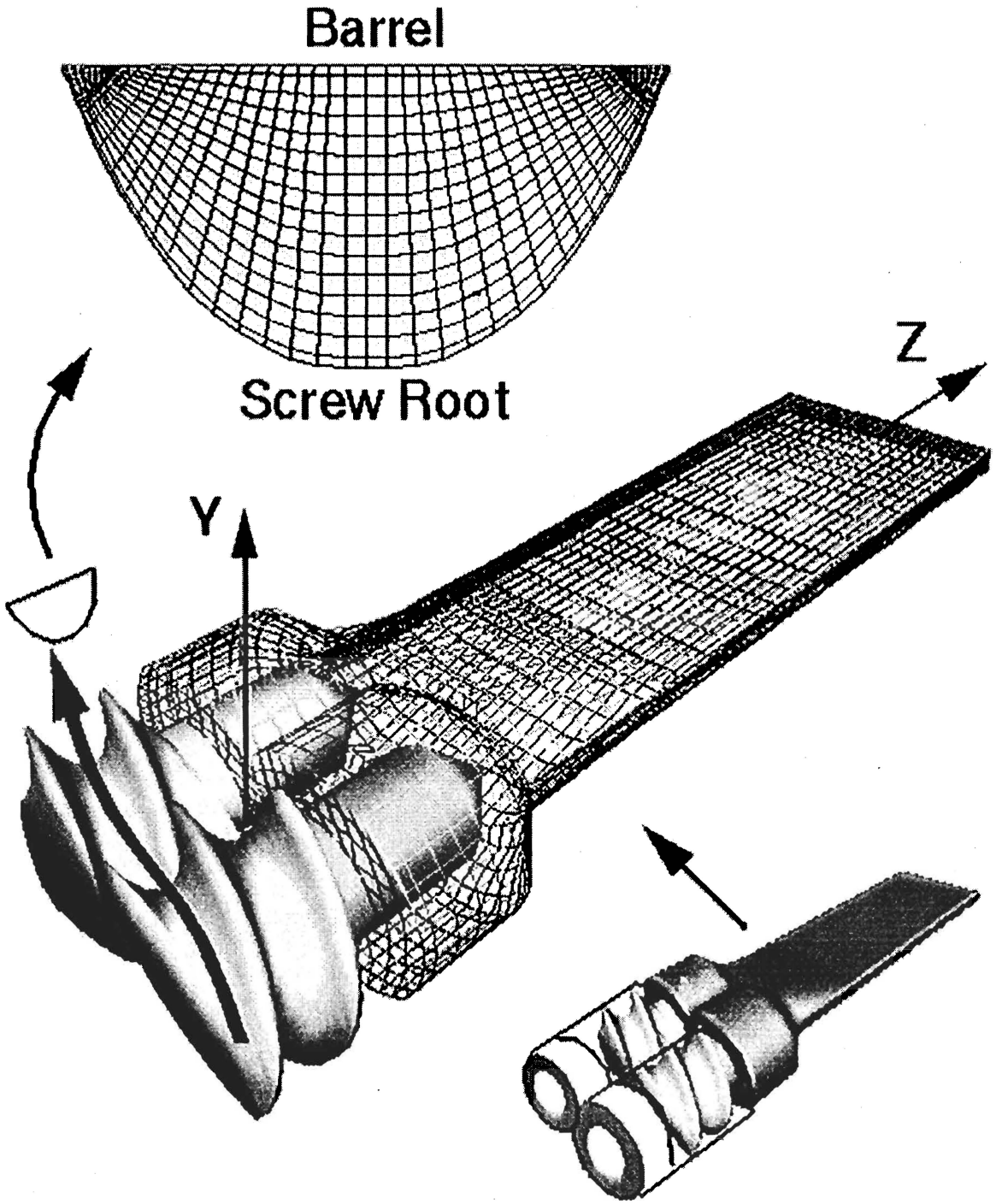


$\Delta P^* = 52$ $\Delta z^* = 0.75$ $Q^* = 0.73$	m = 100 Pa–s n = 1.0 ρ = 1400 kg/m^3

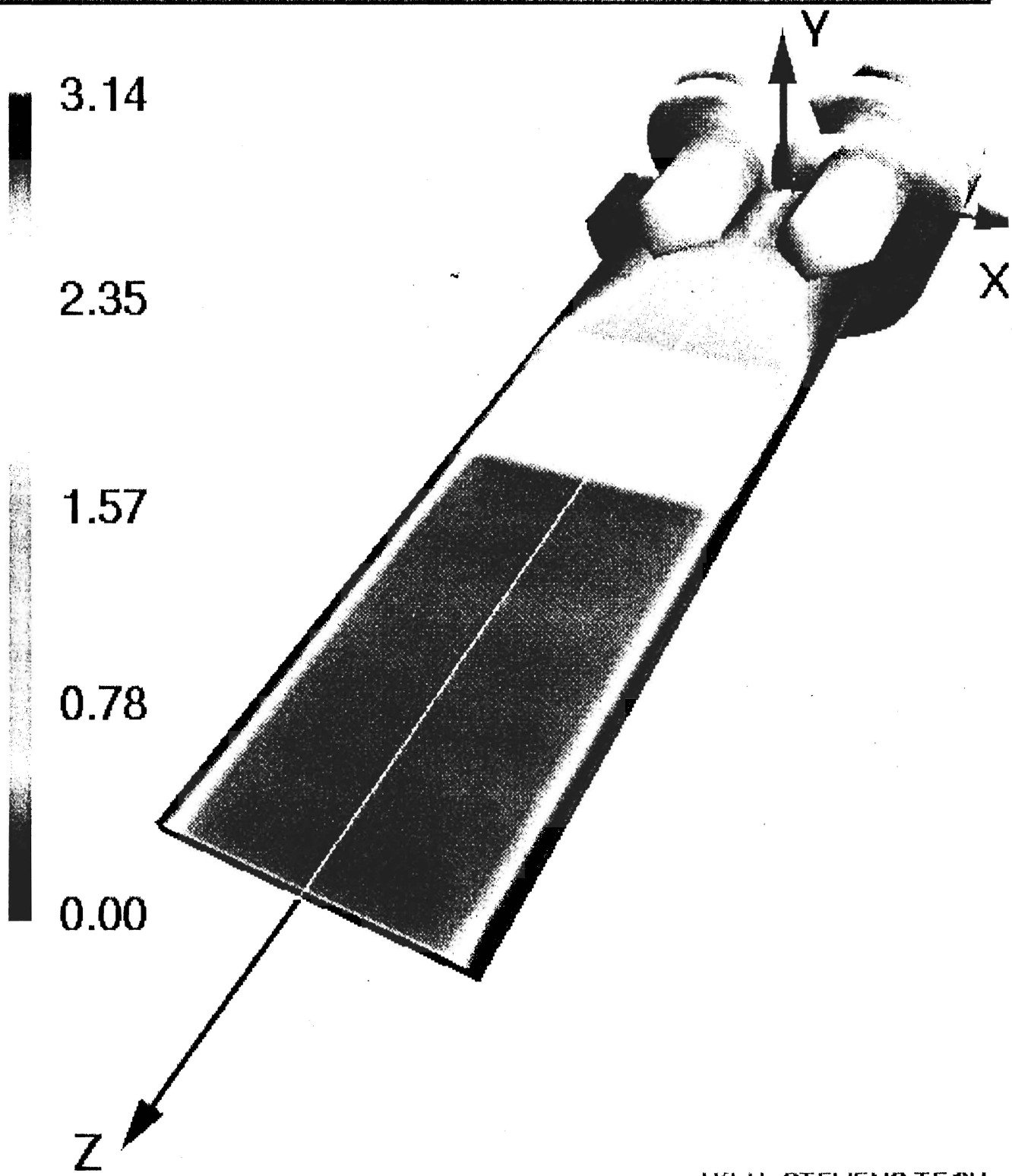


FEM MESH

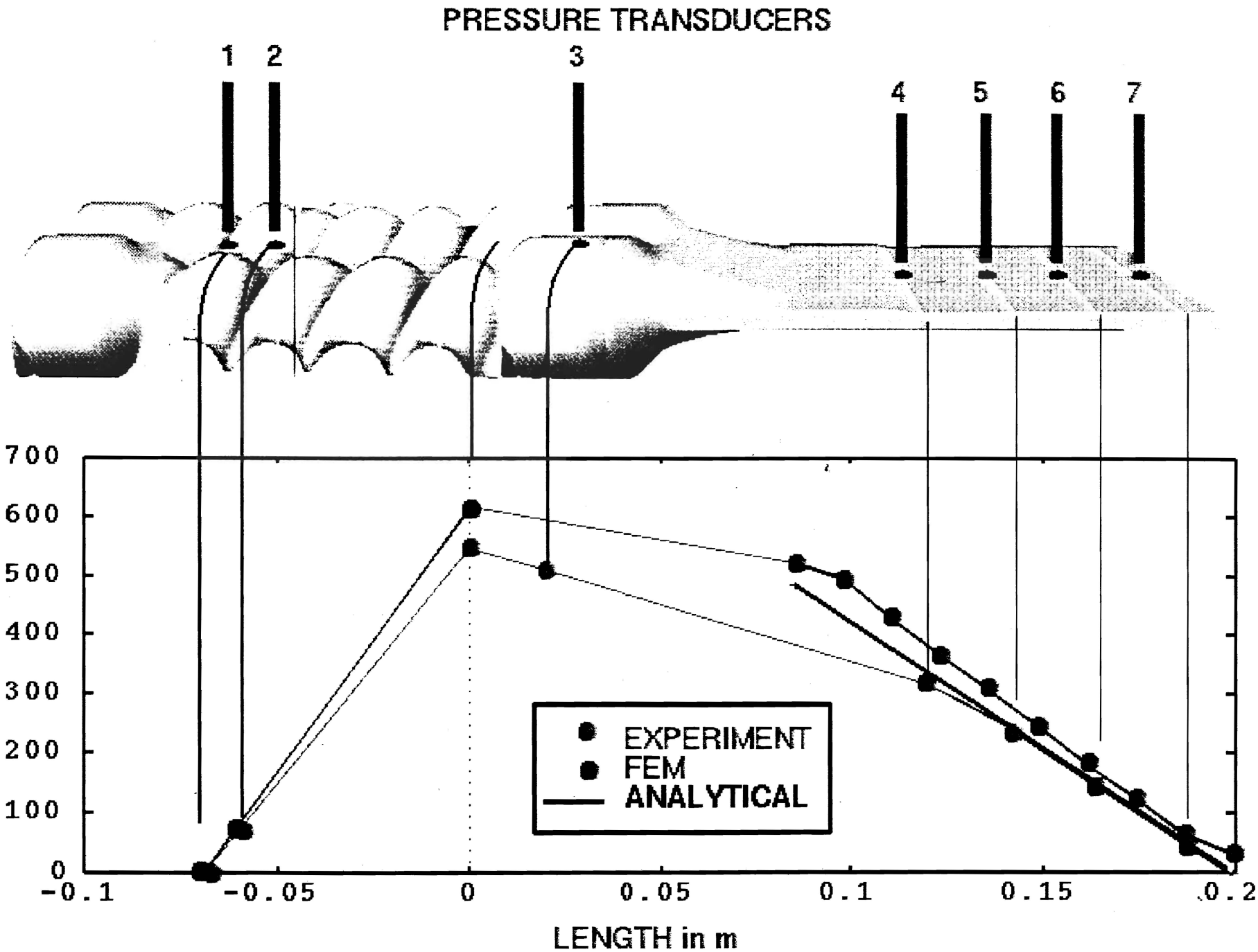



Barrel
Screw Root
Z
Y

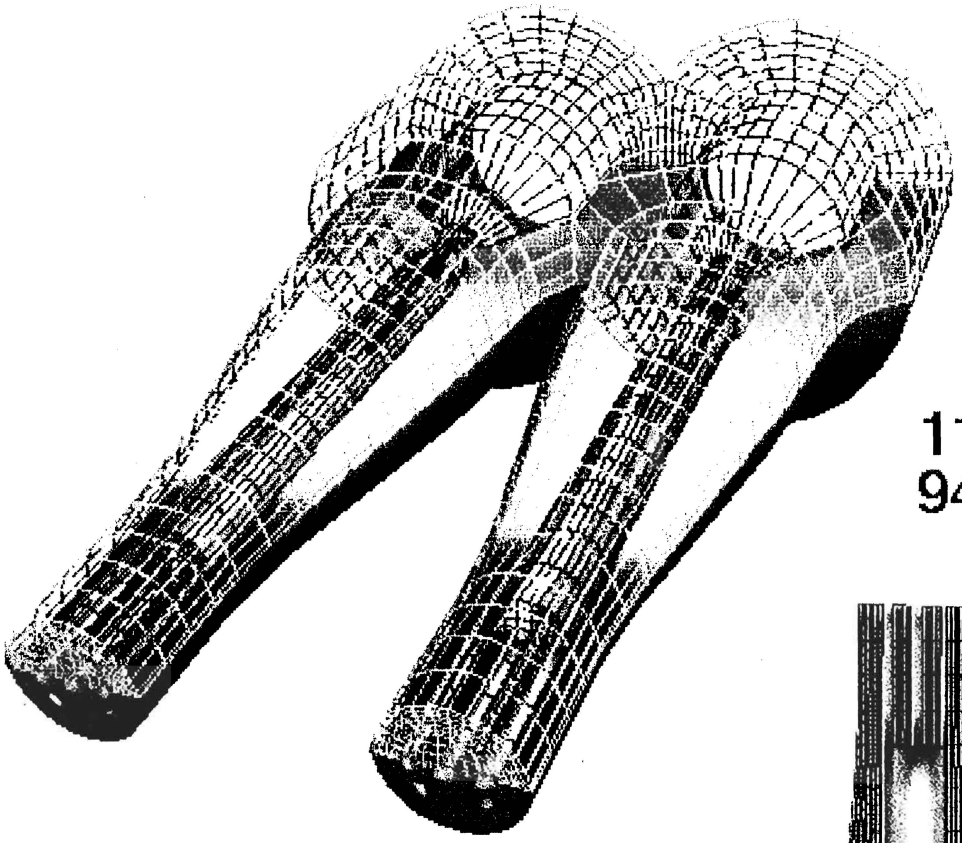
Z–VELOCITY DISTRIBUTION IN AN ADJSTABLE GAP SLIT RHEOMETER



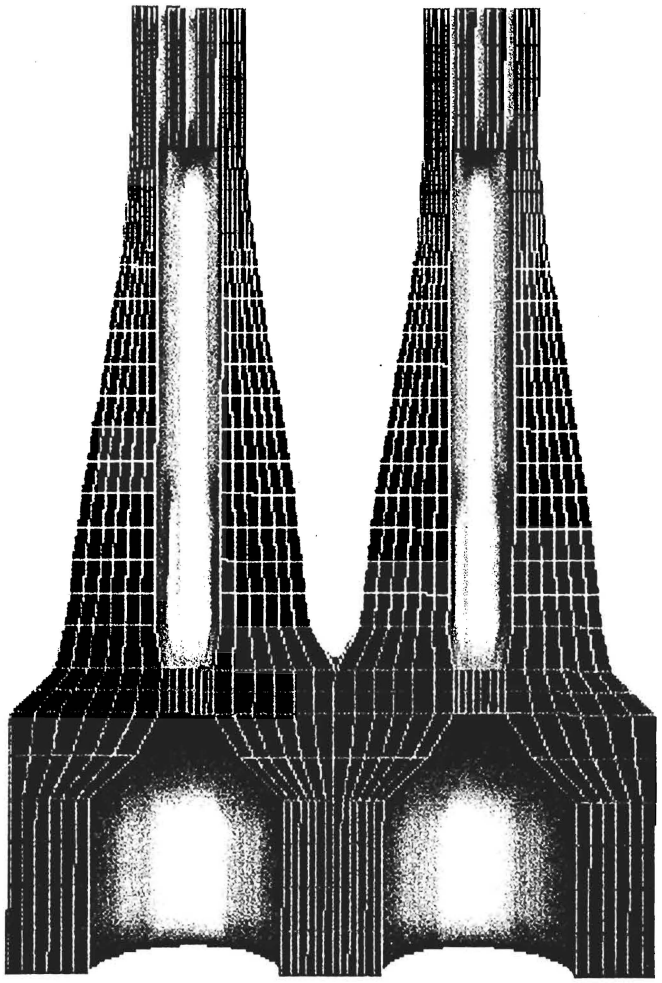
HfM I, STEVENS TECH


PRESSURE TRANSDUCERS
1
2
3
4
5
6
7
700
600
500
400
300
200
100
0
-0.1
-0.05
0
0.05
0.1
0.15
0.2
LENGTH in m
EXPERIMENT
FEM
ANALYTICAL

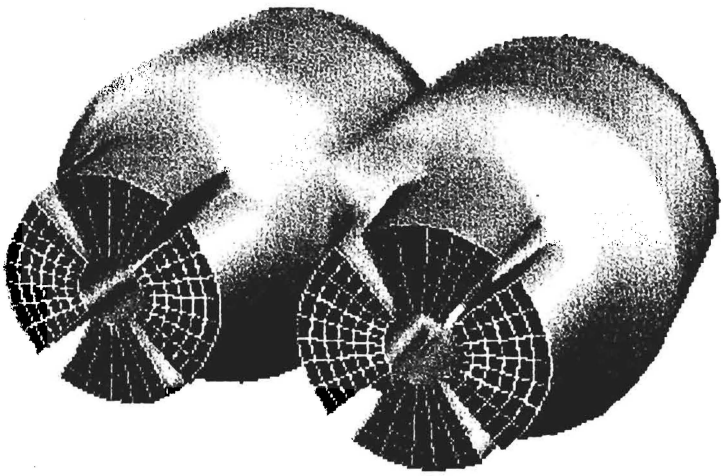
Finite element mesh



11480 Nodes
9412 Elements

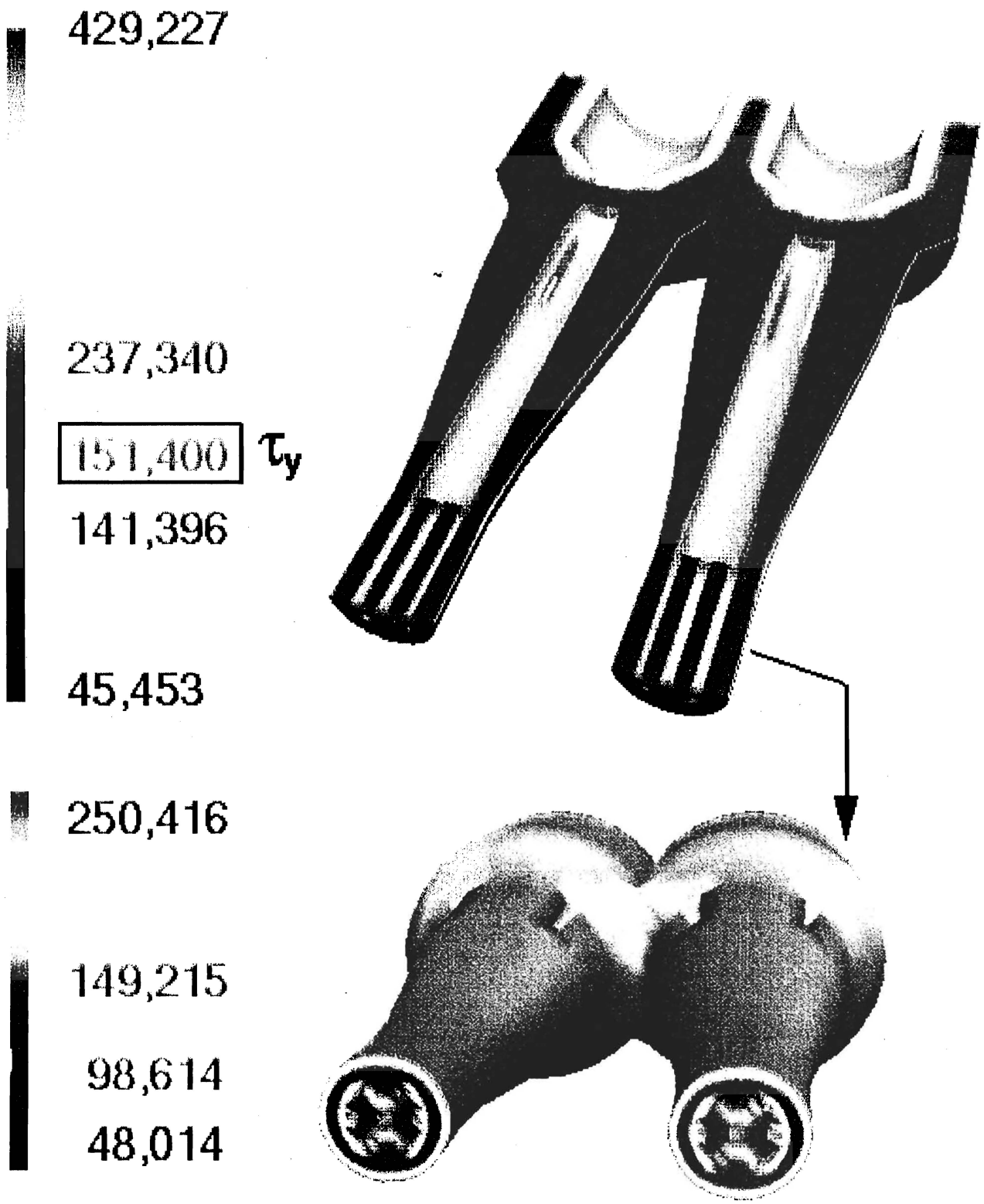


Spider legs



75 Rpm,19 lb/h, T=175 ^{o}F, β = 6.9E−6 mm/Pa−s

2 nd invariant of the stress tensor, Pa



Conclusions

- A general purpose Finite Element computer program is developed for modeling both Twin-screw extruder and Dies in 3-D
- This modeling approach represents the screw and die geometry exactly and Barrel to screw clearances as low as 1% can be modeled
- The program can model:

  Newtonian, Power-Law, Bingham

  and Herschel-Bulkley fluids with temperature dependant viscosity

Conclusions

- Accurate modeling/design of Twin-screw extruders and Dies is possible if special features such as slip and clearances can be incorporated
- Techniques developed at HfMI can be used as DESIGNER TOOLS to avoid costly experimentations

10th JOCG Continuous Mixer & Extruder Users Group Meeting

Ogden Utah
May11-12, 1999

Simulation Based Design and Manufacture of Screws and Dies for the Energetics Industry

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Abstract

The employment of new techniques in design and manufacture of hardware for a first time process results in significant time savings, accuracy of final design and optimum quality of product. These benefits are a result of mathematical modeling of the process, which is then translated into a Computer Aided Drafting (CAD) file. This file is electronically transferred between the simulation staff and the manufacturing engineering staff for concurrent final tuning of the production equipment design. The files are then converted into Computer Numerical Control (CNC) machining programs from the completed CAD files to produce the actual screw and die components. All of this is accomplished using scientific tools and principles, without actual costly and time consuming trial and error runs.

Approximately six months ago, I attended the annual conference of the National Tool Manufacturers Association (NTMA). Some of the background information used to develop the logic behind this technical presentation was extracted from questions and responses from the members of that association regarding the future of production industries in this country. You may be wondering what this has to do with the energetic industry and in particular with the continuous mixer users group. Interestingly, all production facilities in today's competitive market seem to be influenced by the same trends. No one industry is isolated or protected from direct competition and from the pressures of competitive pricing, delivery and technological advancement.

Not even the government itself is protected from industrial and global competition in these categories. In fact, the government purchases various energetic materials from abroad, so the competition is here already.

Working harder will not necessarily salvage the business from these competitive pressures; in many cases if workers were capable of putting in twenty-four hour days, they could not compete with the newest advances in robotics, and automated production methods. Working smarter, being innovative, maximizing efficiency and making every movement effective is not only the way to advance and grow, but in many situations in today's world it is necessary just to survive.

Because this condition is global throughout industry, the basic concepts and positions expounded upon by the NTMA members translate directly to this industry as well. The specific industry requirements may be unique, but the logic and principles are universal throughout industry including this one.

From the meeting, the NTMA reports, the majority of their members responded to the following survey question. "What changes in Technology do you see affecting your business over the next three years." The participants cited several issues repeatedly when responding to this question. Chief among these issues was the change occurring in Computer Numerical Control (CNC) technology. Participants noted their interest in the future development of personal computer (PC) based and open architecture controls.

The development of CNC technology along these paths has and will lower costs and reduce service time according to participants. These are essential improvements if shop owners are to continue holding cost and improving on delivery times both issues cited in the survey concerning future customer relationships.

New manufacturing processes were cited frequently as having a potentially significant impact on future business. New rapid prototyping technologies, higher speeds, and new materials topped the list of new technologies business owners believed would have an impact on their business. Amongst these responses it was clear that the participants expect a dramatic shift in the types of materials being produced within the next ten years.

Following new manufacturing processes, were issues related to precision and quality. Chief among the specific points relating to quality was increased customer demand for pre-qualified parts or zero defects. This related to the increased use of manufacturing automation as many participants noted that the use of automation would increase to address the zero defects or in-process, non-destructive testing of production issues.

Another question asked participants, "What changes do you foresee in your customer base and requirements of your customers". While there were numerous responses, two stood out as overwhelming issues, higher quality and shorter lead times. Participants noted that while customers are requiring a standardization of their processes they are also pointing to zero defects on parts with much tighter tolerances. Certification and quality issues are expected to be a major challenge to the manufacturer/customer relationship in the future.

Almost as important and related to the subject of quality and tolerances are customers' expectations that lead times will continue to shrink in the future. Participants noted that customers' expectations only increase when on-time deliveries improve. Some customers are even projecting lead time improvements in the requests for proposals they send to the contractor or manufacturer. It is clearly expected that this trend will continue and that customers in turn will expect better delivery performance from material suppliers and higher up-time percentages from their capitol equipment suppliers.

Three other issues were cited; impact of outsourcing, globalization and consolidation. Participants noted an increase in outsourcing of complete projects by manufacturers and their first tier suppliers. While it means that there is more business trickling down to their businesses, customer expectations and business risks have increased. The outsourcing trends have led first and second tier suppliers to look for full service shops with a broad range of capabilities, including engineering. Participants that noted the outsourcing trend as a significant factor also noted the efforts of first and second tier suppliers to reduce the number of suppliers. The combination of the two developments has made relationships both more important and at the same time riskier. As supplier lists consolidate and more work travels into these smaller groups, opportunities for business become all or nothing situations. In many cases, your line of distribution and your customers' relationship in the supplier chain become more important than your capabilities.

Globalization of markets will accelerate the process. Both the contract shop's customers and their customers' customer are driven to compete on a global basis. As the major manufacturers strive to simplify their production process, they will repeatedly go back to the team of suppliers with which they have had the greatest success. In effect, duplicating their successes in a cookie cutter fashion around the world. This is increasing the international competition which U.S.-based contract shops will face daily in both international and their geographic markets.

It is also clear that pricing pressure is increasing, along with smaller lot sizes and in new materials to work. It is also clear that smaller lot sizes and pricing pressure issues are related.

This brings us to the issue of "What changes do you see occurring in your supplier relationships with machine tool builders." Four basic points develop the response to this question. Suppliers clearly plan to pass through the expectations their customers have for improved delivery, reliability and price onto their machine tool suppliers. Add to this list that contract shop owners expect that their opportunities to select a broad range of suppliers will continue to expand as product quality of builders in developing countries improve.

However, participants do not expect to have a larger list of bidders and suppliers. Like their customers, they expect to reduce the number of suppliers with which they operate. This change will provide the opportunity to partner with their suppliers. Respondents noted that in partnering with suppliers, particularly in machine tool sales, they expect their partners to keep them in the know on new technologies, applications and process improvements. Respondents expect that through partnering, their suppliers will share with them productivity improvements and the savings that they represent.

As an example, in the development of continuous processing techniques for energetic materials, extrusion of mixed materials through a die to develop geometric characteristics is a requirement. The development of an optimum die is important. In order to achieve this goal, data on the physical geometry of the die (in conjunction with the need to mate it to other processing equipment), and the physical characteristics of the process material (rheological data) at the expected operating conditions must be obtained and modeled.

The following constitutes a general listing of the minimum information that must be available before one can begin to develop an optimum die design.

Physical data:

- Drawings of the end of the extruder that the die must mate to.
- Type of die (hot cut, Underwater Pelletizer, Stranding, Forming)
- Cross sectional geometry of extrudate.
- Requirements for breaker plate/screen pack, including level of screen filtration.
- Design parameters, i.e. design pressure, method of heating or cooling, ratings of heaters, desired materials of construction, requirements for instrumentation, thermocouples/transducers (for control / for process evaluation). Metric/Imperial dimensioning and hardware. High power voltage/frequency. Control power voltage/frequency. NEMA type or other standard enclosures. Special provisions, (shielded wire) etc.
- With or without rupture disc or shear device.
- Any envelope requirements, (i.e. clearance to other objects, handling equipment, space for removing, swing away design etc.)

Process Data:

- Flow Rate.
- Allowable or desired flow rate per hole (determines number of holes).
- Operating Temperature.
- Process material (s), Basic description/ formulation. Rheology data on material (s). (see photo's #1 & #2)
  Viscosity as a function of temperature sensitivity (Poise).
  (Also the no flow temperature)
  Viscosity as a function of shear rate or shear stress (Poise)
  (Shear sensitivity exponent, commonly known as the power law exponent for the fluid)
  Melt density (Grams per cubic Centimeter. As close to operating temperature as possible.
  If the materials have Bingham, or Dilatant characteristics, we would need to know the parameters at which the changes occur as a function of shear rate (or shear stress) and temperature. (see sketch #1A & #2)

For a given die design,(hole geometry, channel geometry, breaker plate configuration, screen pack requirement etc.) and a given material and flow rate, an overall pressure drop can be calculated, as well as a pressure drop across the face of the die. (see sketch #1-B)

The value of obtaining the pressure drop across the face of the die is to ensure uniformity of strand (or sheet) velocity. One would then need to go back and calculate (or mathematically model) the extruder screw performance ("A" term-"B" term) for the given screw design that is to be utilized. This is done to ensure that it will produce the volumetric displacement at the calculated pressure drop within the required temperature rise to achieve the desired results for the application. (see sketches #1-C & #1-D)

For these calculations, more characterization and geometric data is required. A design evaluation of mechanical properties for the die is also a requisite to ensure proper safety factors exist in relation to the design pressure specified. Finally, a manufacturing evaluation is required to ensure the components and assembly can be constructed within the design parameters for the projected costs and delivery needs.

Other factors may also influence the development of an optimum die for a unique application. However, it is plain to see that it is not likely that one entity would have all of the expertise, equipment and technology to perform all of these tasks with the desired results. This is where the value of partnering of resources becomes obvious.

Partnering is nothing more than developing a team effort to solve a problem (capitalizing on an opportunity). Except that partnering is a long-term relationship. When implemented most effectively, it will develop on a macro scale and a micro scale. Macro scale inter company, Micro scale intra company. It is an approach that involves good project management, an overall knowledge of the requirements and a good experience level with qualified support groups. Unlike standard approaches to new product/production development, which traditionally are series flow programs, partnering normally yields series/parallel efforts. This nominally reduces the time to get the product to market and subsequently simplifies the equipment design, lowering production equipment and operational costs. This process requires excellent communication and information exchange with cross sharing as necessary. Each team member brings a technical competence in a specialized technology to the project. In most cases this technology is considered state of the art. Co-ordination and common sense oversight of the project and its activities is essential for efficient practical application of the technology. The group effort must all be directed toward the same goal with a sense of urgency. This concept when implemented properly will address the issues of minimal lead time, lowest product cost with optimum performance, and highest quality of the end product.

The employment of these techniques in the design and manufacture of hardware for a first time process results in significant time saving's, accuracy of final design and optimum quality of product.

The application of this philosophy can be demonstrated in this industry through reviewing examples of several joint development efforts for new production capabilities at Naval Surface Warfare Center (NAVY) in Maryland, and Picatinny Arsenal (ARMY) in New Jersey. Team members resided in NJ,MD,UT,MI.

During the last two years three dies for Naval Surface Warfare Center , IndianHead Md. (NSWC/IH) and Picatinny Arsenal (PA) were designed and manufactured using mathematical modeling.

These included the 2.75" rocket motor die for the CPOCP program and two-strand gun propellant die (involving an energetic thermoplastic elastomer) for the Green Energetics Manufacture (GEM) program for NSWC and a single strand die for continuous extrusion of PAX II explosive. For each die, the following steps were taken.

1. Highly filled Materials Institute @ Stevens Institute of Technology (HfMI/SIT) evaluated the ingredients of the formulation and identified/calculated key characteristics including sizes of particles, and maximum packing fraction.
2. The formulations were prepared in batch and their rheological behavior was characterized at NSWC under Dr. S. Prickette's direction for the CPOCP and GEM program and at Thiokol for the PAXII program. The data were electronically transferred (e-mailed) to HfMI/SIT.
3. HfMI/SIT used the data to determine the parameters of viscoplastic constitutive equations and Navier's wall slip condition using a combination of Finite Element Model (FEM) calculations and analytical models of viscometric flows.
4. HfMI/SIT identified the key performance and product requirements including density, weld-line strength, variation in particle concentration.
5. Analytical methodologies were used to select candidate geometry's for each die. FEM meshes of the candidate die geometry's were built at SIT.
6. The mathematical model of the die flow was carried out using three dimensional FEM at SIT for the internal channels of the die. The model provided the three dimensional distributions of velocity, stress, strain, shear rate, residence time and temperature.
7. Modifications on the internal geometry were made until all performance criteria were satisfied.
8. This geometric form was translated to an AutoCAD file and then transmitted electronically to Shear Tool, Inc. in Saginaw MI. At the same time Naval Surface Warfare Center in Indianhead, Maryland transmitted the geometry of the machine that the die was to mate up to too Shear Tool via the same electronic media.

Shear then designed the external features of the die from the internal surface model and the external mating requirements of the extruder and propellant grain receiver. When looking at the design, they applied techniques based on efficient manufacturing methods and split components accordingly. They also made minor modifications to the insides of the die to improve manufacturability.

9. Preliminary computer drawings were then electronically transferred to SIT and NSWC. SIT evaluated the suggestions by either analytical or numerical simulation means for their impact on the thermo-mechanical history of the energetic material in the die and ramifications on performance criteria. Various alternatives were also evaluated. These were discussed on conference calls between the two groups until the best compromise of manufacturing and process requirements were reached.
10. Meanwhile NSWC reviewed the design for operation, maintenance and safety issues. Their concerns and suggestions were electronically transferred to Shear for incorporation. Some of these affected component manufacture and conference calls between Shear and NSWC resolved these issues with the best compromise for both parties. Additionally, materials of construction, surface finishes tolerances, back up components and a recovery strategy in case of performance problems were deliberated and solved via joint conference communication. Shear provided strength calculations and certifications while NSWC provided safety-modeling calculations.
11. From this evaluation, the final design in an Auto-CAD file emerged. After a final review all parties signed off on the final design and the die went into production.
12. Computer Aided Drafting (CAD) designs were electronically transferred and loaded into computers to develop programs used for running computer numerical controlled machines. The programs were transferred to the manufacturing equipment through post processors which automatically translate them to the unique machine language before being sent to the specific manufacturing machinery through a distributed control system network. The manufacturing materials were then loaded in the machines and the parts were produced.

13. The die, when completed in manufacturing, was inspected by Shear Tool, SIT and NSWC or Army personnel. Upon acceptance the die was installed and tested using first simulant, then the live formulation.
14. The pressure and temperature history data were compared with the predictions of the mathematical model to assure that the changes in the material did not take place and the rheological data and geometry's, were accurate.
15. The ultimate properties of the extended grains and the compliance with performance criteria including density, weld-line strength etc. were evaluated to ensure that the die was acceptable without reservations.(see photos #3 & #4)

For the three dies designed and manufactured this procedure resulted in three dies, which were found to be acceptable without reservation. No trial and error procedures were ever required during this program and the project was successful.

A similar project was run with another department at NSWC where the problem was the replacement of a nine-strand ram extrusion die. The die was worn out from many years of use, and was in dire need of replacement. The department head had previously discussed with Shear Tool, the possibility of replacing this die.

He said the performance of this system which produced propellant over a 20-year period, nominally ran 40 + % rejects, (scrap rate) on the final product. Shear looked at the old die and suggested from practical experience, the die looked like a poor design based on today's technology. They suggested the department head contact HfMI to see if they felt important performance improvements could be achieved by modifying the process flow path through the die.

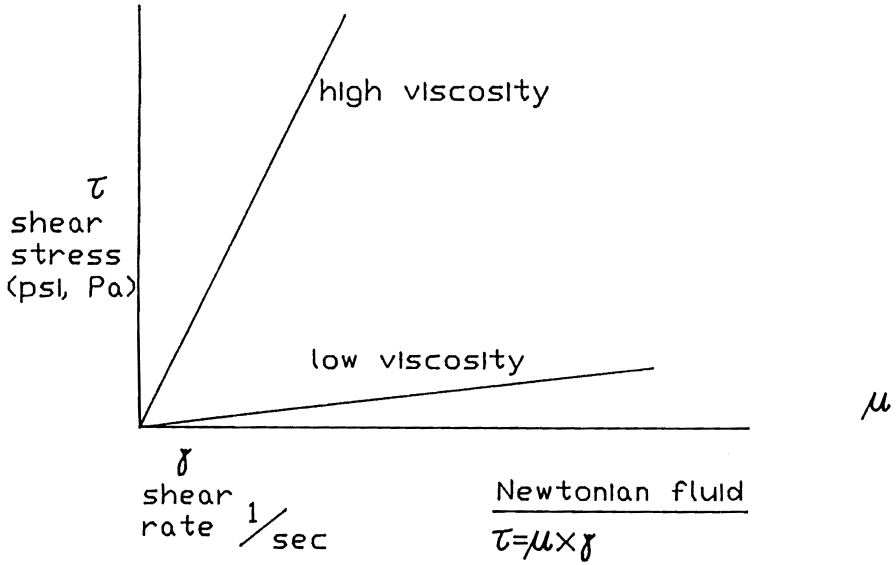
After consultation, HfMI felt that a redesign could result in a dramatic performance improvement. HfMI's materials characterization showed the product to be extremely sensitive to thermal excursions, and saw major fluctuations in viscosity as a function of this. The material was also very shear sensitive. HFMI modeled the inside for flow characteristics while Shear looked at a redesign of the cooling channels and circulation paths for improved temperature control as well as isolation from the large thermal mass effect of the ram extruder. The same team/partnering concept was employed, using computer simulation, computer design, computer manufacturing, co-ordinated by electronic data transfer and Tele-conferencing. The result was a process system that went back into operation with a reject (scrap rate) of 1%. The department manager received the NSWC annual "Cost Reduction Award" award for this achievement. (see photo #5)

The same concepts were employed at Picatinny arsenal for the development of a die insert for Low Vulnerability Ammunition. When the die inserts were coupled with the right screw design, the results were very positive, with the extrusion of an acceptable propellant for a foreign consumer.

While most of the work with government groups has revolved around the design of extrusion components, many commercial producers have utilized these techniques very successfully to develop complete production systems. In these instances, more development/planning work is required and more support sub teams need to be formed. Each has to address their unique area of expertise individually at first, then be brought into an overall concept review to integrate, via the best compromise, everyone's best efforts into the final production system.

In order for this process to work effectively, some key elements are necessary. There must be mutual respect for and trust in each partners technical area of expertise. Each must be willing to learn from the others while providing their best input. All must be willing to compromise for the good of the whole. Every partner must be willing to share all his or her information with the others. Everyone must be thinking quality, economics, performance, efficiency, and expediency.

It's not difficult to optimize products and processes, but it does require a combination of technical expertise, good planning, expedient execution and genuine co-operation. All common sense issues and all steps to future expansion, future growth and in general a positive future.

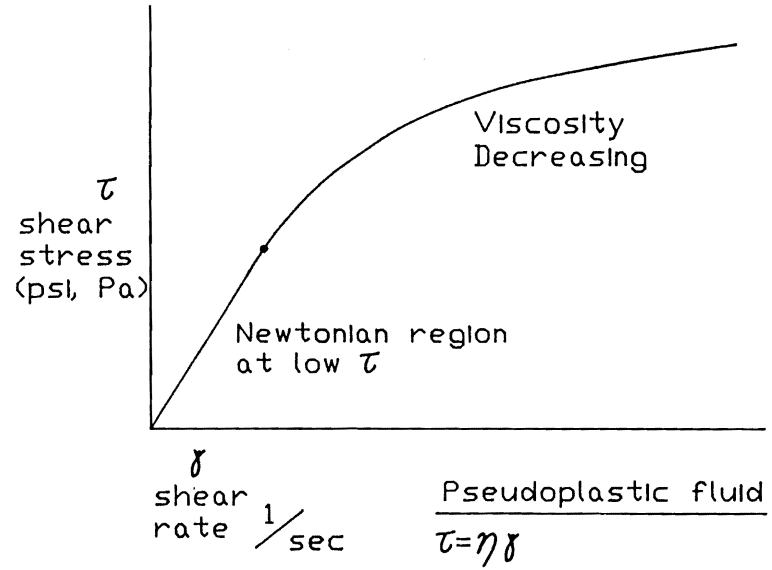


μ

Newtonian fluid

$\tau = \mu \times \gamma$

is constant



η

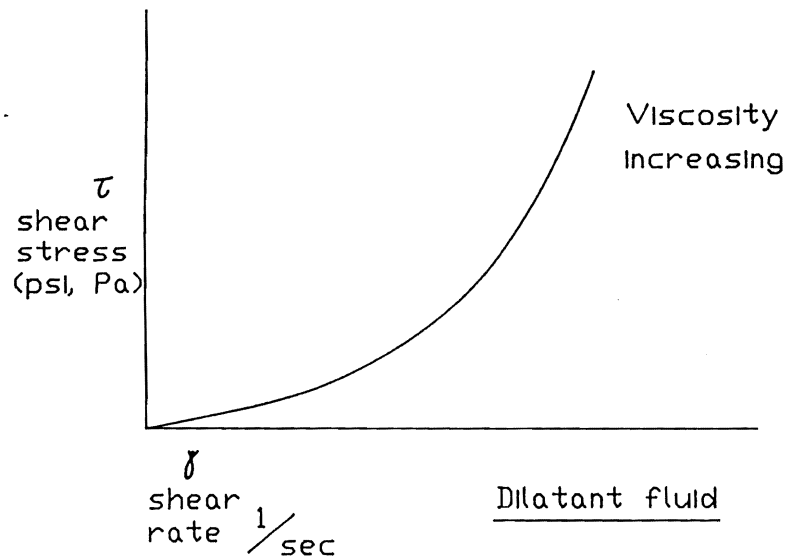
γ

Pseudoplastic fluid

$\tau = \eta \gamma$

Power law fluid

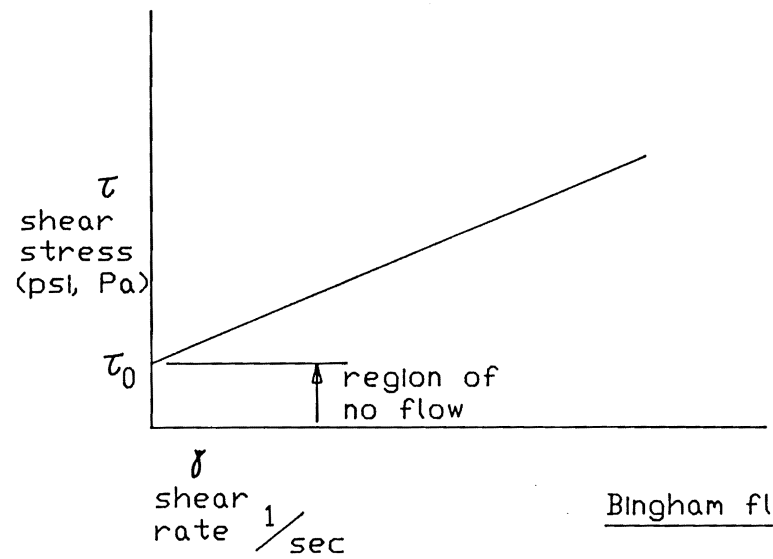
if $\eta = m\gamma^{n-1}$ in certain regions



Dilatant fluid

Clay dispersions

Silly putty

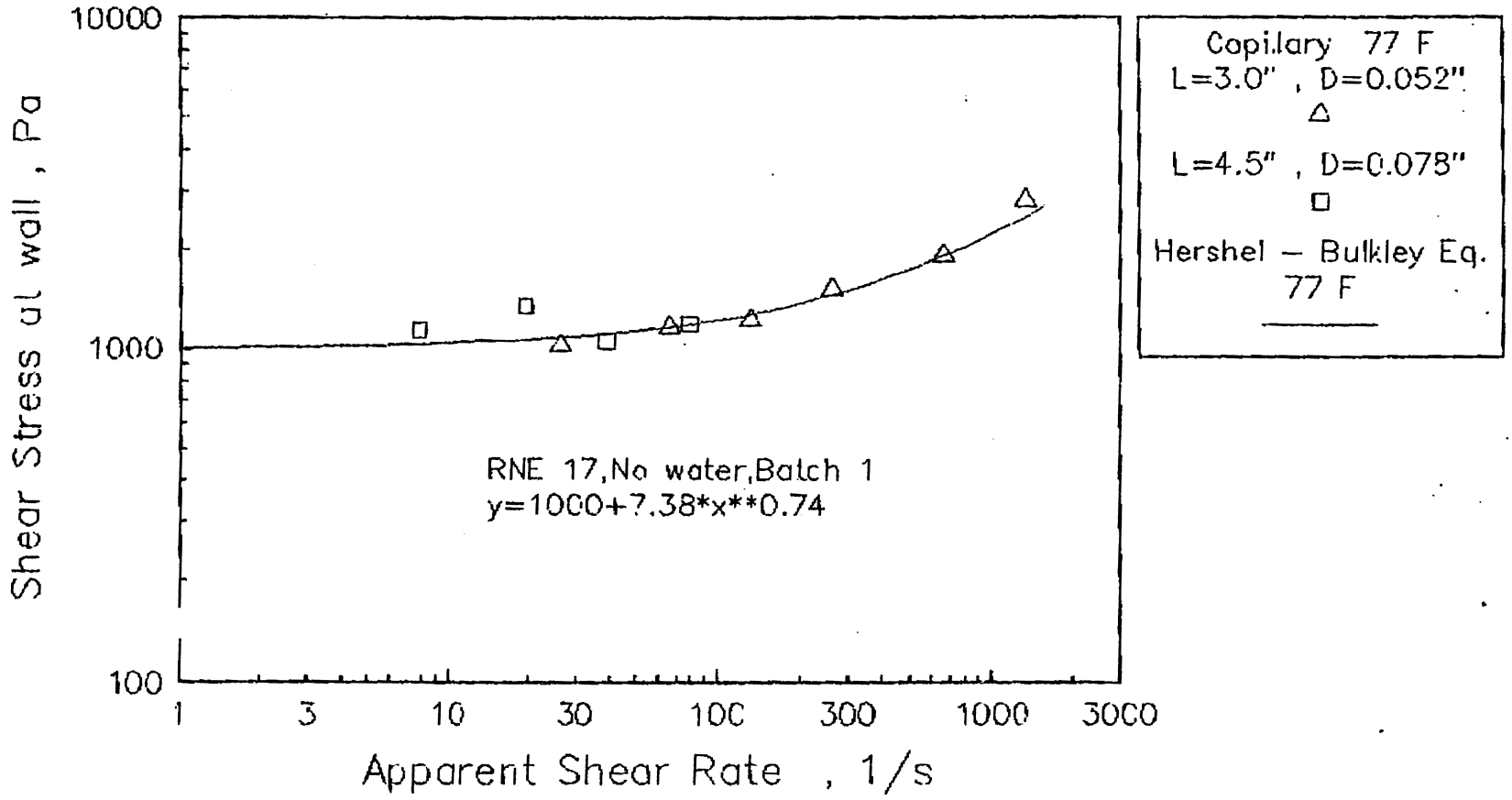


Bingham fluid

Ketchup

SKETCH

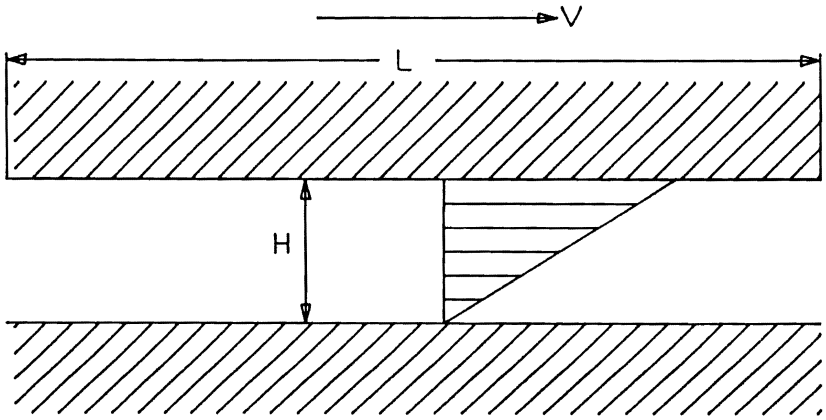
1-A



SKETCH -2

TYPICAL EXAMPLE BINGHAM FLUID

FROM CH CHE STEVENS 201 216 8240



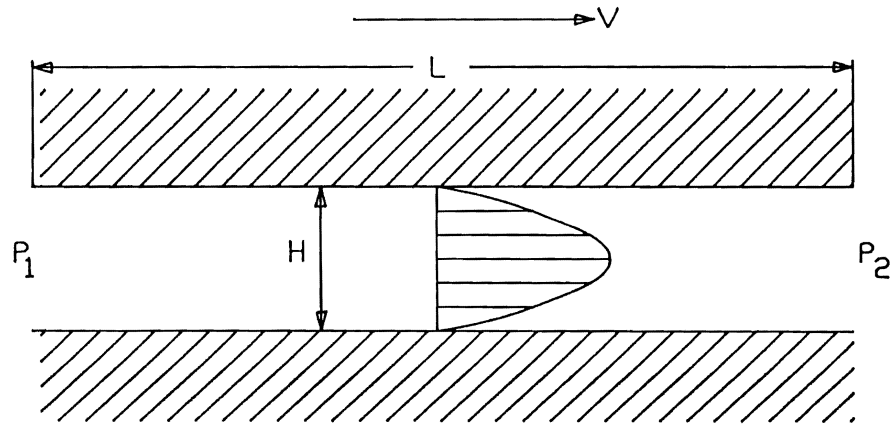
Drag flow

Shear rate $\gamma = \frac{V}{H}$

Shear stress $\tau = \mu \gamma$

Flow rate (per unit width): $Q = \frac{VH}{2}$

Force (per unit width) $F = L\tau$

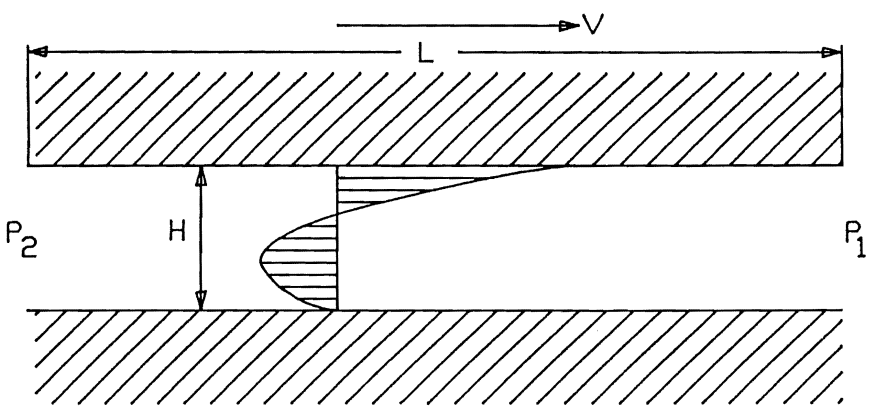


Pressure flow

Shear stress (τ_w at the wall) $= \frac{H(P_1 - P_2)}{2L}$

Shear rate (γ_w at the wall) $= \frac{H(P_1 - P_2)}{2\mu L}$

Flow rate (per unit width): $Q = \frac{H^3(P_1 - P_2)}{12\mu L}$

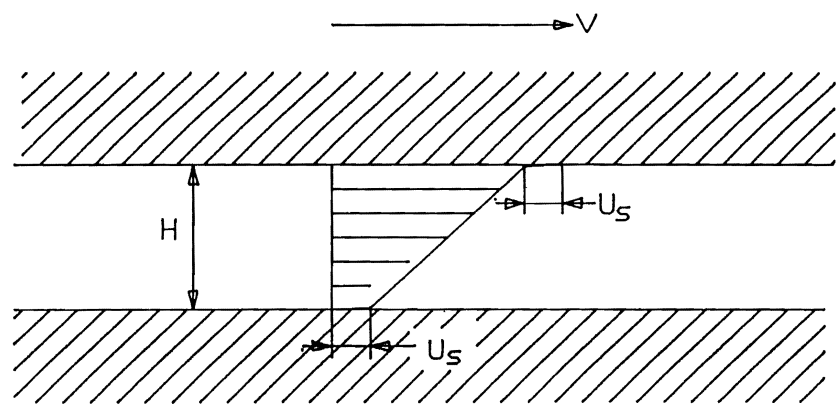


Drag- and pressure flow

Shear stress at the wall:

$$\tau = \frac{V}{H}\mu \pm \frac{H(P_1 - P_2)}{2L}$$

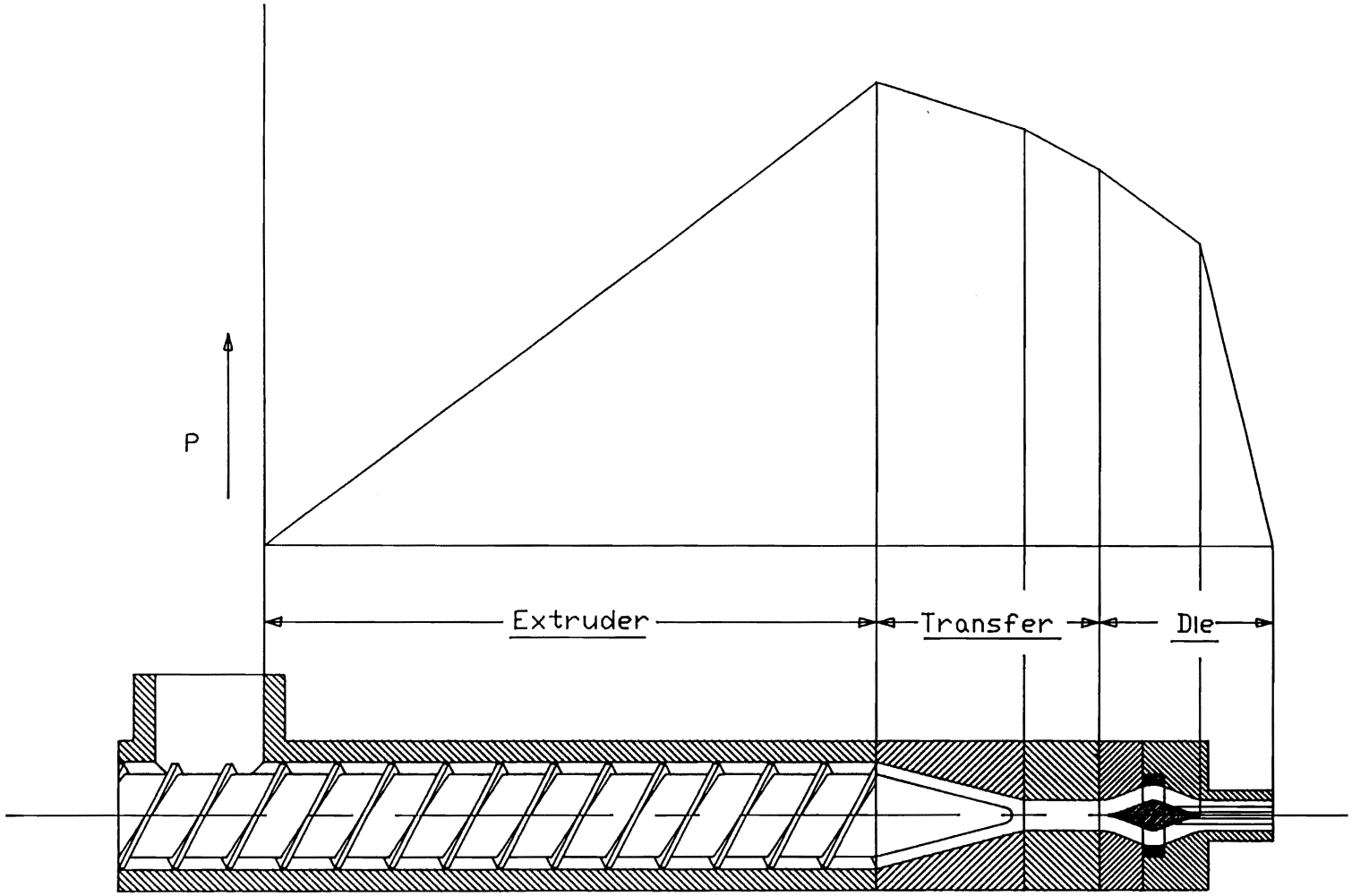
Flow rate (per unit width) $Q = \frac{VH}{2} - \frac{H^3(P_1 - P_2)}{12\mu L}$



Wall slip

$U_s = C_1 \tau_w^{n_1}$

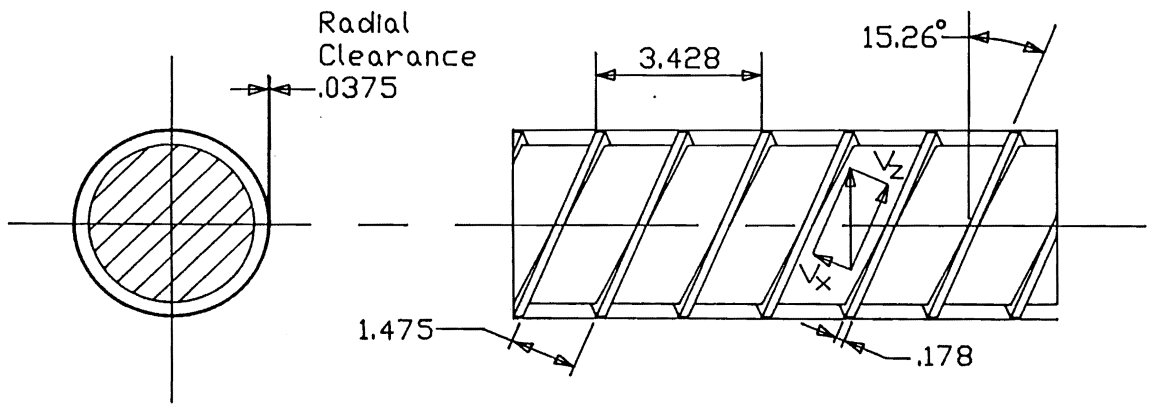
SKETCH 1-B



Schematic pressure diagram

SKETCH
1 – C

A.S.R.M. Extruder screw data



Geometry

$$Q = A*n - B\frac{\Delta P}{\mu}$$

$$A = \frac{W*(H-c)*V_z*2}{2}$$

$A = 4.48\ in^3$

$$B = \frac{W*H^3}{12*L}2$$

$B = .000134\ in^3$

Viscosity

$$\eta(\gamma,T) = \sigma_0(T) + k(T)\gamma^{m(T)}$$

$$\sigma_0(T) = -55.2 + 2.17T$$

$$k(T) = 1.09*10^4 - 113T$$

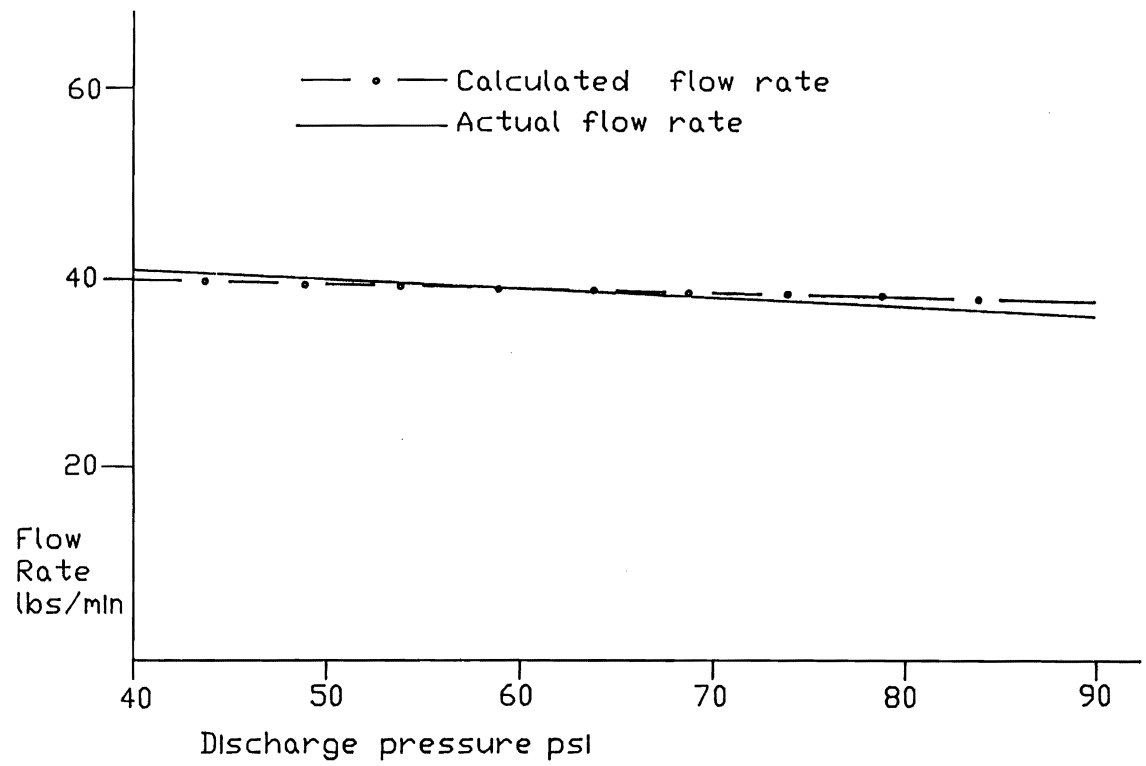
$$m(T) = -0.141 - 1.76*10^{-4}T$$

Wall slip

$$\tau_{w,PA} = 13860 + 200\gamma_w$$

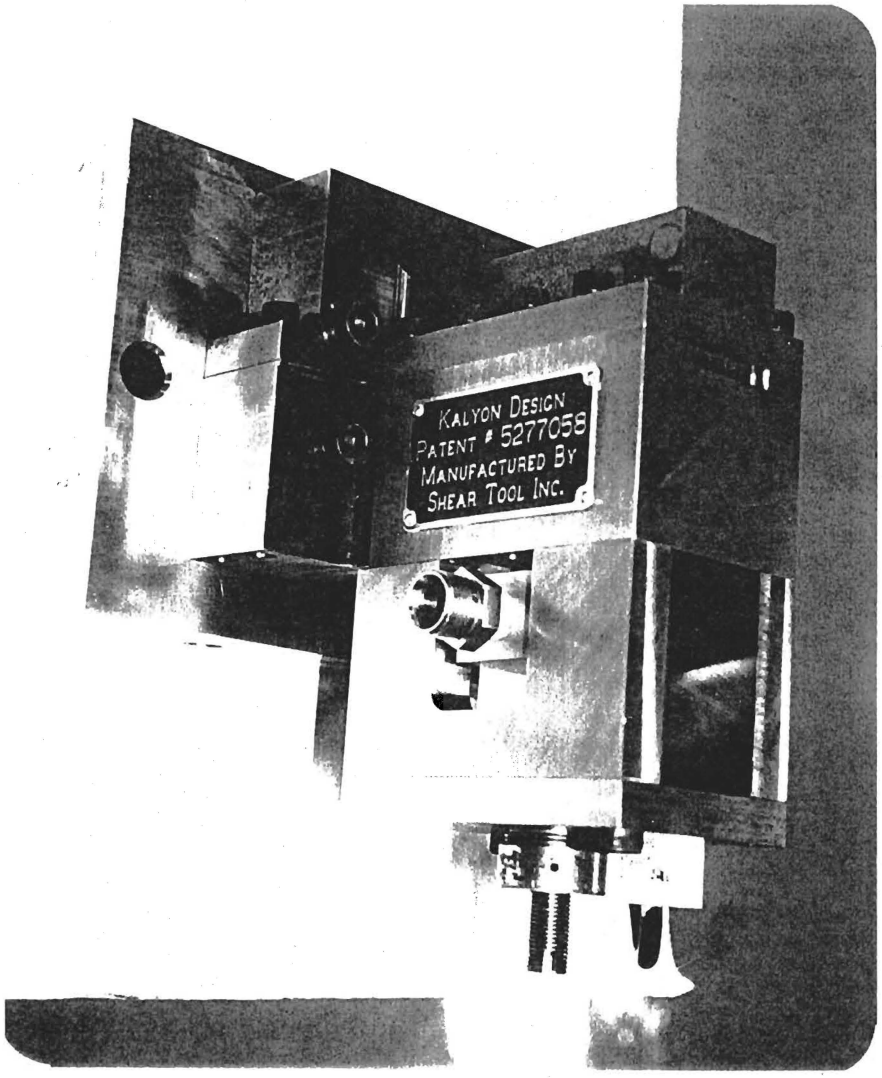
$$Us_{mm/sec} = 0.0016\ \tau_w^{0.805}$$

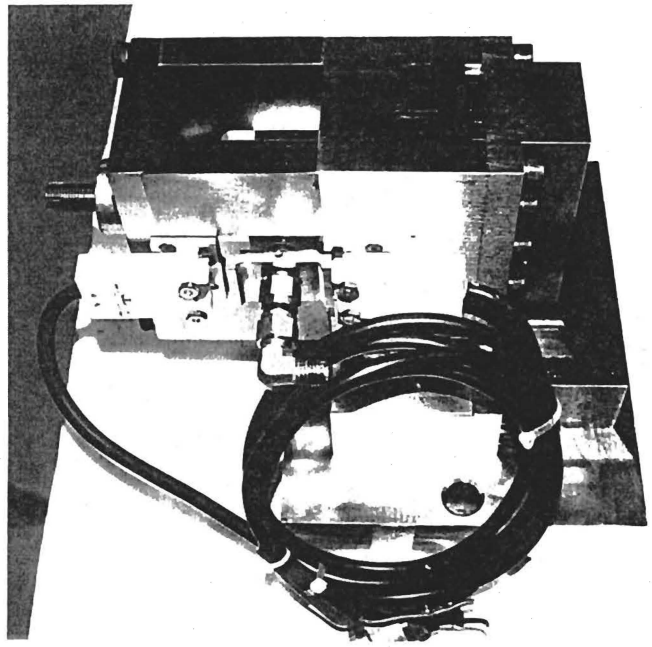
Results

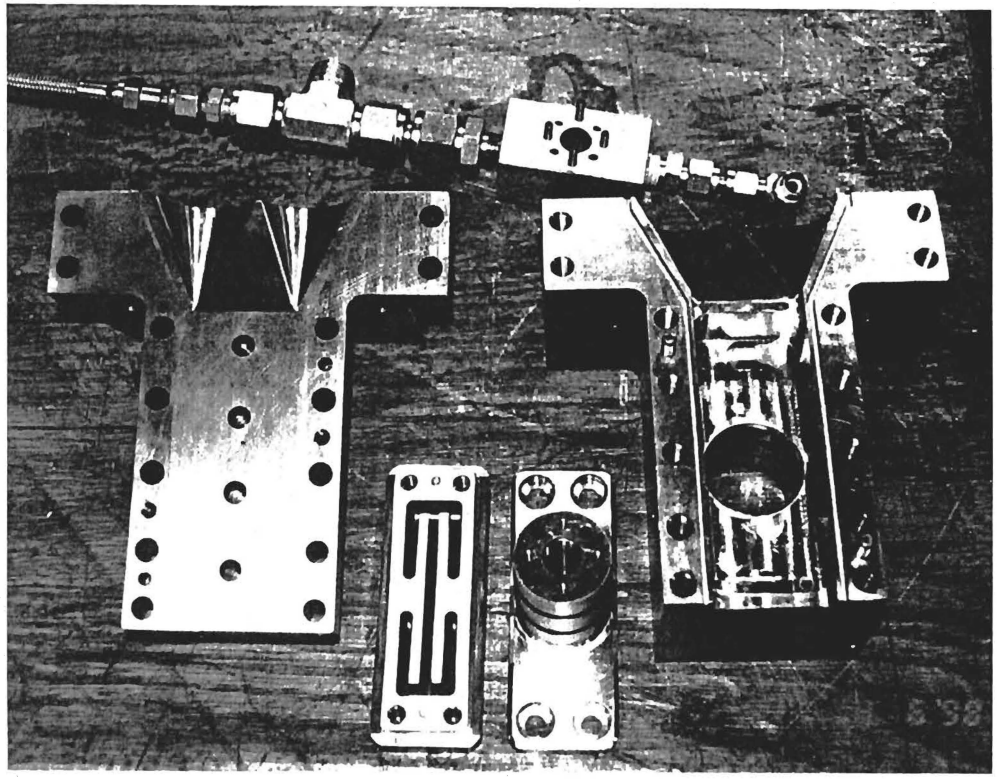


Example of calculated flow-vs-actual flow

SKETCH 1-D







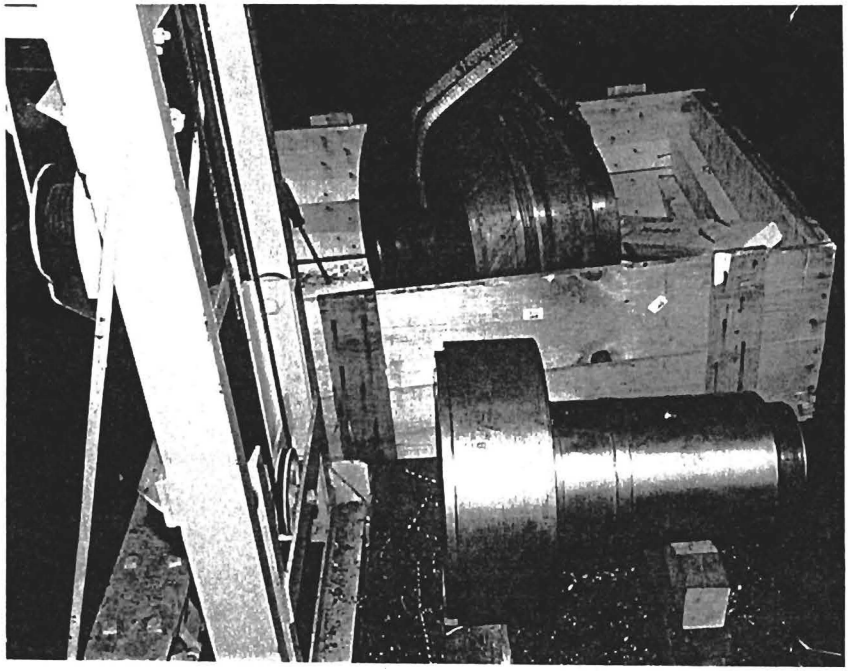
KALYON~Adjustable Gap~RHEOMETER

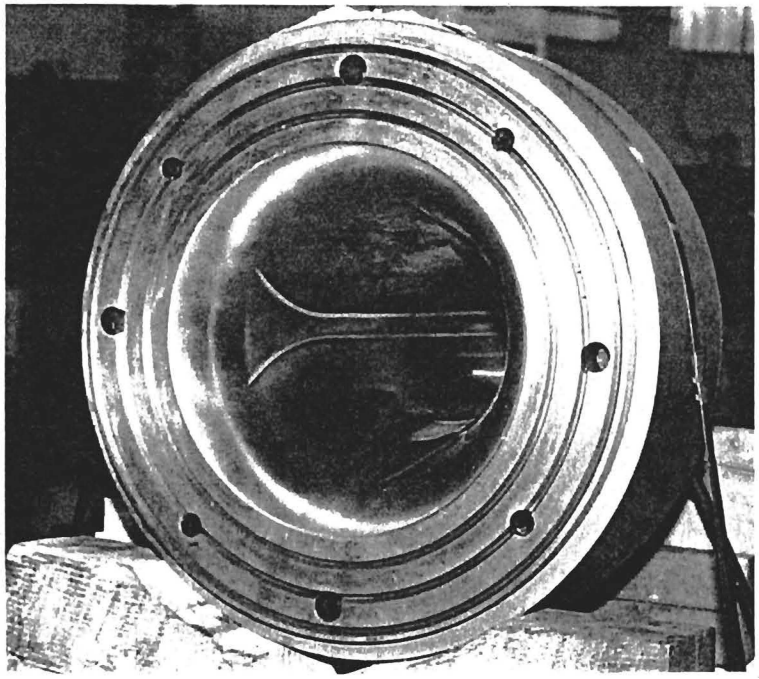
Measurement of

Shear Viscosity & Wall Slip Behavior

from On-line EXTRUDERS- Twin or Single Screw or Ram

Photo #1

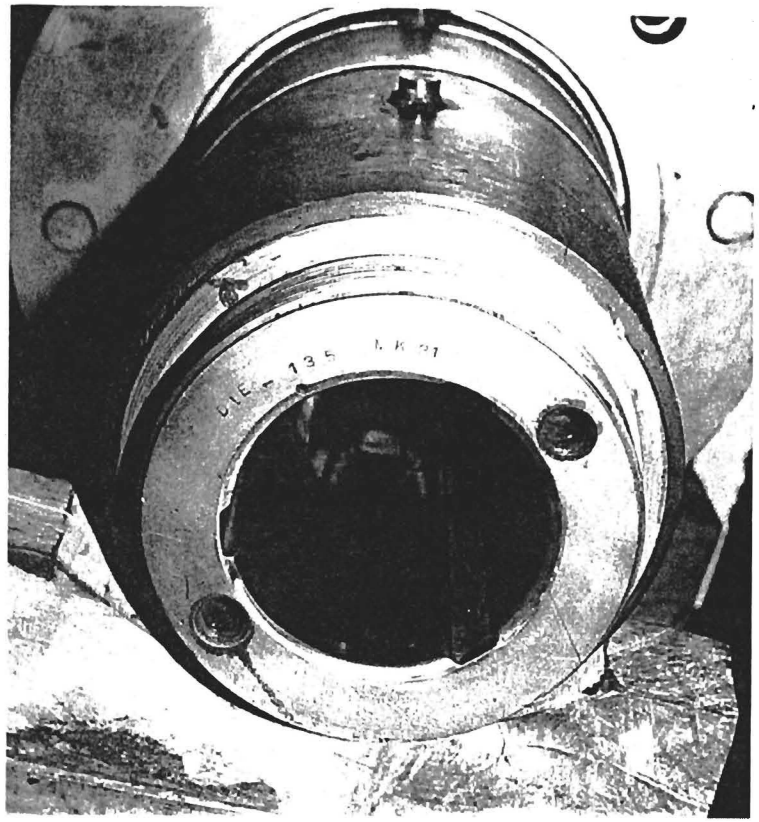




Existing Die Unit

Modified for

Rheology Data Acquisition



Jacketed Die Retainer Body

Retrofitted

for

Thermocouples & Pressure Transducers

for

Acquisition of Rheology Data

Photo #2

EXTRUSION DIE

for

2.75" Rocket Motor

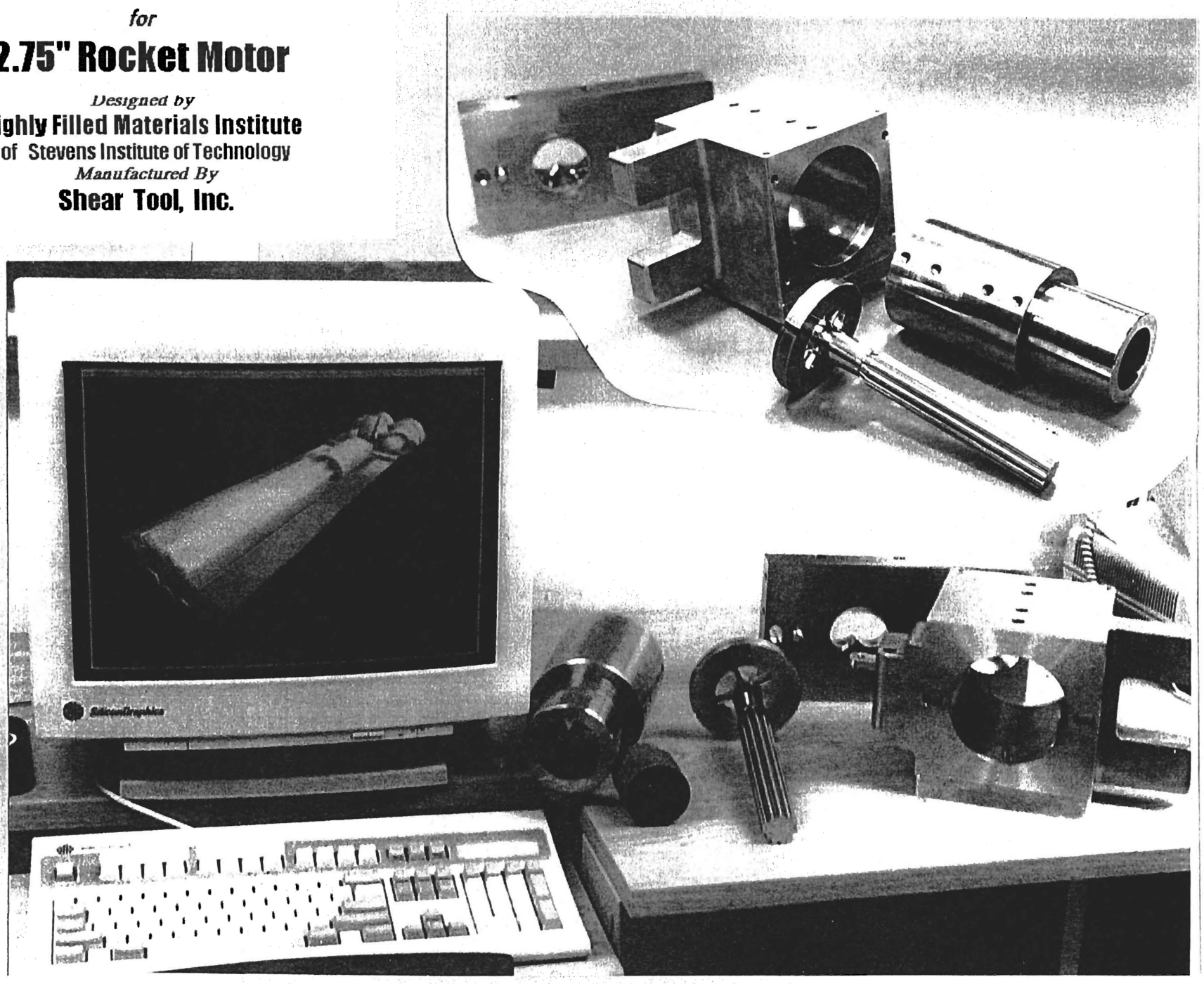
Designed by

Highly Filled Materials Institute

of Stevens Institute of Technology

Manufactured By

Shear Tool, Inc.



Photo#3

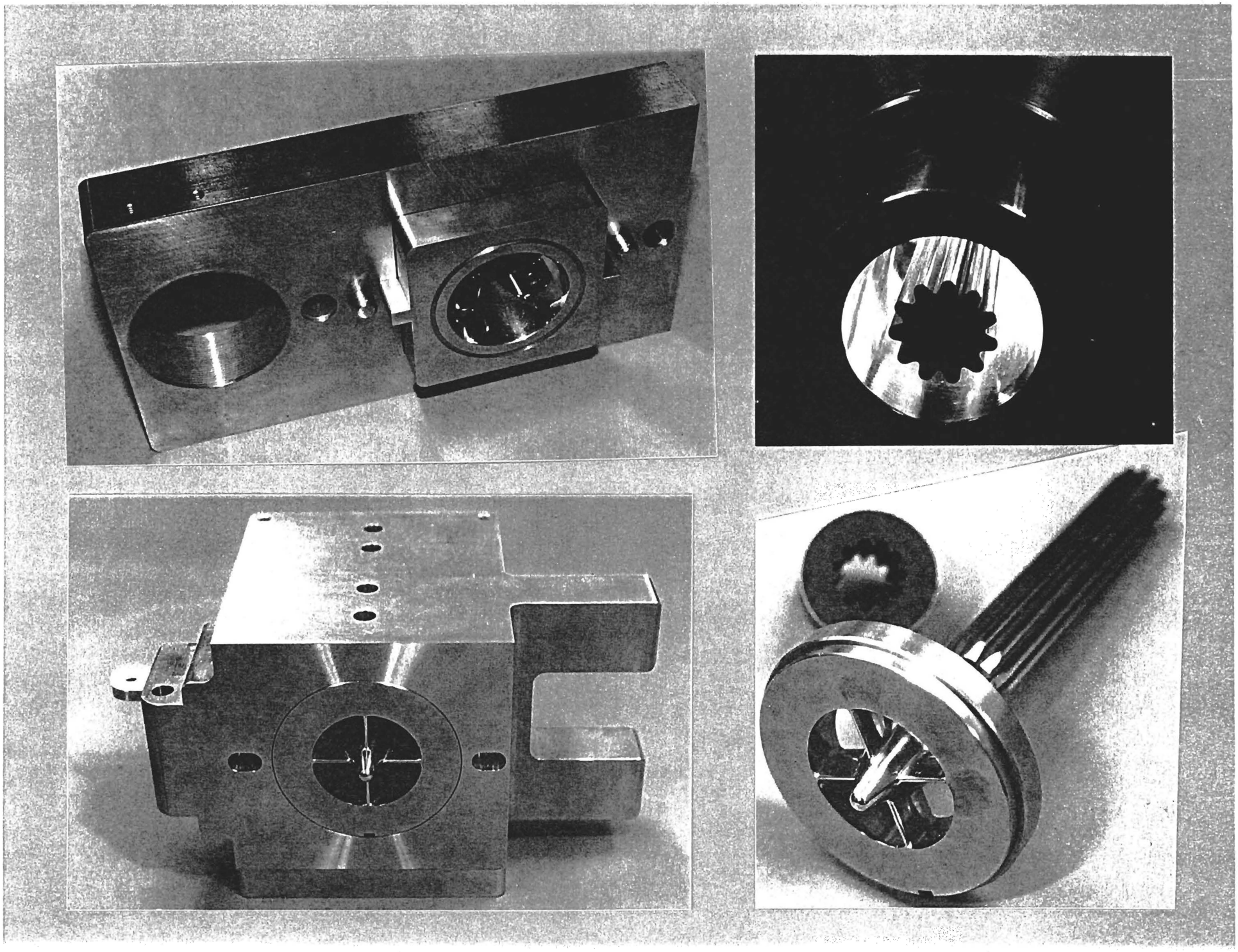
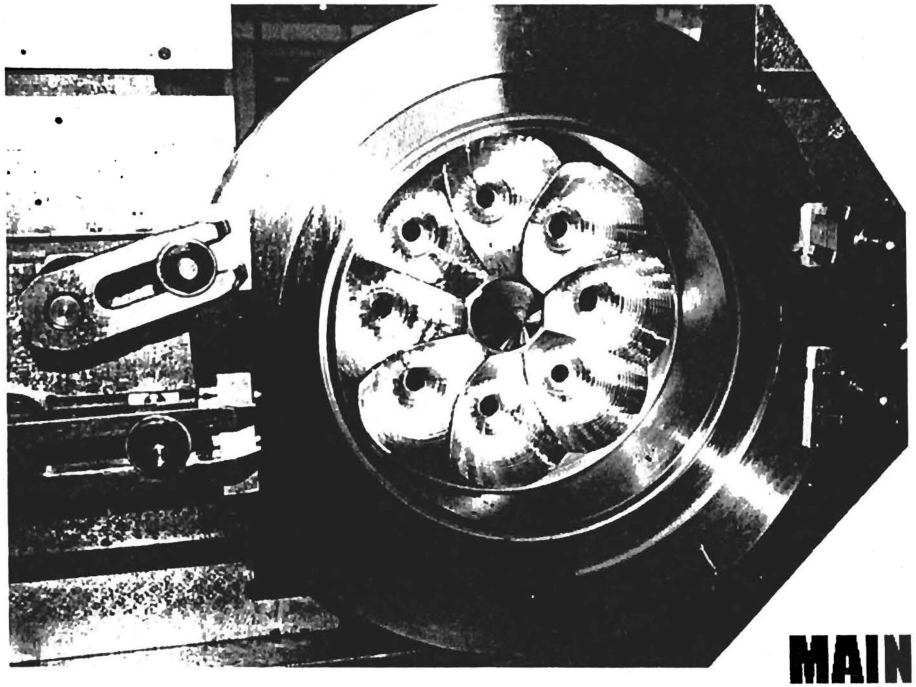
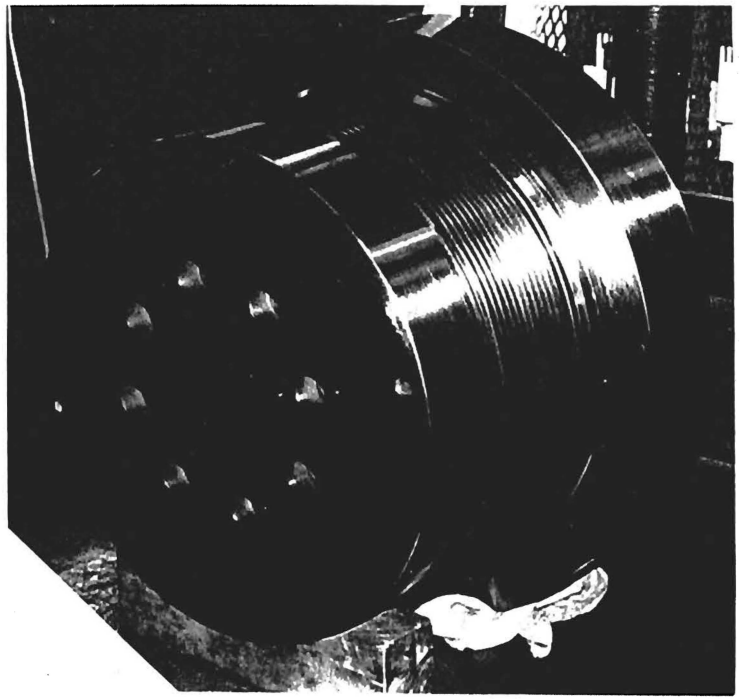


Photo # 4



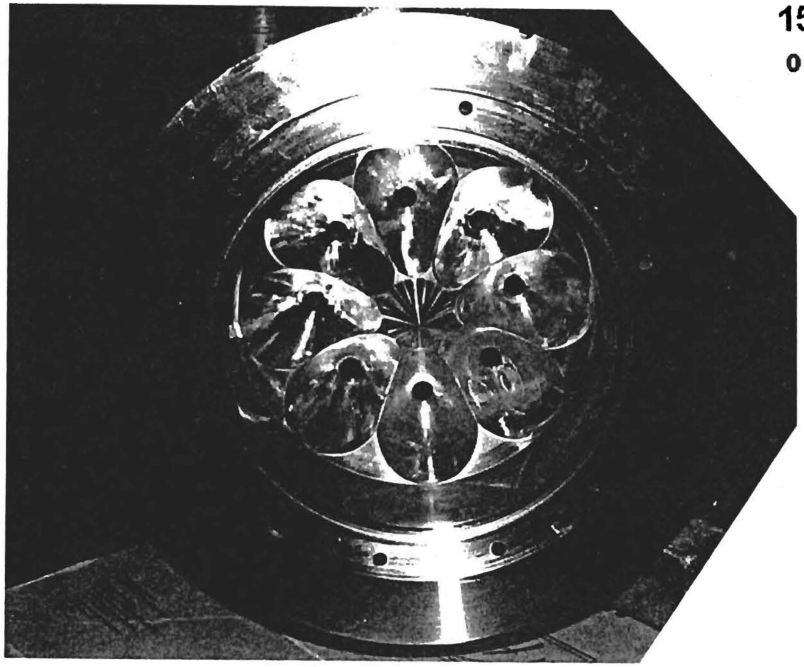


MAIN DIE BODY

Eight Hole-Insert / Cylindrical / Jacketed

for

15" Ram Press Extruder on Energetics Application



Cost
Reduction
Award

Jose R. Leon

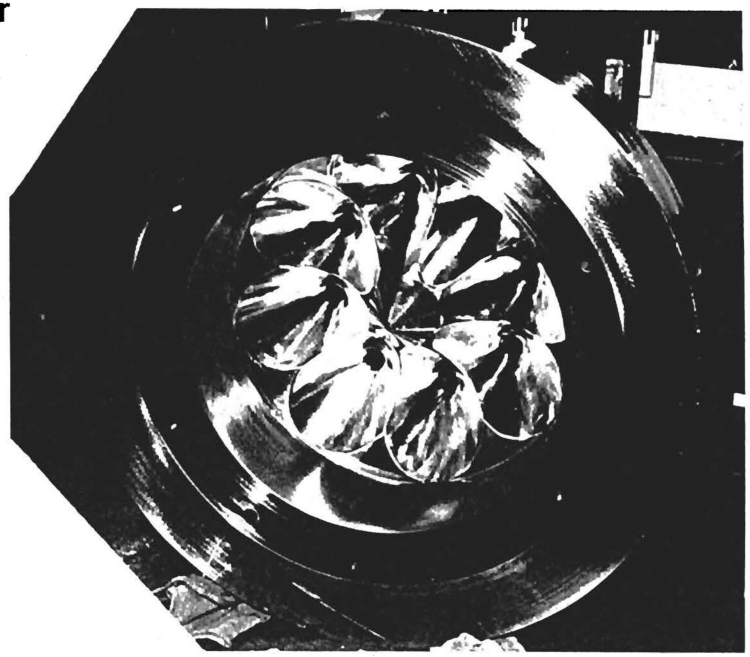


Photo #5

ON-LINE AND OFF-LINE ANALYSIS OF THE EXTRUDED AND CAST ENERGETRIC MATERIALS FOR MICROSTRUCTURAL DISTRIBUTION

Rahmi Yazici and Dilhan M. Kalyon

Highly Filled Materials Institute
Stevens Institute of Technology, Hoboken, NJ
www.hfmi.stevens-tech.edu

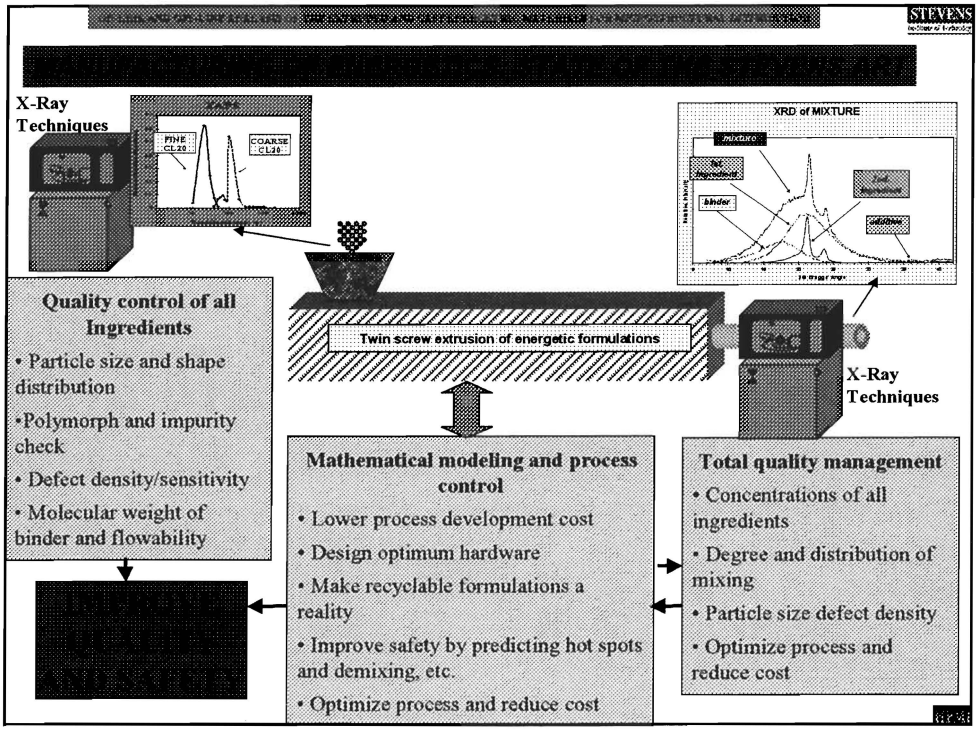
1999 Extruder Users Group Meeting

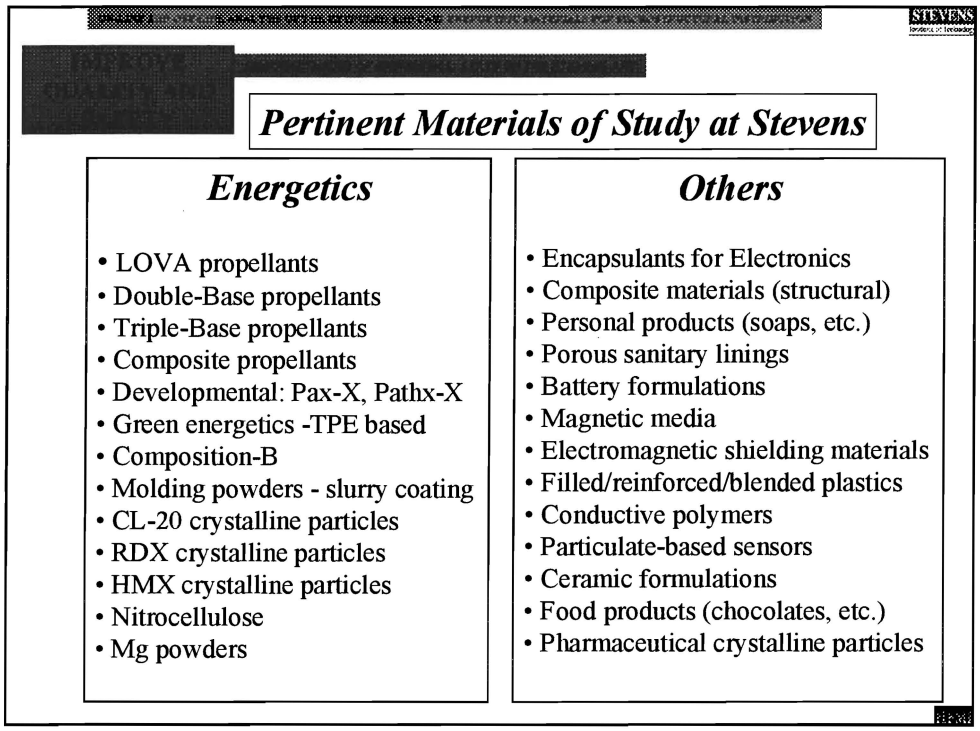
SUMMARY

The performance of energetic grains depend on the concentration distributions of the ingredients of the formulation, the degree of mixedness as a function of the scale of examination and location, the particle size, shape, orientation and defect density distributions and the coating thickness of the particles.

In this presentation comprehensive x-ray techniques developed at SIT will be reviewed in conjunction with recent case studies involving propellants, rocket fuels and other industrial materials.

The techniques are amenable to on-line analysis and will be demonstrated with SIT's new state-of-the-art on-line set-up established at its twin screw extrusion facility in Hoboken, New Jersey.

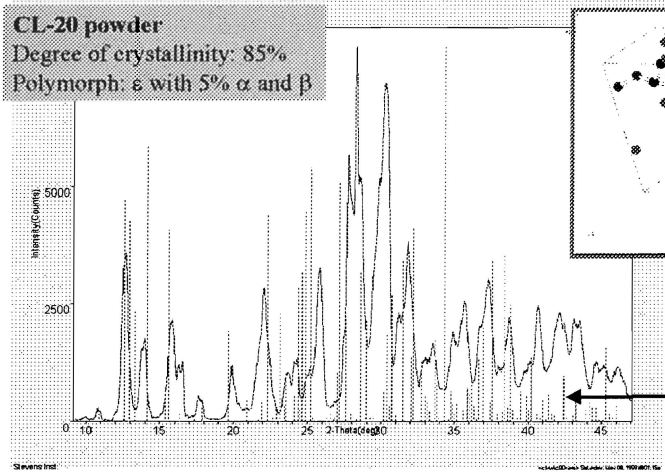

STEVENS
X-Ray Techniques
FINE CL-20
COARSE CL-20
XRD of MIXTURE
mixture
binder
Quality control of all Ingredients
• Particle size and shape distribution
•Polymorph and impurity check
• Defect density/sensitivity
• Molecular weight of binder and flowability
Twin screw extrusion of energetic formulations
X-Ray Techniques
Mathematical modeling and process control
• Lower process development cost
• Design optimum hardware
• Make recyclable formulations a reality
• Improve safety by predicting hot spots and demixing, etc.
• Optimize process and reduce cost
Total quality management
• Concentrations of all ingredients
• Degree and distribution of mixing
• Particle size defect density
• Optimize process and reduce cost

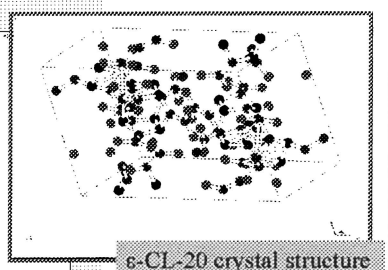

STEVENS
Pertinent Materials of Study at Stevens
Energetics
• LOVA propellants
• Double-Base propellants
• Triple-Base propellants
• Composite propellants
• Developmental: Pax-X, Pathx-X
• Green energetics -TPE based
• Composition-B
• Molding powders - slurry coating
• CL-20 crystalline particles
• RDX crystalline particles
• HMX crystalline particles
• Nitrocellulose
• Mg powders
Others
• Encapsulants for Electronics
• Composite materials (structural)
• Personal products (soaps, etc.)
• Porous sanitary linings
• Battery formulations
• Magnetic media
• Electromagnetic shielding materials
• Filled/reinforced/blended plastics
• Conductive polymers
• Particulate-based sensors
• Ceramic formulations
• Food products (chocolates, etc.)
• Pharmaceutical crystalline particles

Evaluation of process ingredients: *powders*

Particle polymorph, degree-of-crystallinity, molecular packing analysis by wide angle x-ray diffraction.

CL-20 powder
Degree of crystallinity: 85%
Polymorph: ε with 5% α and β

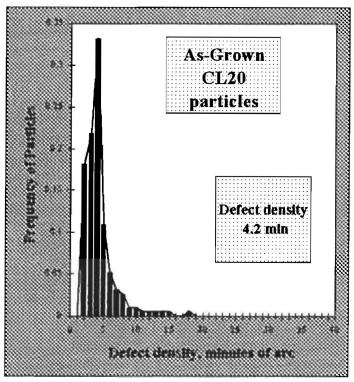


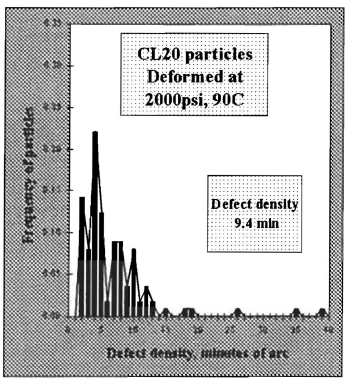


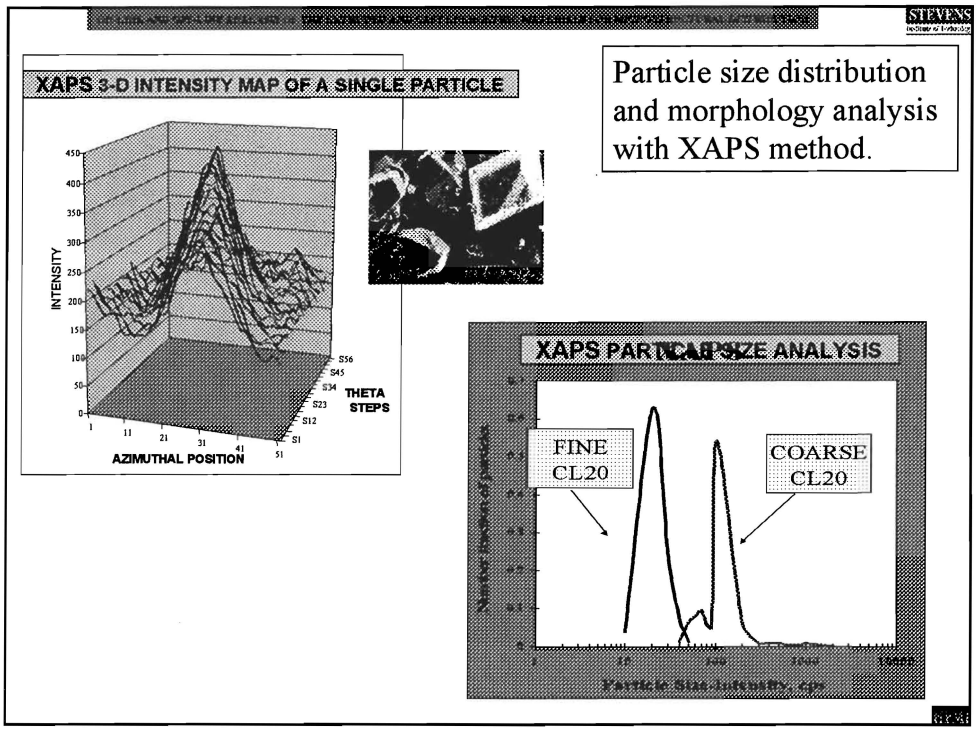
ε-CL-20 crystal structure and molecular packing

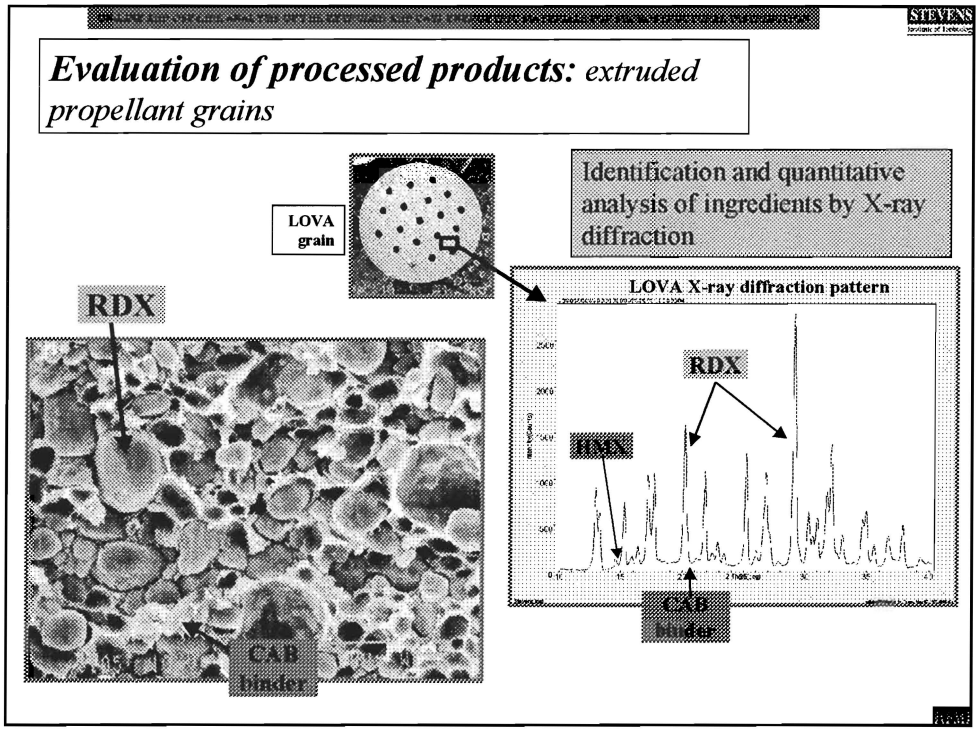
ε-CL-20 diffraction pattern (lab-model)

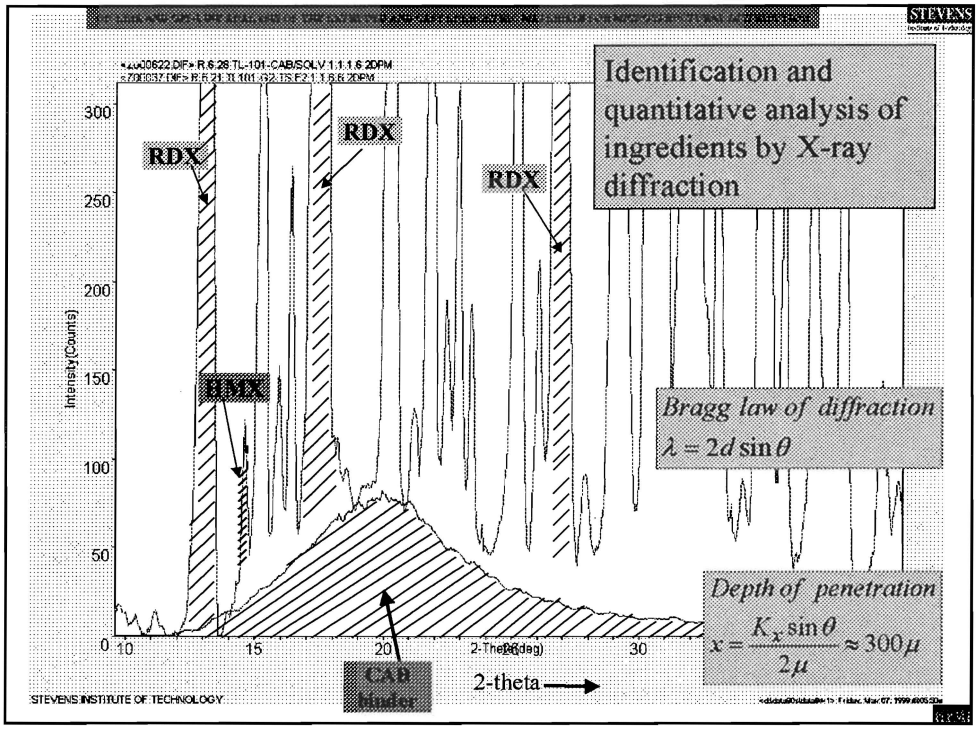
PARTICLE DEFECT DENSITY DISTRIBUTION ANALYSIS by novel **XAPS** method






STEVENS
XAPS 3-D INTENSITY MAP OF A SINGLE PARTICLE
INTENSITY
AZIMUTHAL POSITION
THETA STEPS
Particle size distribution and morphology analysis with XAPS method.
XAPS PARTICLE SIZE ANALYSIS
FINE CL20
COARSE CL20


STEVENS
Evaluation of processed products: extruded propellant grains
LOVA grain
Identification and quantitative analysis of ingredients by X-ray diffraction
LOVA X-ray diffraction pattern
RDX
HMX
CAB binder
RDX
CAB binder



MEASURE

$$CAB\ relative\ x-ray\ diffraction\ intensity = \frac{I_{CAB}}{I_{RDX}}$$

DETERMINE

$$CAB\ relative\ volume\ fraction = \frac{I_{CAB}}{I_{RDX}}$$

the absorbtion coefficient of a mixture

$$\mu_m = \phi_1\mu_1 + \phi_2\mu_2$$

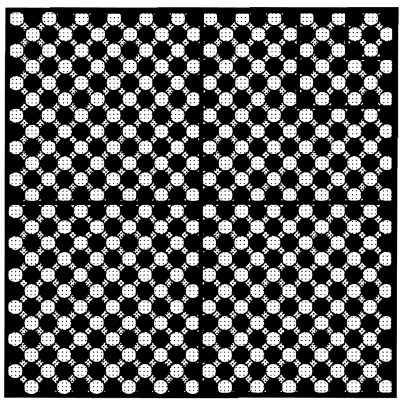
integrated intensity from ingredient 1

$$I_1 = K_1\phi_1 / \mu_m$$

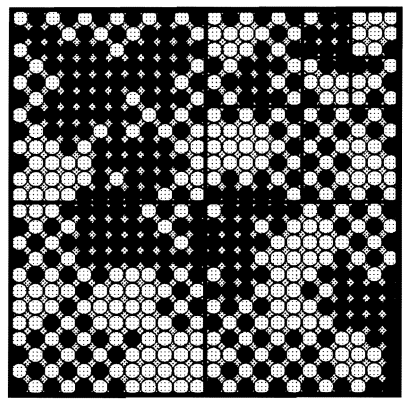
relative intensity ratio of ingredients 1 *and* 2

$$\frac{I_1}{I_2} = \frac{K_1\phi_1}{K_2\phi_2} =$$

DEGREE OF MIXING ANALYSIS BY
WINDOW MEASUREMENTS AT VARYING SCALES OF EXAMINATION



Ideal degree of mixing
- variance is negligible

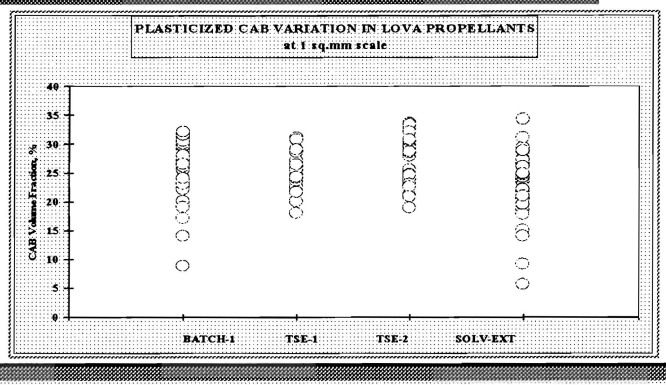


Lower degree of mixing
- variance is large

STATISTICAL PARAMETERS OF "DEGREE OF MIXING"

mean	$\bar{c} = \frac{1}{N}\sum_{i=1}^{N} c_i$
variance	$s^2 = \frac{1}{(N-1)}\sum_{i=1}^{N}\left(c_i - \bar{c}\right)^2$
coefficient of variation	$CV = S / \bar{C}$
mixing index	$MI = 1 - S / S_0$
maximum variance	$s_0^2 = \bar{c}\left(1 - \bar{c}\right)$
intensity of mixing	$I_{mix} = 1 - S^2 / S_0^2$
intensity of segregation	$I_{seg} = S^2 / S_0^2$
segregation index	$SI = S / S_0$

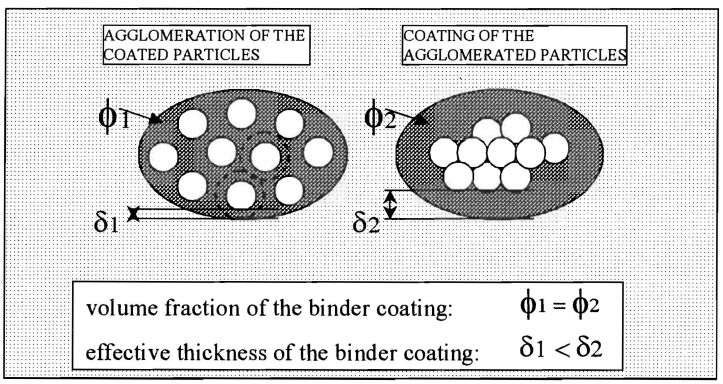
Degree of mixing parameters of LOVA propellants.



LOVA plasticized CAB statistical parameters at 1 sq.mm scale

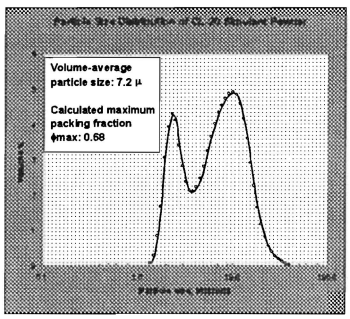
Volume Fraction Parameters	Batch 1	multi-feed TSE-1	multi feed TSE-2	fully solvate TSE-3
mean	24.7	25.5	27.2	22.6
standard deviation	6.32	3.44	4.19	5.80
coefficient of variation	.26	.13	.15	.26
mixing index	.85	.92	.90	.86

Quantitative determination of coating quality

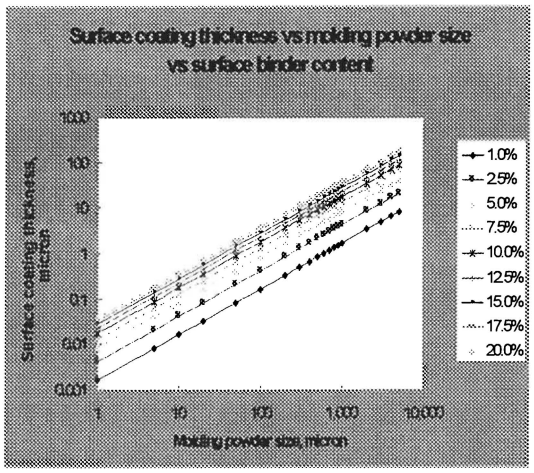


Microstructural features that are measured to evaluate the goodness or "quality" of coating in molding powders.

Quantitative determination of coating quality



Particle size distribution of the simulant powder



Results of the coat-a-powder calculations for a typical molding powder system

On-Line X-ray Analysis of Twin-Screw Extruded Formulations

STEVENS PROCESSING LAB

- concentration distributions of the ingredients
- the degree of mixedness as a function of the scale of examination and location
- the particle size, orientation and
- defect density distributions
- coating thickness of the particles

CONCLUSIONS:

•These experiments demonstrated the importance of quantitatively determining the concentration distributions of the ingredients of the formulation, the degree of mixedness as a function of the scale of examination and location, the particle size, shape, orientation and defect density distributions and the coating thickness of the particles.

•Such measurements enable a detailed understanding of the effects of processing parameters and product quality.

•The comprehensive x-ray techniques developed at Stevens proved to be powerful tools for quantitative analysis of energetics and other filled materials; and amenable for on-line applications at production facilities.

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Naval Surface Warfare Center
INDIAN HEAD DIVISION

10th JOCG CONTINUOUS MIXER & EXTRUDER USERS GROUP MEETING

Continuous Processing On-Line Quality Control Using Near Infrared (NIR) Spectroscopy

Robert Thompson & Mark Michienzi
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue, Building 695, Code 9420M
Indian Head, Maryland 20640-5035

Naval Sea Systems Command

NSWC
Naval Surface Warfare Center
INDIAN HEAD DIVISION

Objective

Develop a real-time, on-line technique to determine ballistic and physical properties while continuously manufacturing extruded composite propellant using a 40 mm W&P twin screw extruder

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Approach

- Measured NIR spectra of feedstreams
- Investigate depth of penetration & probe fouling
- Develop models using batch processed propellant then refine models using TSE processed propellant

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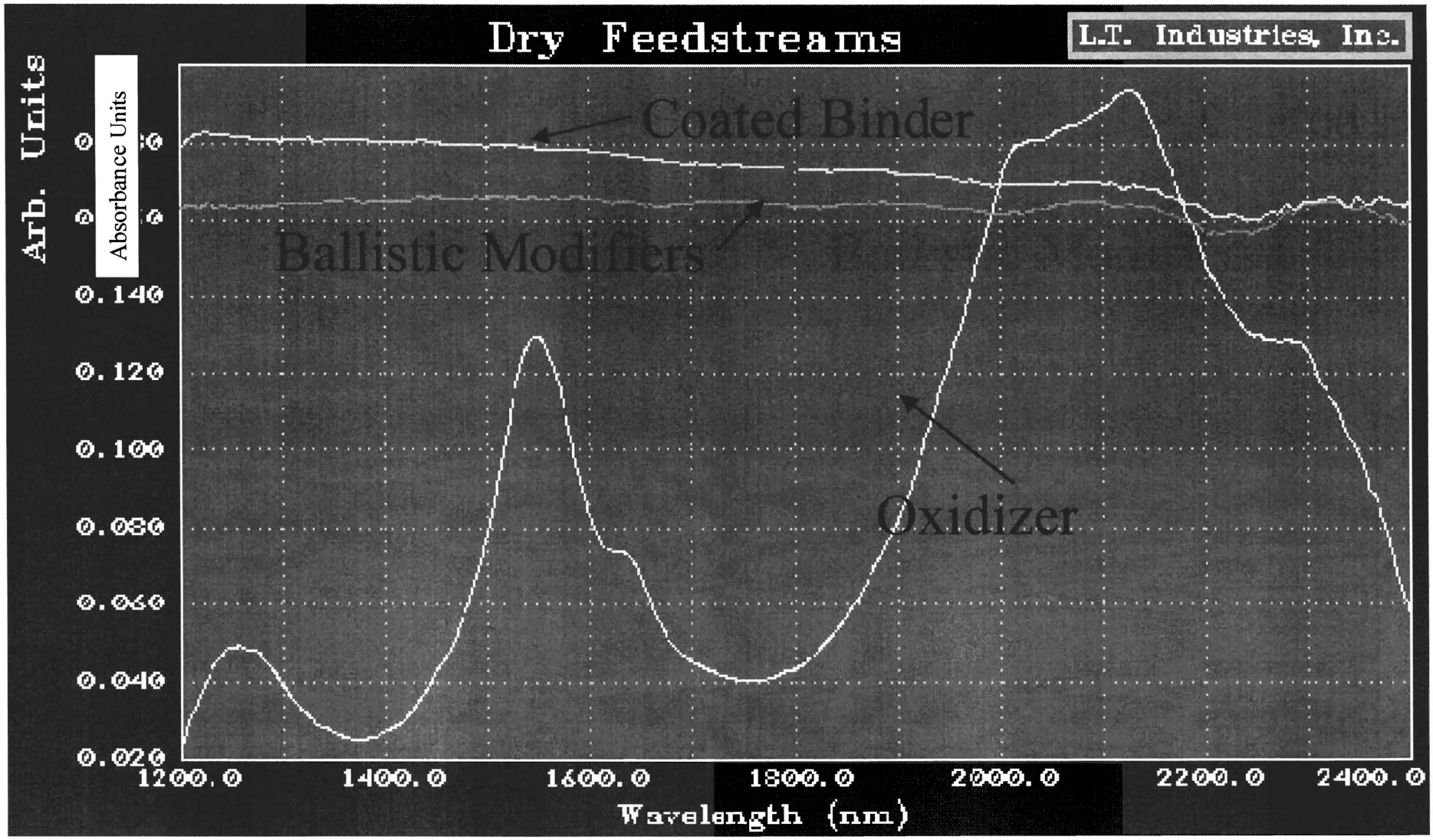
Dry Feedstream Spectra



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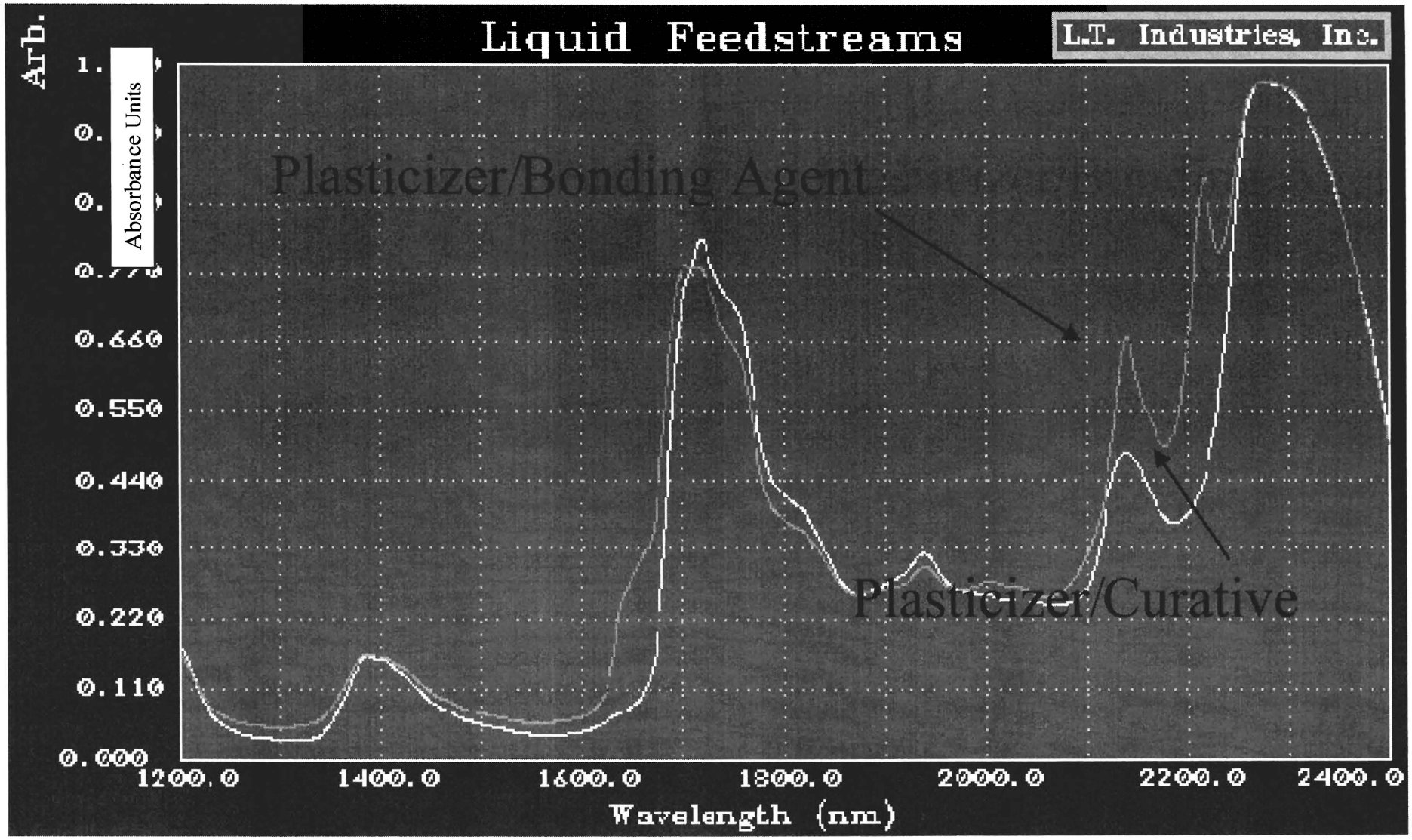
Liquid Feedstream Spectra



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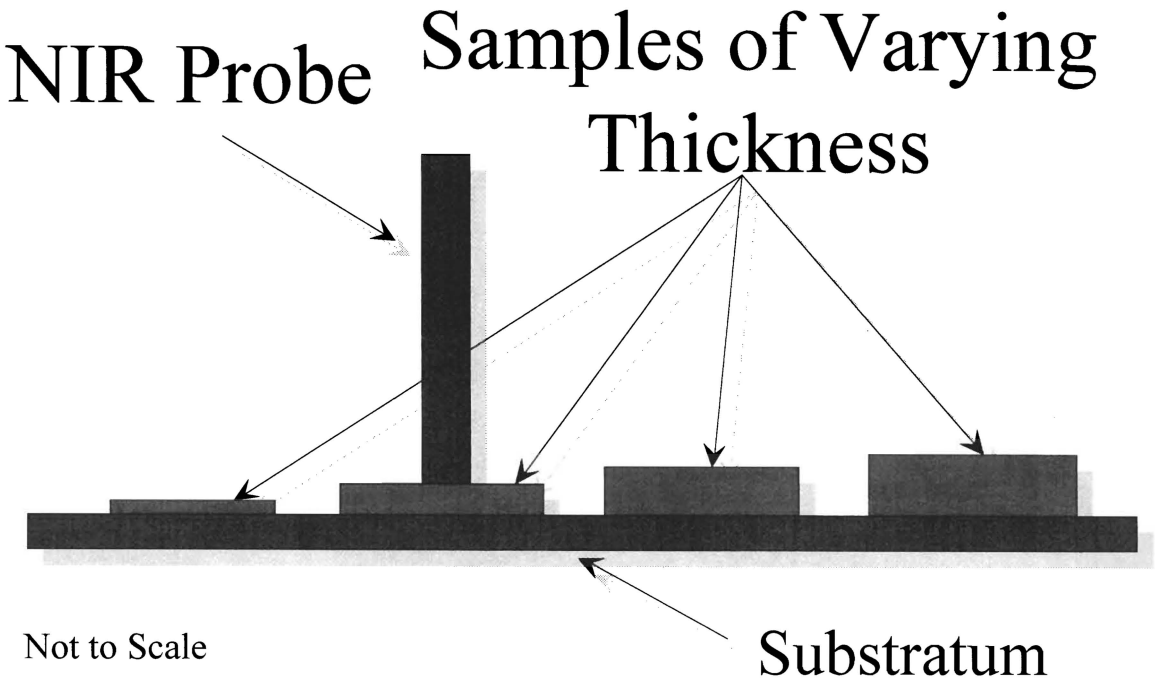
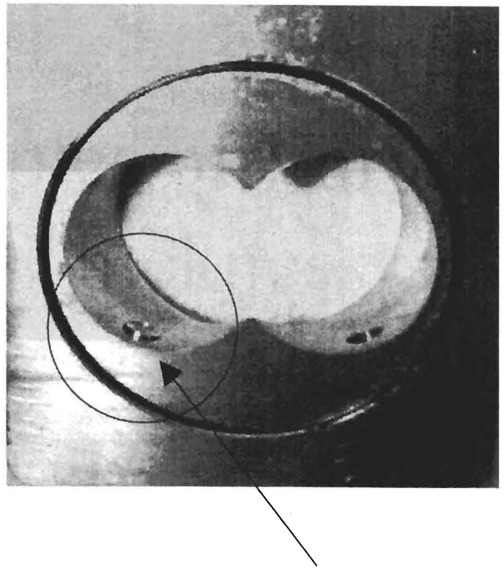
Depth of Penetration

Test Setup



MFU w/ NIR Probe Installed



NIR probe extends into propellant stream 2 mm

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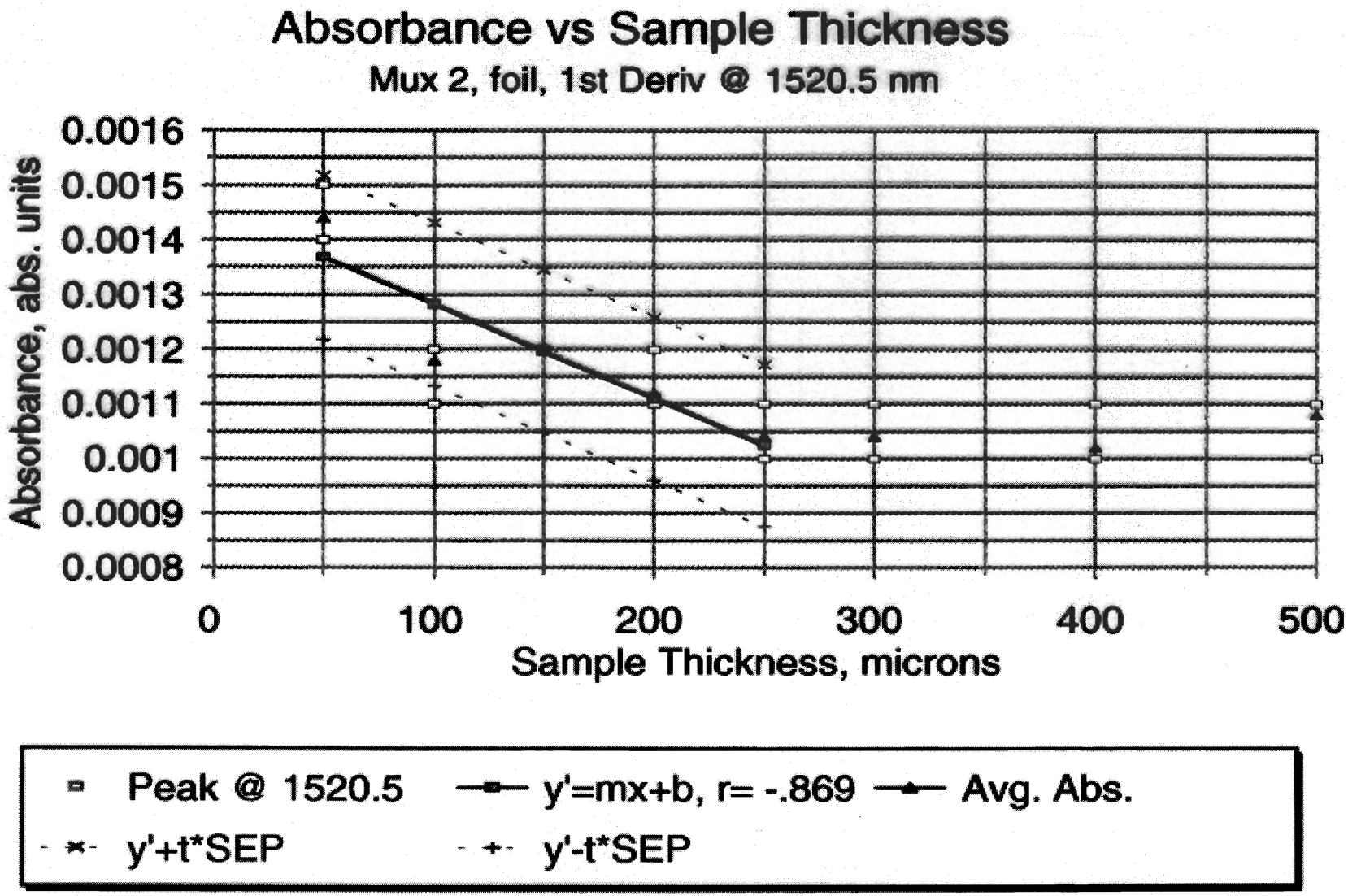
Depth of Penetration



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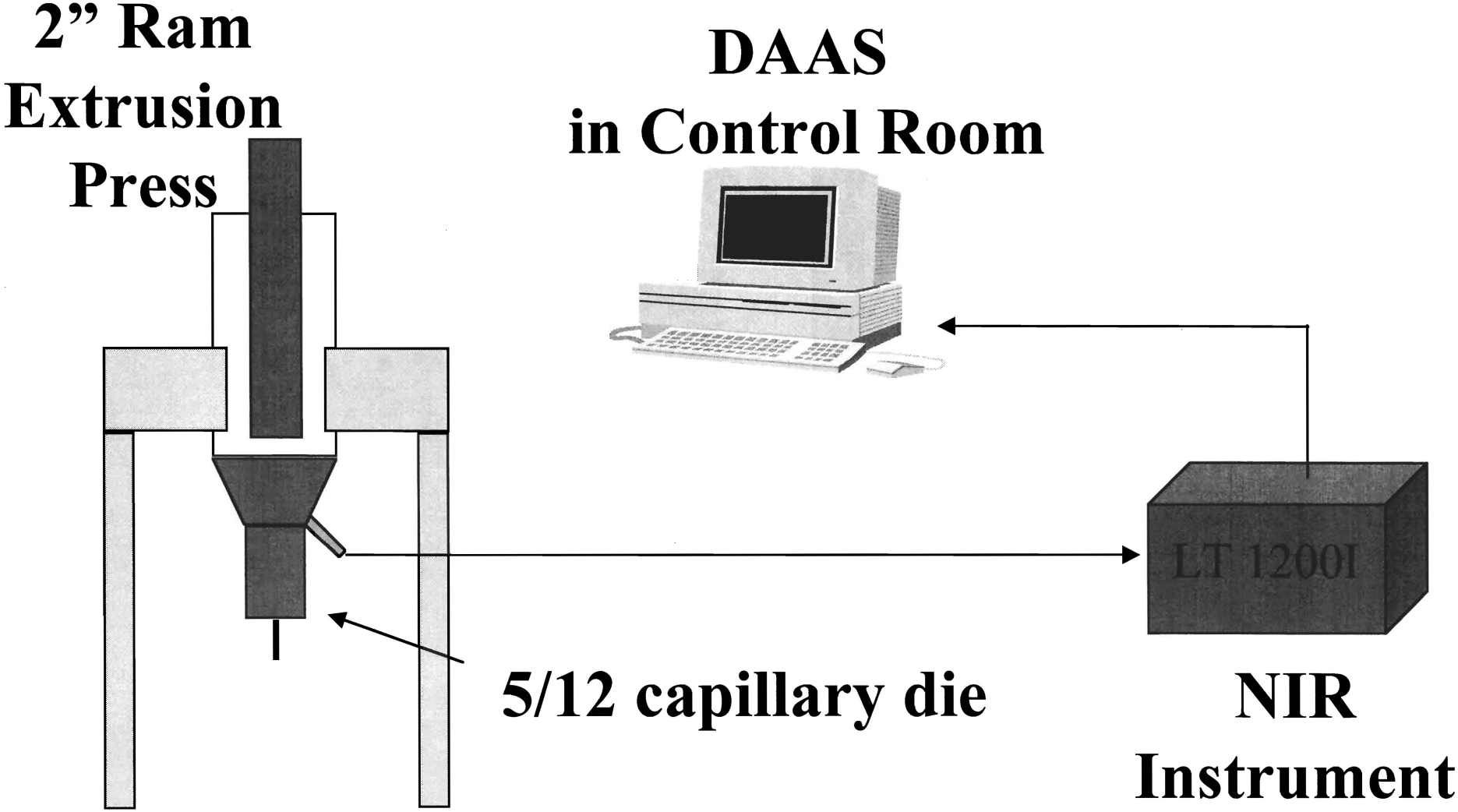
2” Ram Press Setup



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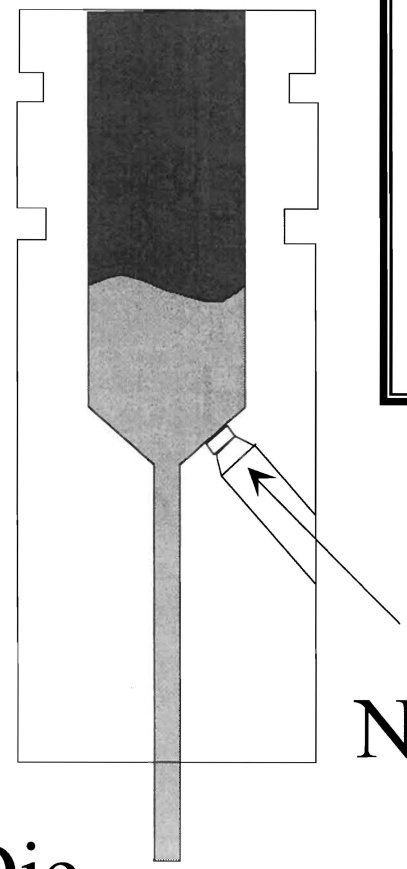
Sensitivity & Fouling

Low Solids
(83%) Propellant

High Solids
(89%) Propellant



NIR Probe

5/12 Capillary Die

Test Setup

Can the probe detect ingredient variations? Does the probe lens foul during extrusion?

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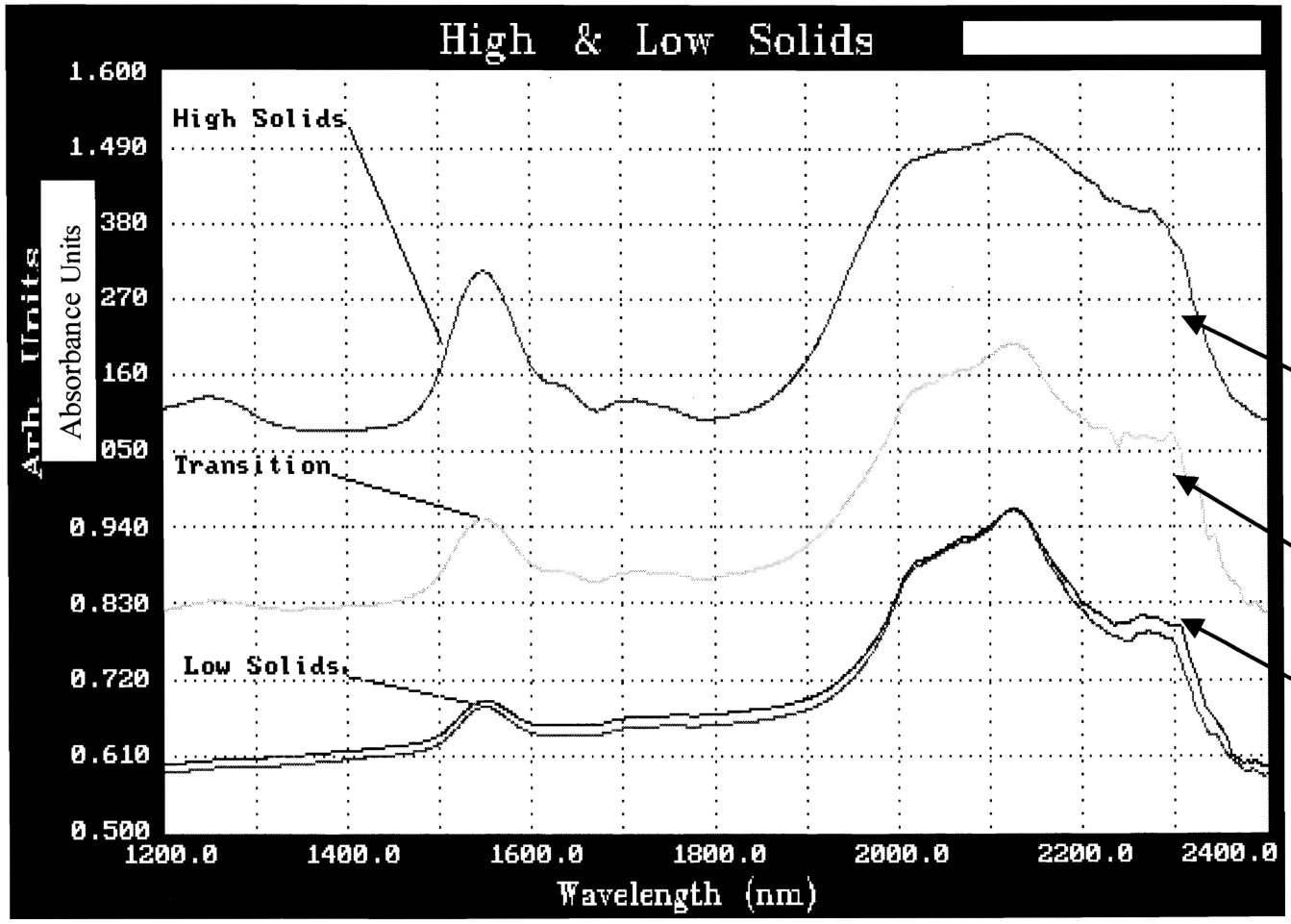
Sensitivity & Fouling



High Solids (89%) Loading

Transition to Low Solids Loading

Low Solids (83%) Loading

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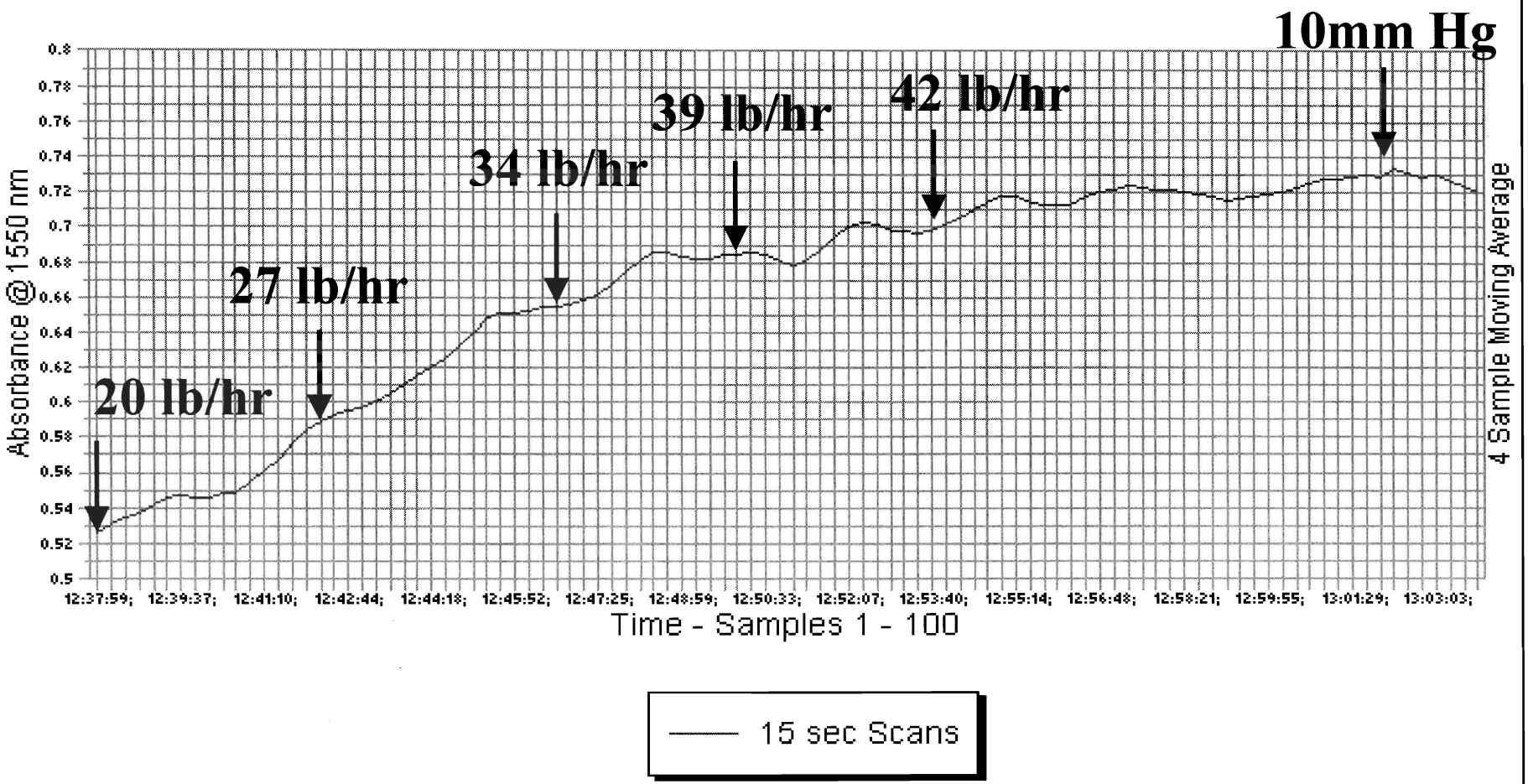
TSE Ramp Up

Ramp Up to Steady State Processing



Batch Work

- Used MODDE (MODeling & DEsign) software to design test matrix
- Made 20 batch mixes
  - ∗ Varied feedstream percentages around baseline
  - ∗ Extruded propellant and collected spectra
  - ∗ Measured density, burn rate, and HoE
  - ∗ Developed models for each property using PLS and PCR
- Predicted properties of two validation lots

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Batch Models

Batch models predicting batch propellant

Density (g/ml) Model

	PCR/1st derivative data/Kubleka-Monk correction			
Validation Lot	Measured	Predicted	Delta	%
1	1.7537	1.7538	-0.0001	-0.01
2	1.7434	1.7436	-0.0002	-0.01

Burn Rate (in/sec) Model

	PCR/log (1/T) data			
Validation Lot	Measured	Predicted	Delta	%
1	0.594	0.611	-0.017	-2.86
2	0.535	0.563	-0.028	-5.23

HoE (cal/g) Model

	PCR/log (1/T) data			
Validation Lot	Measured	Predicted	Delta	%
1	1400	1353	47	3.36
2	1275	1281	-6	-0.47

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TSE Work

- Collected NIR spectra from 4 live TSE runs
  - ∗ Varied screw design and screw speed - DoE
- Measured density, burn rate, and HoE
- Refined models by incorporating TSE data

Validation Run

- Final run to validate results of the DoE
- 11 Grains produced
  - Three fired in heavy weight hardware
  - Several others dissected for analysis
- Density, HoE, and burn rate measured
- Models used to predict ballistic and physical properties

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INDIAN HEAD DIVISION

TSE Processed Propellant

- **Difference between measured and predicted propellant properties using revised model**

  ∗All models: Kubleka-Monk corrected 1st derivative absorbance data and PLS correlation

Property	Delta
Density (g/ml)	0.005
Burn Rate (in/sec)	0.019
HoE (cal/g)	58

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Conclusions

- NIR capable of measuring changes in spectral response of extruded composite propellant

- NIR spectra correlate well with density & burn rate
  - ✶ predictive accuracy adequate for process and quality control

- Single wavelength analysis is valid approach for certain types of analysis

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Plans

- Collect spectra during all live runs
  - ✶ Nitramine propellants show great promise
- Feedback to operator DAS
- Closed loop control

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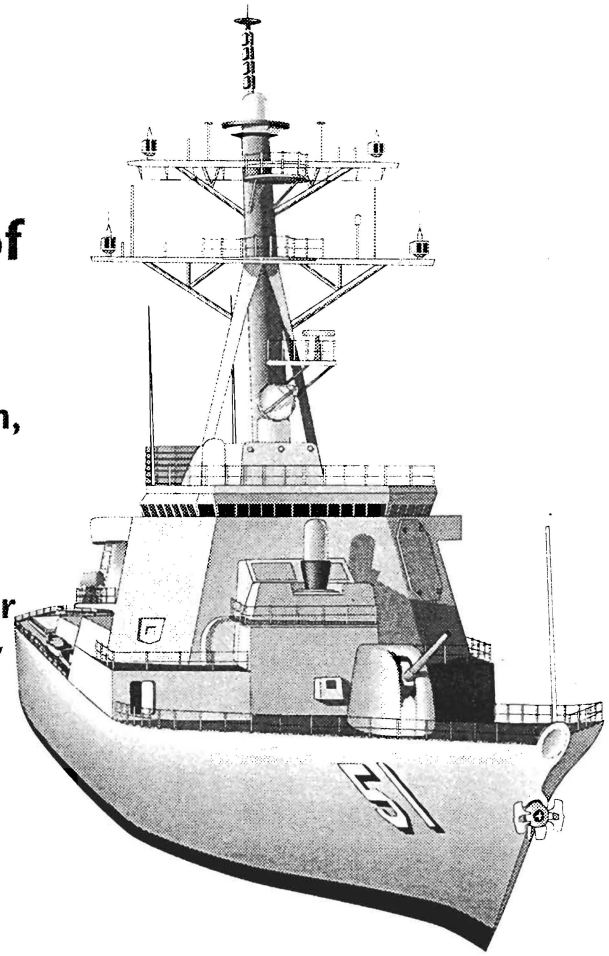
INDIAN HEAD DIVISION

Twin-Screw Processing of GEM Gun Propellant



M. Gallant, W. Newton, S. Johnson,
S. Prickett and C. Murphy
NSWC/Indian Head Division

D. Kalyon, A. Lawal, and S. Railkar
HfMI/Stevens Inst. of Technology

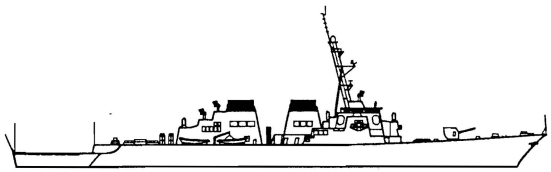
12 May 99

Extruder Users' Group Meeting
Ogden, UT

Objective

Produce 200 lbs. for a Test Firing

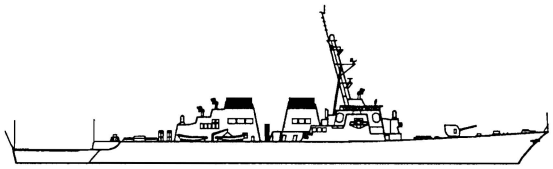
- No solvents
- Ingredients to grains in one facility
- Develop method for continuous processing of TPE propellant
- Rheological Characterization & Die Design
- Demonstrate Recycle and Reuse of TPE Propellant



Approach

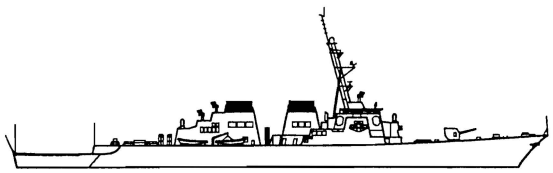
Accomplished the First Five Goals

- Develop Feeding Method for Virgin Polymer
- Melt, Mix & Extrude Quality Strands
- Design Take-Away Equipment & Integrate Bofors Cutter
- Rheological Characterization & Die Design
- Produce 200 lbs. Test Quantity
- Develop Recycling Scheme Including Reduction, Feeding, Melting, & Extruding

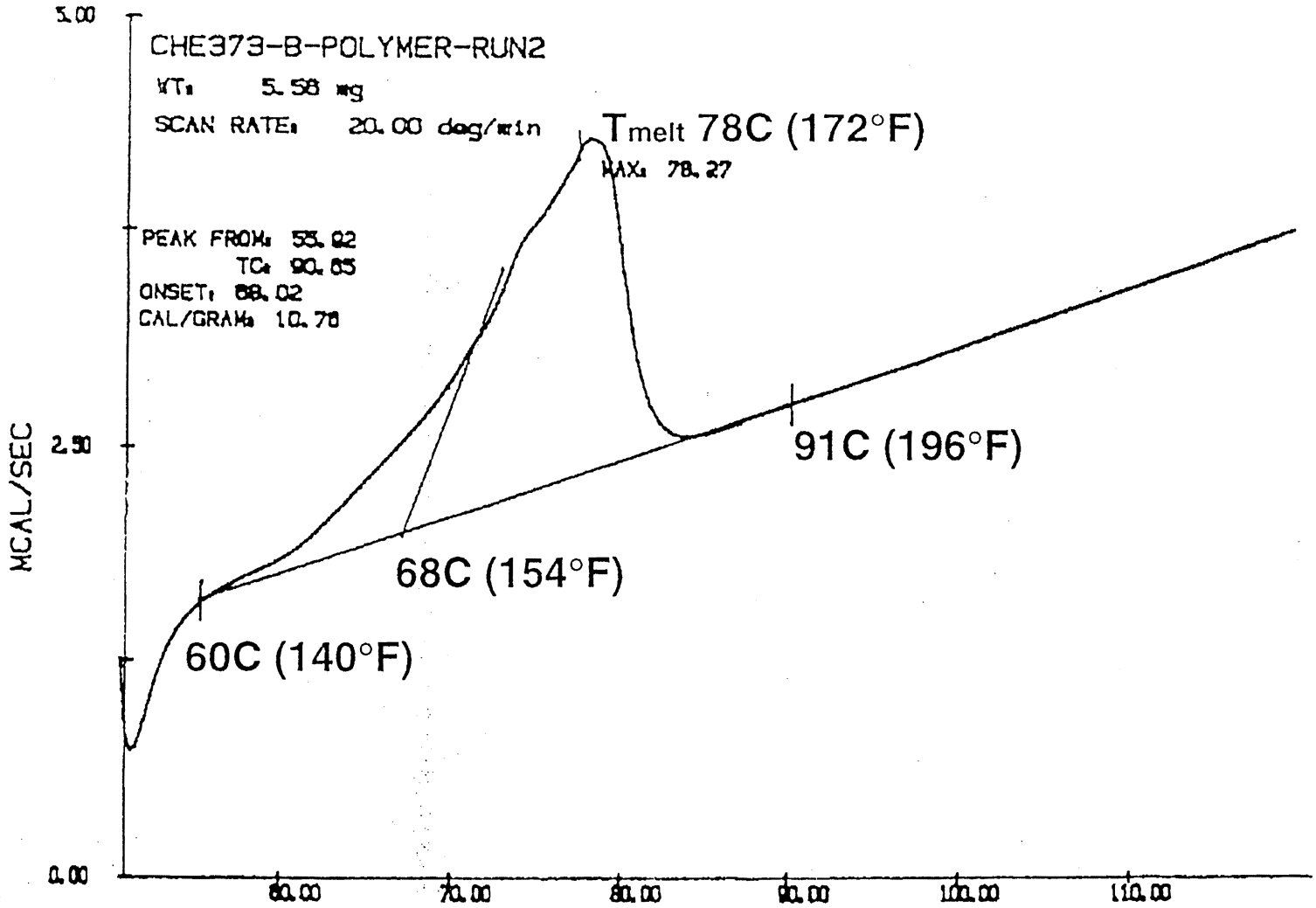


Processing Temperatures

- DSC
- Thiokol
- (show video)



Thermal Analysis of poly(BAMO/AMMO)



Dr. R. Yazici, HIMI

NSWC
Naval Surface Warfare Center
INDIAN HEAD DIVISION

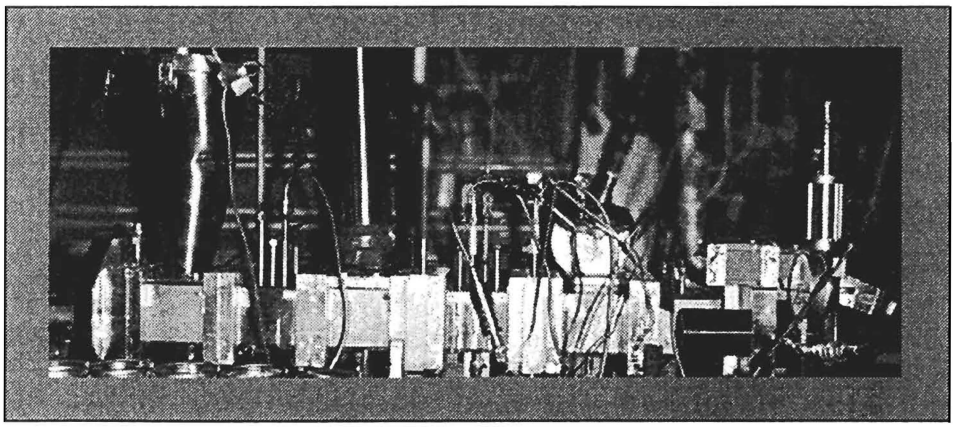
Live Processing Trials

Single Strand Batch LOVA Die

- Conserve Polymer - Run at Very Low Throughput
- Design Screw for Vacuum Processing (Degree of Fill)
- Minimize Viscous Heating in Solids Mixing Section
- Evaluate Effect of Process Parameters on Extrudate Quality
- (show video)

Feed Locations

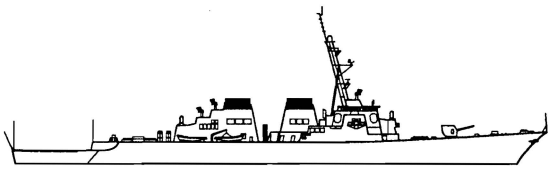
Side View of ZSK Set-up for GEM Gun Propellant



Processing Conditions Screening Study

Four-Fold Objective

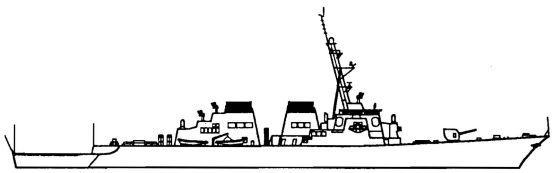
- Screw Design for Vacuum Processing (Low Throughput)
  - Surging: Function of Screw Fill, Geometry, and RPM
  - Screw Fill: Throughput and RPM
- Screw Design to Minimize Viscous Heating (VDH)
  - Function of Screw Fill, Screw Geometry, and Barrel temperature
- Melting & Mixing Efficiencies
  - Temperature, Throughput & RPM Effects
- Extent to Which Conditions Affect Extrudate Qualityuality



Rheology Drives the Train

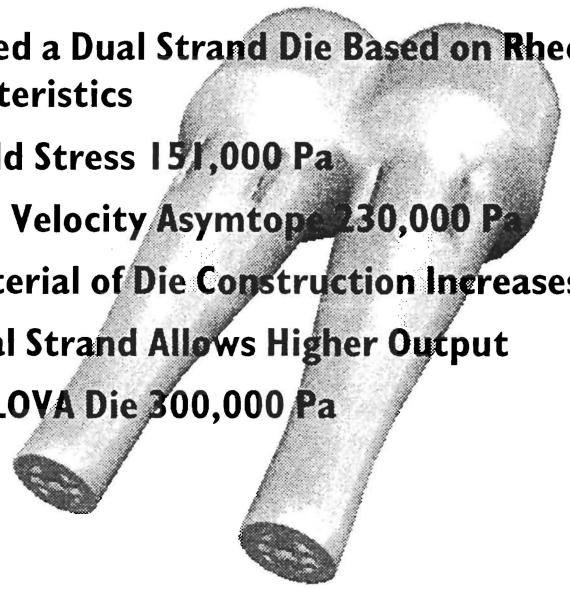
- Modern Energetic Materials Increasingly Non-Newtonian
- Material Generated From First RDX-filled Tests
- NSWC Characterized Rheology Using Capillary Dies (Dr. Prickett)
  - Various Diameters and Lengths
  - 2" Press with Vacuum & Temperature Control

Material from the first live runs



Smart Die Design

Drs. Kalyon, Railkar, & Lawal, HIMI/SIT



- Designed a Dual Strand Die Based on Rheological Characteristics
  - Yield Stress 151,000 Pa
  - Slip Velocity Asymtope 230,000 Pa
  - Material of Die Construction Increases the Ceiling
  - Dual Strand Allows Higher Output
- NSWC LOVA Die 300,000 Pa

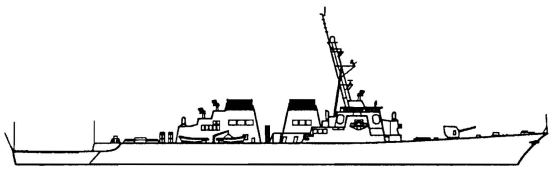
Show vu-graphs of design, modeling and hardware

- (vu-graphs of Modeling)
- (vu-graph of design sketch)
- (vu-graphs of finished design)

Die Implementation

Wrong Assumptions & Poor Conventional Wisdom

- Keep Strand as Cold as Practical: Chilled Water in Take-Away
  - Ensure Best Perforation Formation
  - Best for Cutting
- Mix at Low Barrel Temperatures
- Extrude at Low Die Temperature: Air Cool Extrudate



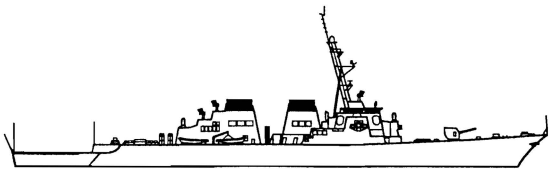
Successful Extrusion

No Die Air-conditioning for Extrudate

Raised Die Temperature (15°F)

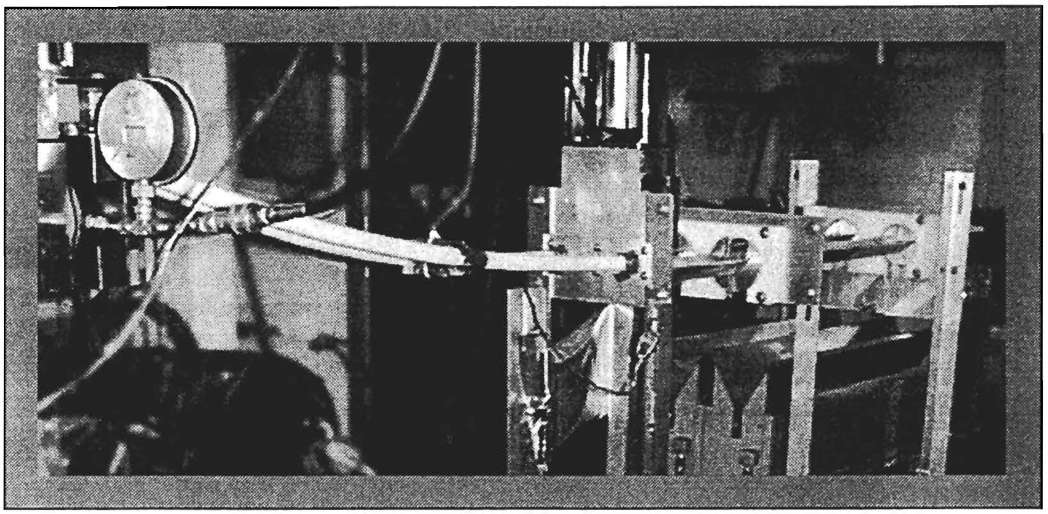
Independent Heat Control for Die Zone

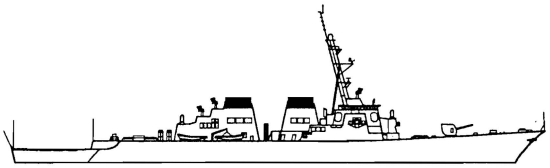
Location of Controlling Thermocouple



Strand Handling x 2

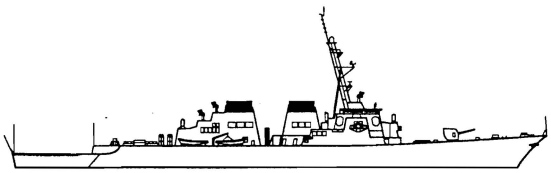
Designed by Bill Newton (father of the Newtomatic)





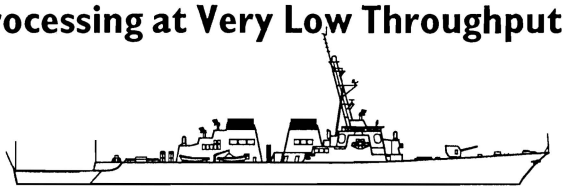
Successful Takeaway

- Initial Cut and Remote Capture
- Warm Water: Issue is Strand Hardness
- Best to Pelletize On-line



Conclusion

- Ingredients to Grains in One Facility
  - Continuous
  - Robust Design
  - On-line Pelletizing Possible
- No Solvents
- Rheological Characterization Critical for Extrusion Success
- Dual Strand Die - Higher Overall Throughput Possible
- Vacuum Processing at Very Low Throughput



New Squeeze Flow Rheometer for the Rheological Characterization of Energetic Materials

Dr. Dilhan M. Kalyon

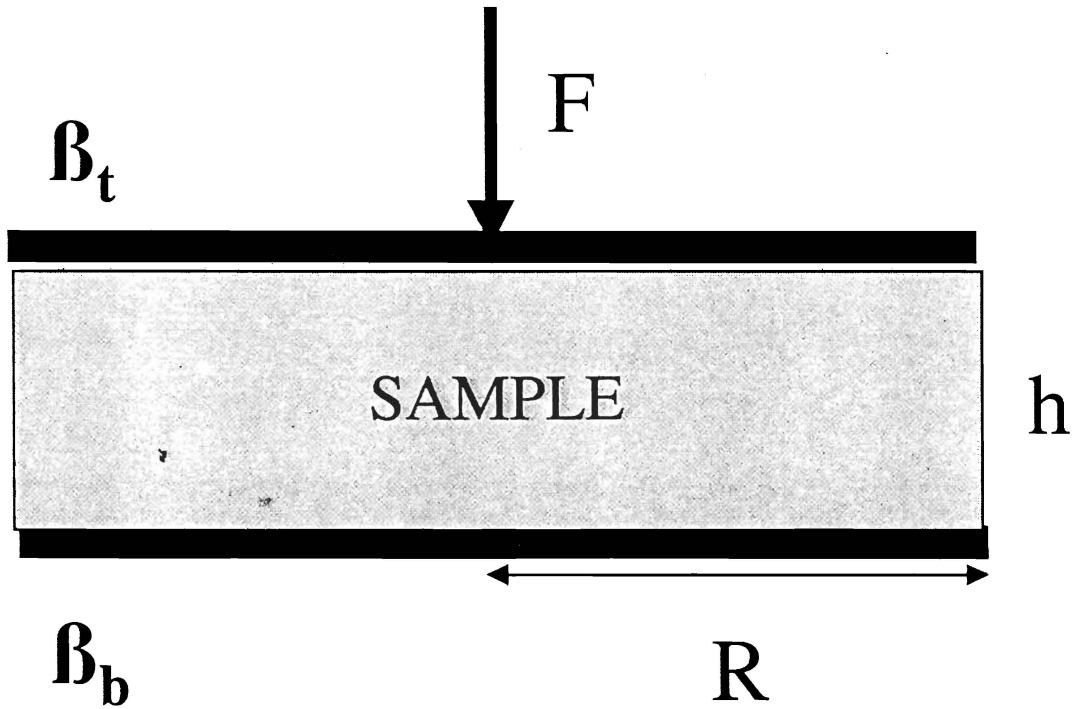
Dr. B. Karuv

Mr. J. Garrow

HfMI, Stevens Institute of Technology

Hoboken, NJ

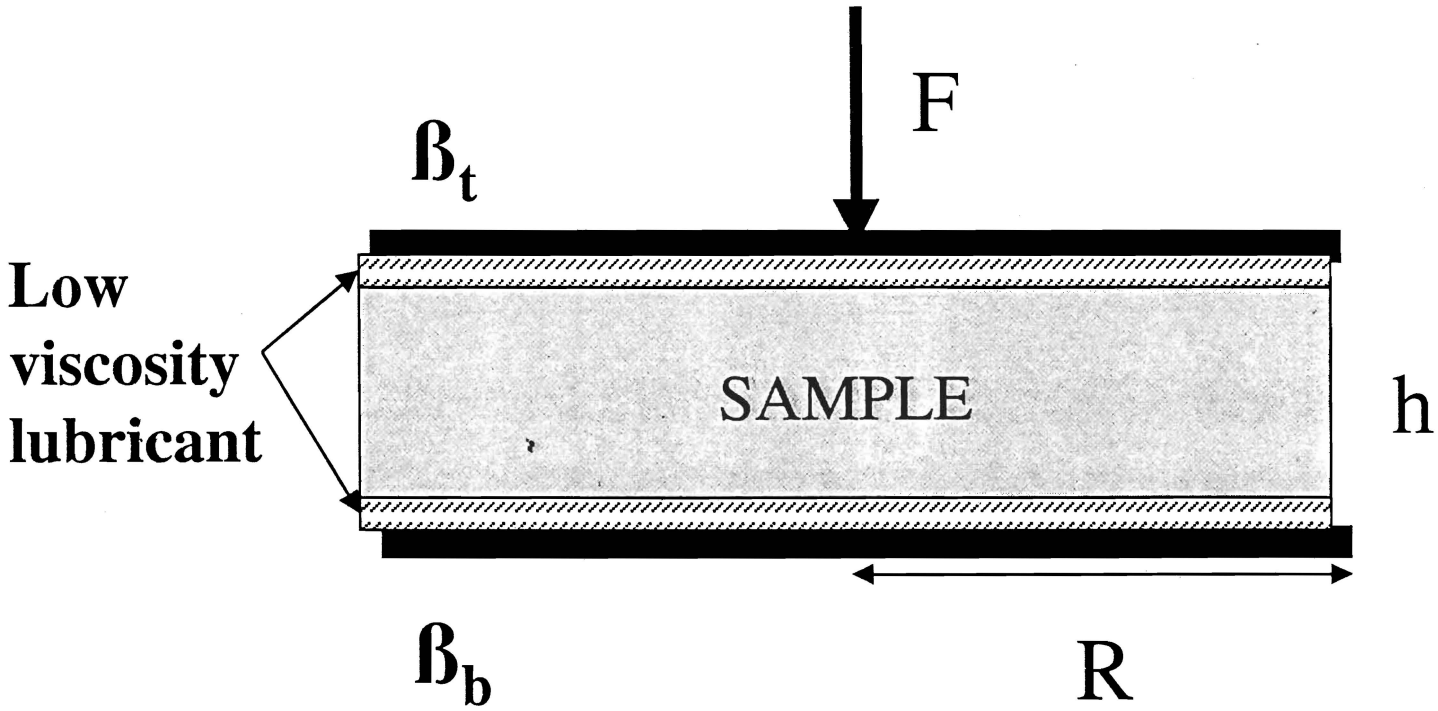
Compressive Squeeze Flow



COMPRESSIVE SQUEEZE FLOW

- Complex deformation field driven by both shear and normal stresses.
- Under small h/R conditions the shear stress dominates over the normal stresses and becomes a shear flow.
- When the disk surfaces are lubricated with a low-viscosity fluid, the normal stresses dominate and becomes biaxial extensional flow

Compressive Squeeze Flow as a Biaxial Extensional Rheometer



$$0 = \frac{1}{r}\frac{\partial r u_r}{\partial r} + \frac{\partial u_z}{\partial z}$$

$$0 = -\frac{\partial p}{\partial r} - \left(\frac{1}{r}\frac{\partial r \tau_{rr}}{\partial r} - \frac{\tau_{\theta\theta}}{r} + \frac{\partial \tau_{rz}}{\partial z}\right)$$

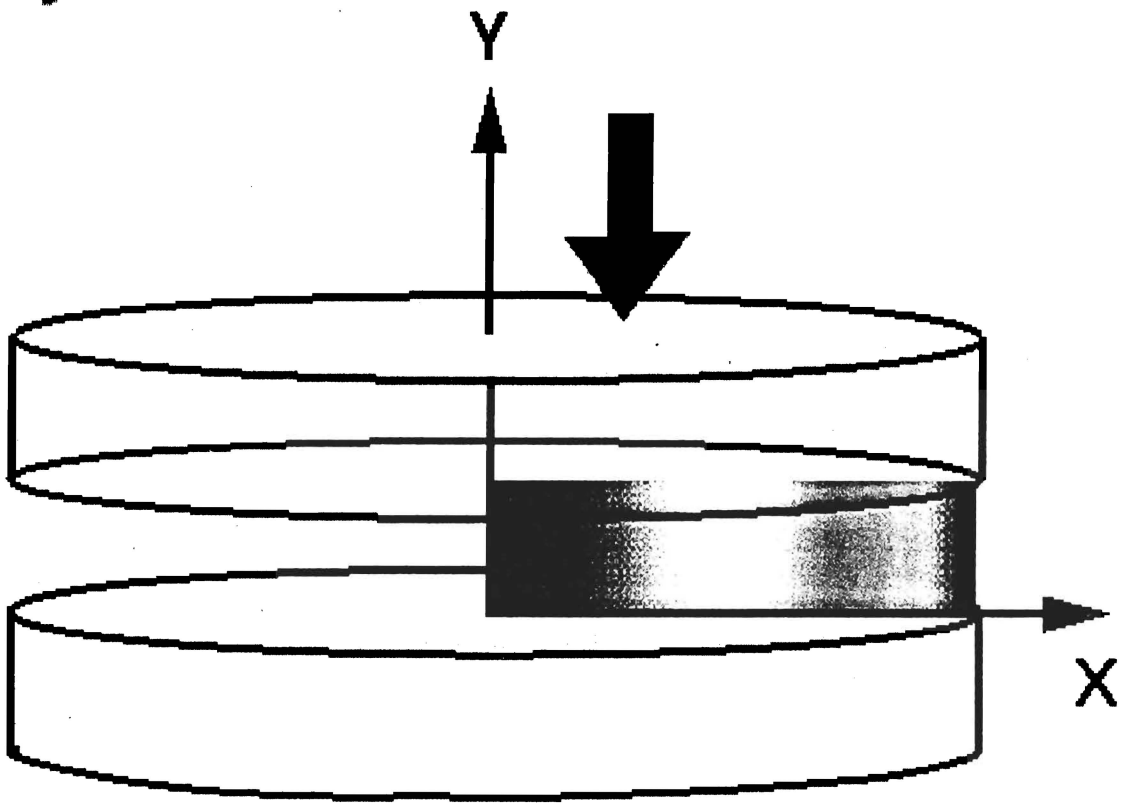
$$0 = -\frac{\partial p}{\partial z} - \left(\frac{1}{r}\frac{\partial r \tau_{rz}}{\partial r} + \frac{\partial \tau_{zz}}{\partial z}\right)$$

Herschel Bulkley Fluid

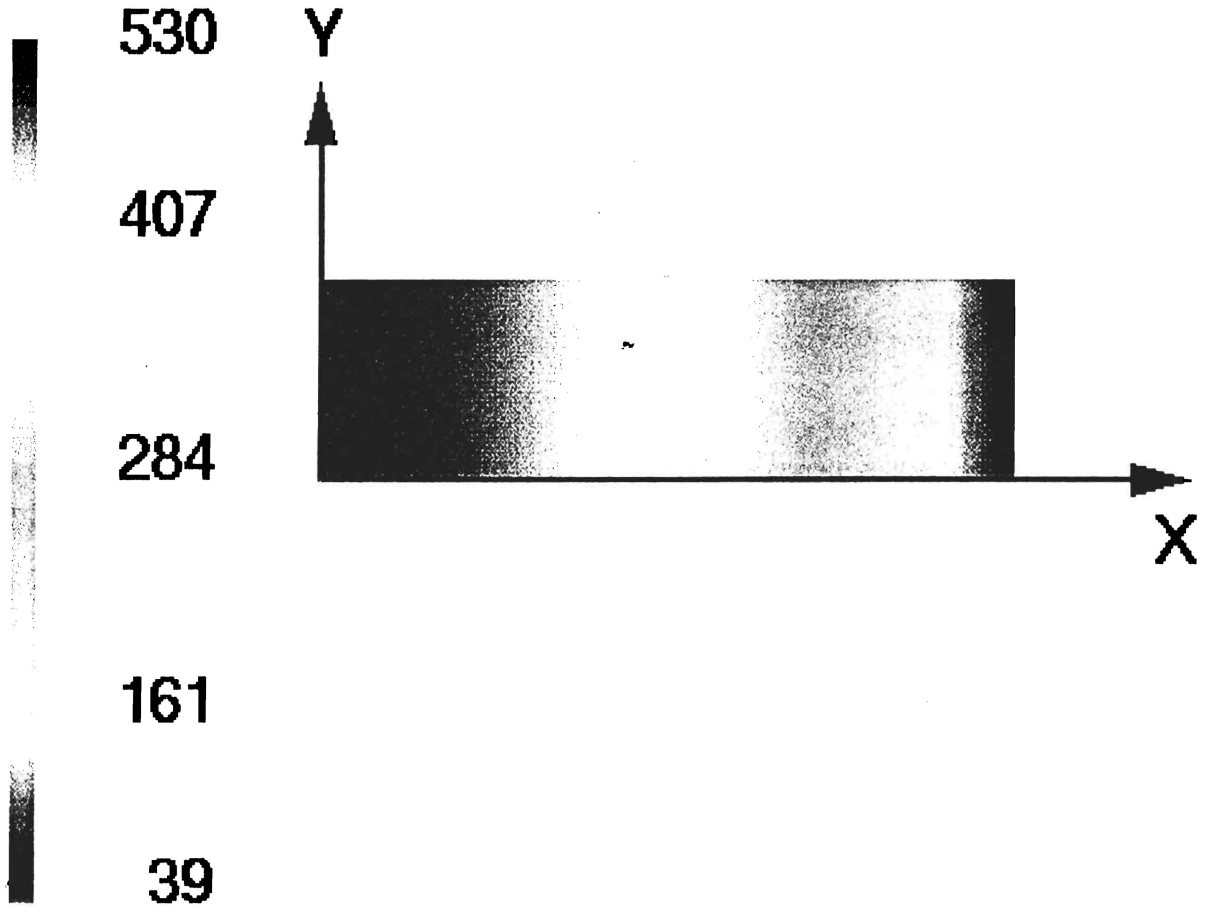
$$\eta = \left[m\left(|\dot{\gamma}|\right)^{n-1} + \frac{\tau_y\left(1 - \exp\left(-\varepsilon|\dot{\gamma}|\right)\right)}{|\dot{\gamma}|} \right]$$

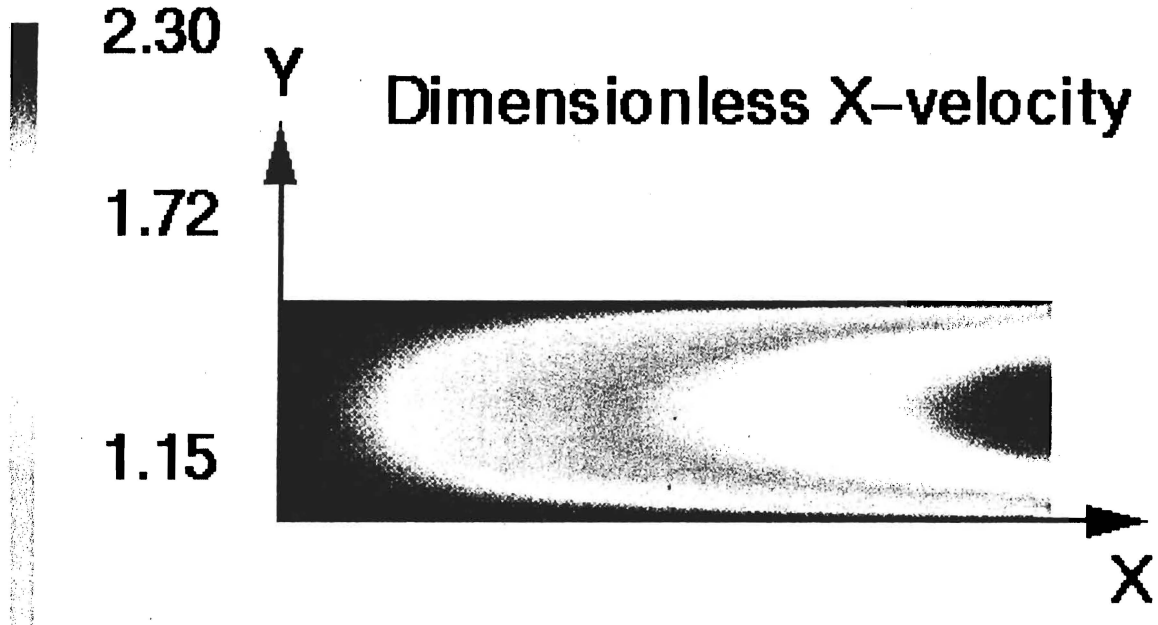
$$|\dot{\gamma}|^2 = 2u_{r,r}^2 + 2\left(\frac{u_r}{r}\right)^2 + 2u_{z,z}^2 + \left(u_{r,z} + u_{z,r}\right)^2$$

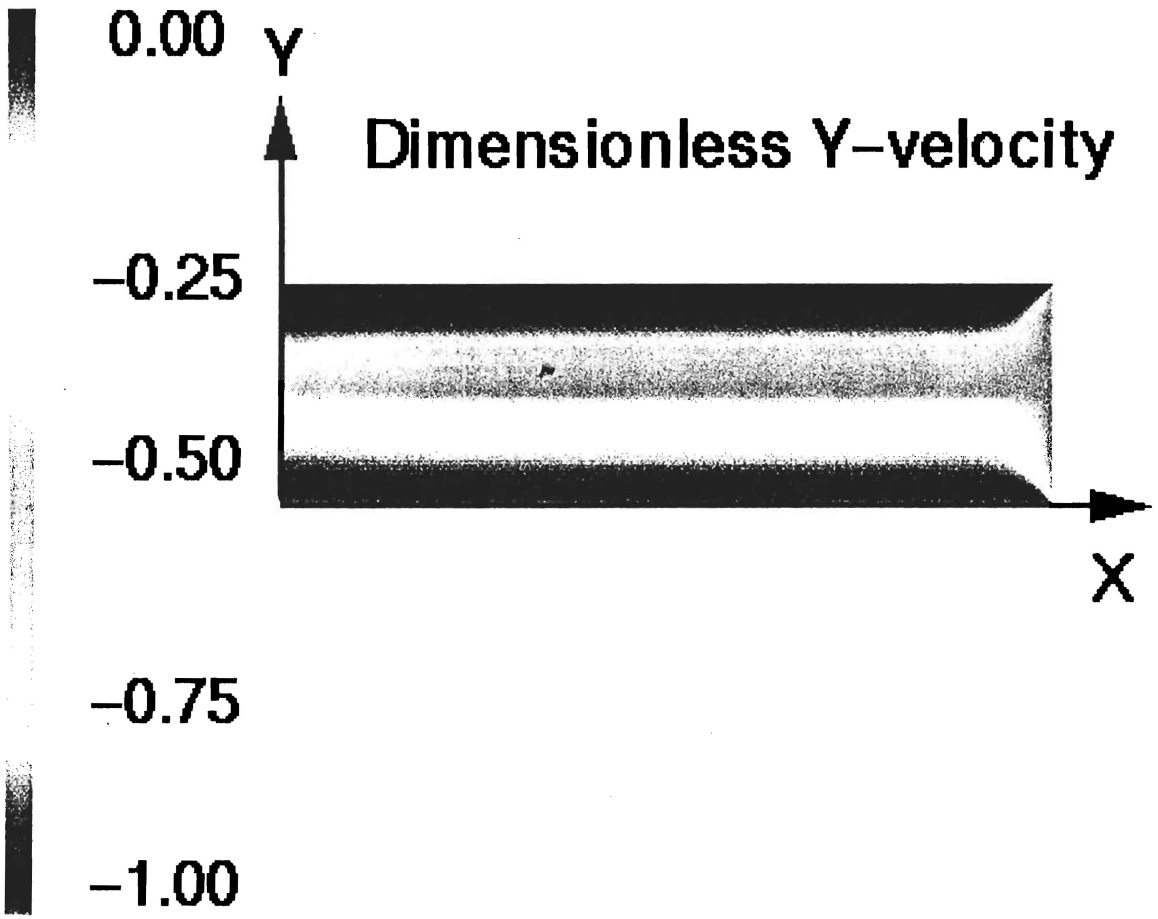
Axisymmetric FEM Model



Dimensionless Pressure

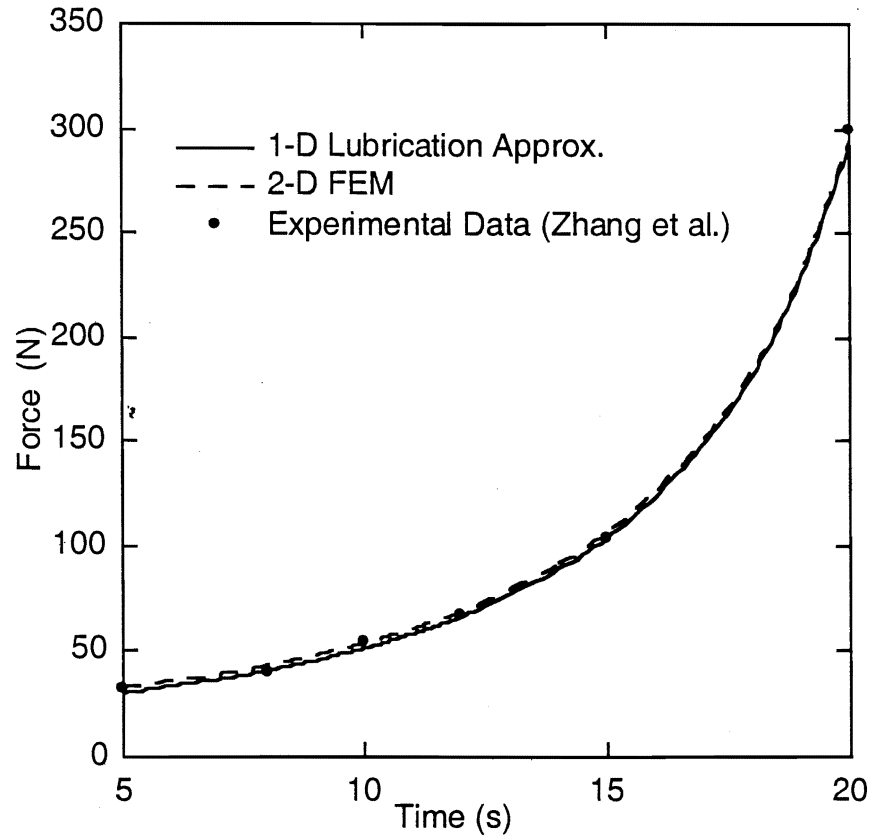



2.30
Y
Dimensionless X–velocity
1.72
1.15
X
0.57
0.00

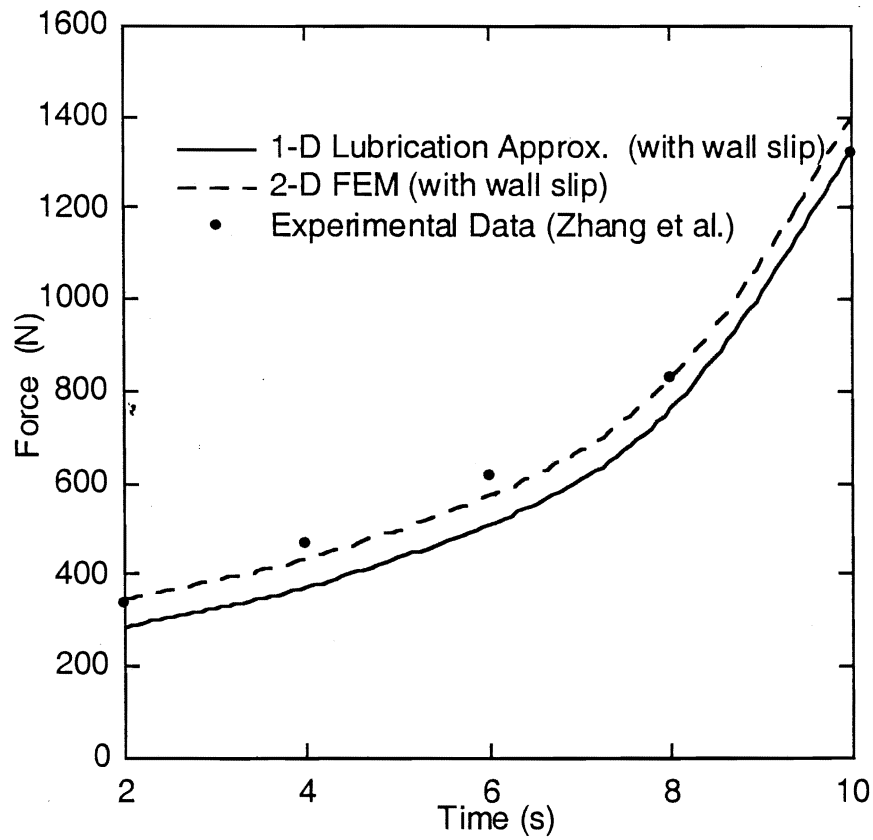

0.00
Y
Dimensionless Y–velocity
–0.25
–0.50
X
–0.75
–1.00

Is it possible to simplify the Equations of Motion to enable analytical methods?

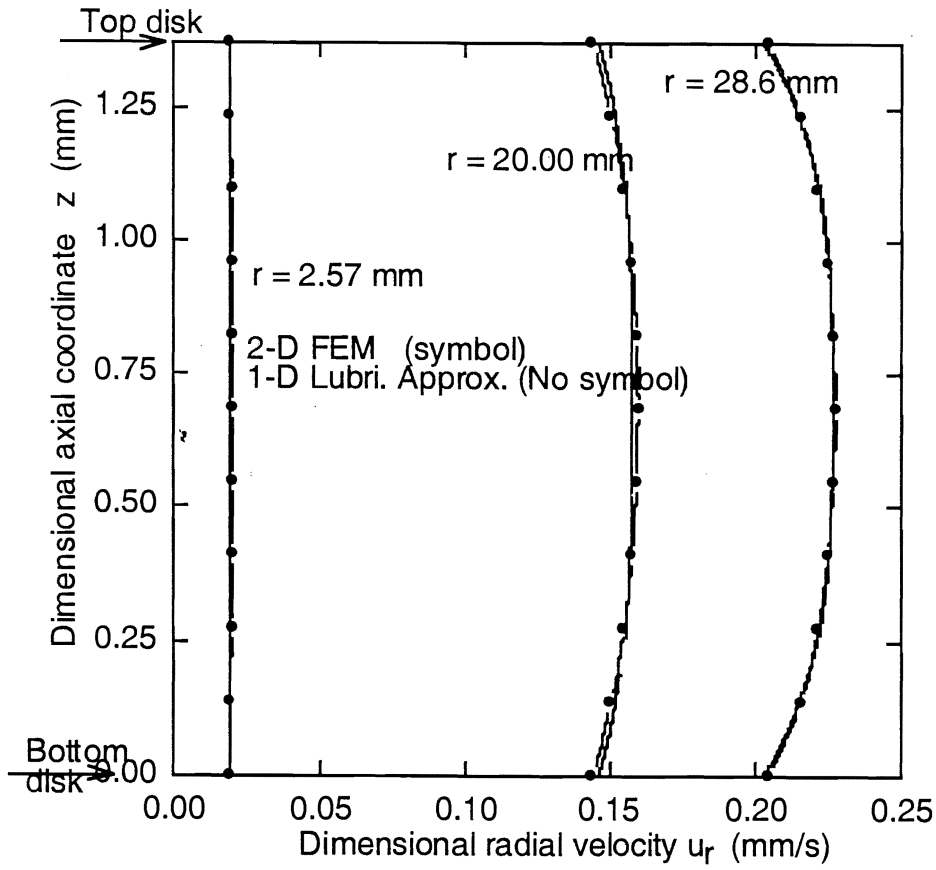
- Allow compressive squeeze flow to be used as a rheometer to characterize the shear viscosity material function.
- The traditional method is to employ the lubrication assumption



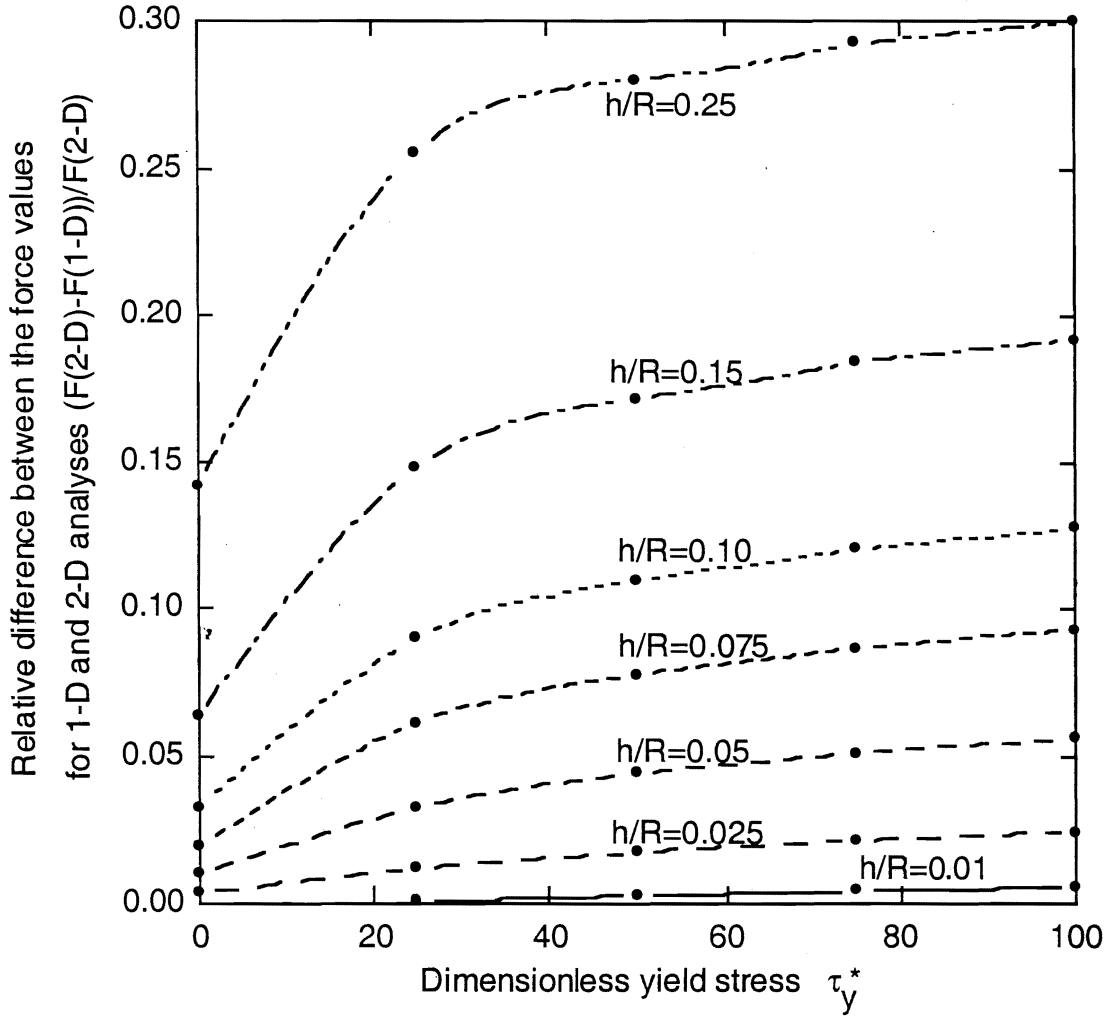
Predictions vs experimental force measurements (Zhang et al., 1995)
LLDPE with m= 5.91×10^3 Pa.s$^{0.6}$ and n=0.6 without wall slip



HDPE DuPont 2005, Zhang et al. 1995 data,
m=44,900 Pa-s $^{0.25}$, n=0.25, ß=5.13E-15m/(Pa $^{2.35}$ s)



Radial velocity predicted by the 1-D and 2-D analyses of a power law fluid with wall slip; n=0.45, ß*=1.0, h/R=0.05.



Force values for the 1-D and 2-D analyses vs. yield stress for Herschel-Bulkley fluids with n=0.45 without wall slip.

Conclusion

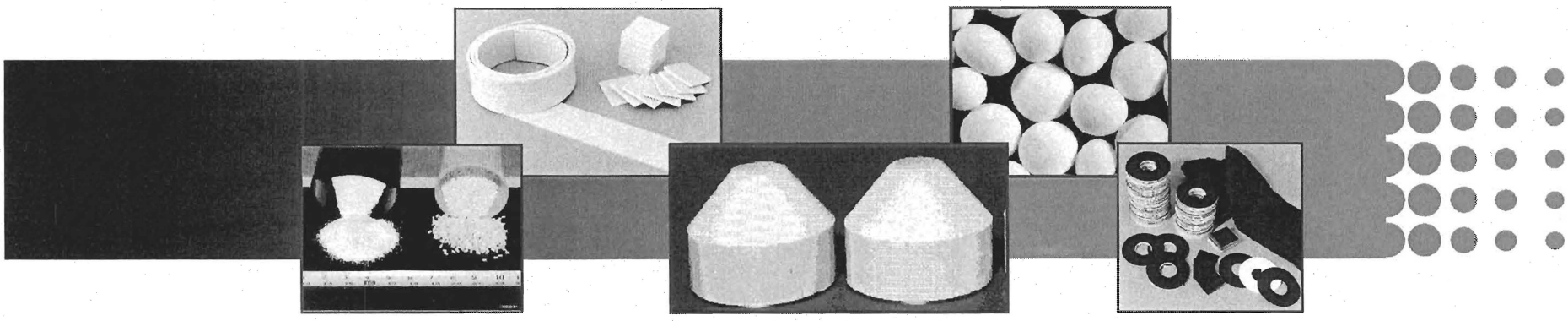
- A new Squeeze Flow rheometer was developed at SIT
- A commercial version of the unit was transferred to Alliant Tech Systems including data acquisition and analysis
- The unit is reliable and Explosion proof
- The squeeze flow rheometer is ideally suited as a product quality/processebility tool.
- The squeeze flow was analyzed by using FEM and by developing an analytical model of the process

10th JOCG Continuous Mixer and Extrusion Conference

Processing of Energetic Materials at Thiokol's 19mm Twin Screw Extrusion Facility

Prepared by:

Andrew Haaland, Quinn Barker, Michael Rose



Thiokol Propulsion™

From Cordant Technologies

May 11-12, 1999

Discussion Topics

- **Overview and Background**
  - Previous Extrusion Processing Efforts at Thiokol
  - Initial 19mm TSE Facility
- **Installation of New Facility at M-241**
  - Feed System Isolation
  - Control System
  - Additional Process Monitoring
- **Processing Results**
  - Process Parameters
  - Extrudate Quality
- **Summary**

Overview & Background

PREVIOUS EXTRUSION PROCESSING EFFORTS AT THIOKOL

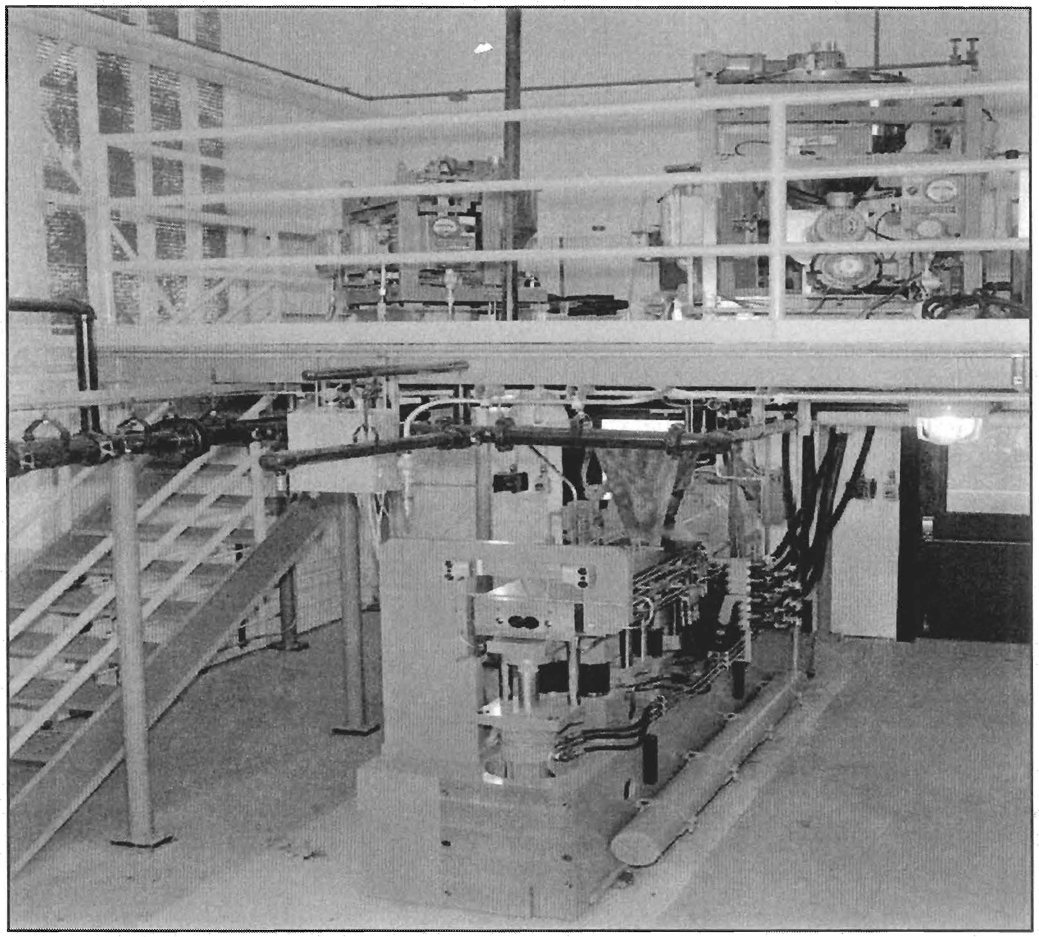
- **Initial twin screw extruder (TSE) installed at Longhorn Army Ammunition Plant (LAAP)**
  - 58mm diameter Werner & Pfleiderer extruder
  - Several liquid and solid feed systems
  - Development of post-processing equipment and techniques
- **Extrusion facility was successfully used to process propellants, explosives, and pyrotechnics**

Material	Ingredients	Quantity, lbs
Rocket Propellant	AP, Al, HTPB, IPDI	500
PAX-4 High Explosive	CAB, HMX , DEGDN, TEGDN	1000
PAX-2A High Explosive	CAB, HMX, BDNPA/F	1000
M39 LOVA Gun Propellant	CAB, Ethyl Centralite, Ethyl Acetate, NC, RDX	200
M43 LOVA Gun Propellant	CAB, Ethyl Centralite, BDNPA/F, NC, RDX	200
TPE LOVA Gun Propellant	LRG-999, RDX, NC	150

- **Facility moved from LAAP to Utah in 1997**

Overview & Background

THIOKOL 58MM TSE LOCATED IN UTAH



- Facility is currently operational processing live materials

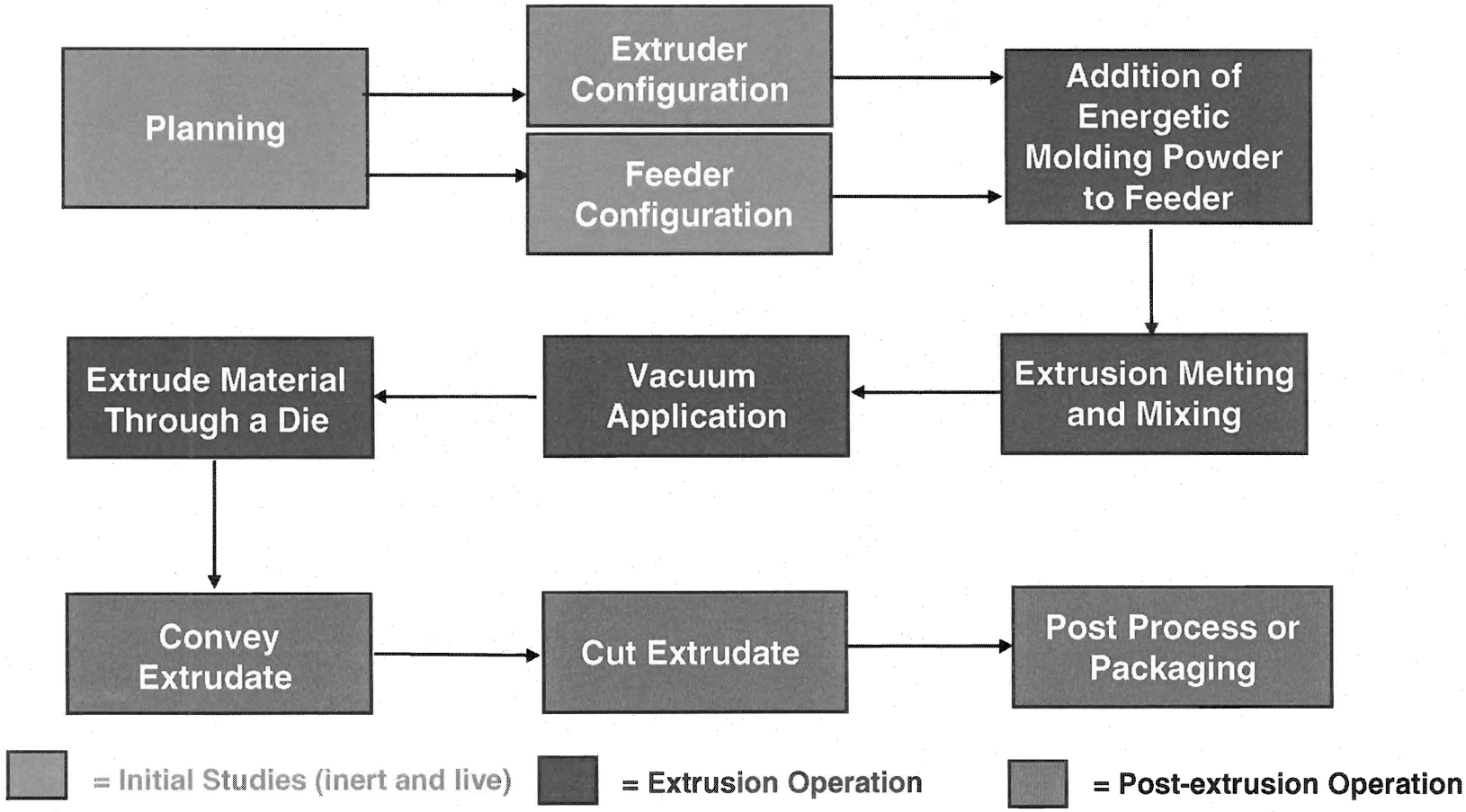
Overview and Background

INITIAL 19MM TSE FACILITY

- **Success at LAAP cultivated a desire to create small-scale TSE facility in Utah**
- **Selected B&P 19mm TSE**
  - 25 l/d, segmented screw, split barrel design
  - 5-10 lb/hour through-put rate
  - Converted from plastics machine to energetic machine
    - Explosion-proof electrical
    - Elimination of metal to metal contact points
    - Remotely located control system
- **Selected Brabender twin screw gravimetric feeder**
- **Successfully processed 280 lb of TPE gun propellant**
- **Thiokol designed product collection equipment**

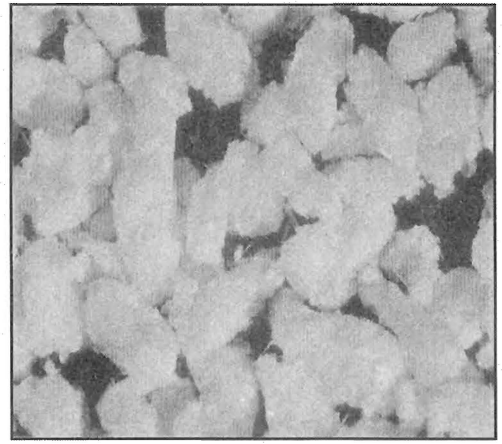
Overview & Background

INITIAL 19MM TSE FACILITY PROCESS FLOW

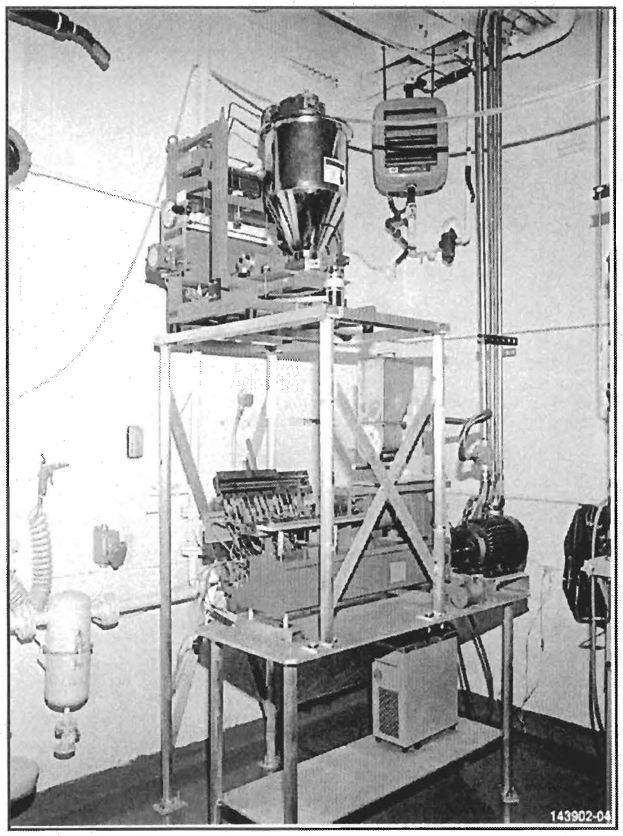


Overview & Background

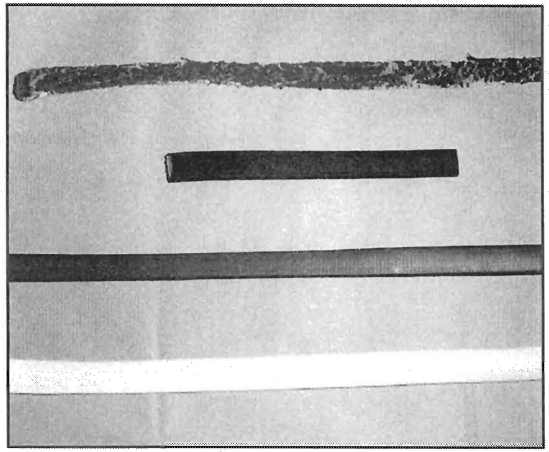
INITIAL 19MM TSE FACILITY



Molding Powder for Twin Screw Extrusion Feedstock



M-56 19mm TSE



Solid Strand Gun Propellant Extrudate

Overview & Background

19MM TSE PROCESSING INCIDENT

- **Incident description**
  - Occurred while processing TPE gun propellant
  - Ignition result of thermal decomposition due to overwork in discharge end of extruder
  - Fire propagated to forward end of extruder, spread up feed chute, and ignited remaining molding powder in feed hopper
- **Damage assessment**
  - Feeder and hopper completely destroyed
  - Sustained minor damage to extruder
  - Significant damage to processing building
- **Provided opportunity to learn and build a better facility**

Overview & Background

19MM TSE INCIDENT

- **Investigation results**
  - Improve data collection and control capability
  - Install better instrumentation on extruder
  - Isolate feeder from extrusion process
  - Isolate process from other areas of the plant
- **Incident was viewed as an opportunity**
  - Initial extrusion efforts were viewed as successful
  - Lessons learned could be applied to construction of a newer, more capable small scale facility
  - Investigation results could be applied to a wide range of energetic materials processes

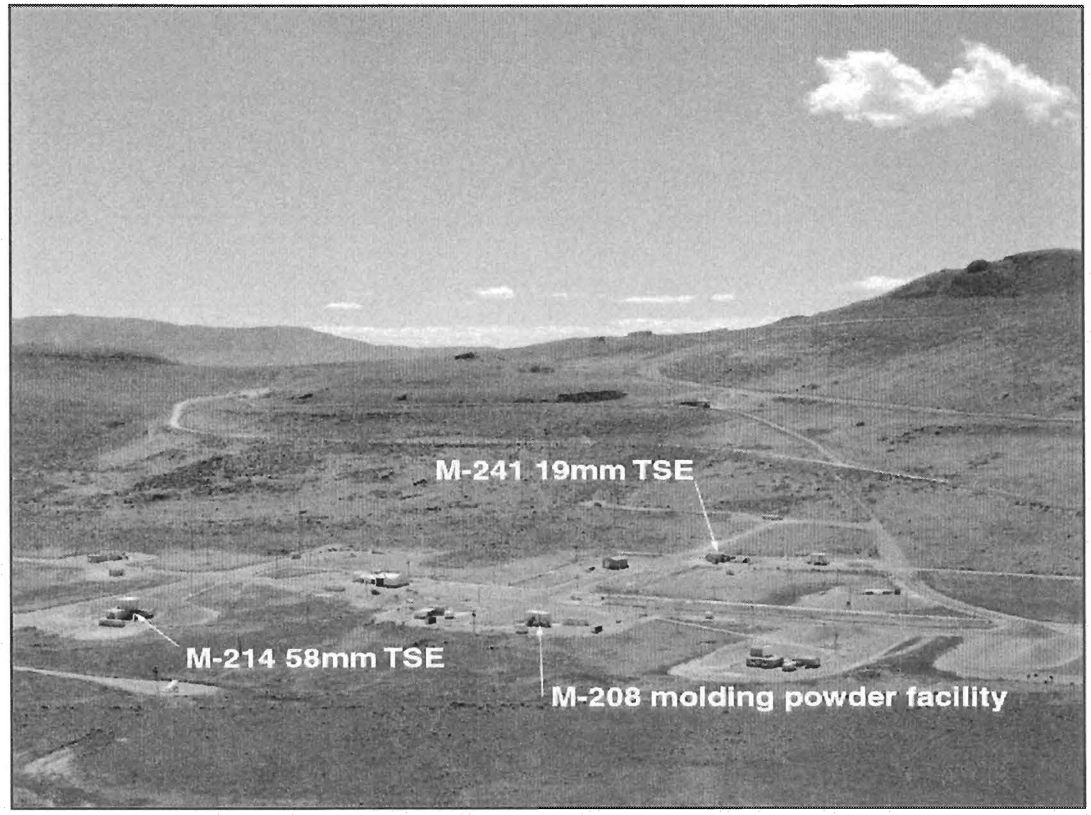
Extrusion Facility Installation

FACILITY DESIGN ACTIVITIES

- **Facility design divided into four major areas**
  - Search for new location
    - More remote location
    - Dedicated, bunker style control room
  - Feed system isolation studies
    - Extruder fire would not propagate to feeder
    - System would allow fire detection and deluge operation
  - New control system design
    - Computer based
    - Real-time trending capability
  - Improved process instrumentation
- **All activities were undertaken in a parallel manner**

Extrusion Facility Installation

FACILITY DESIGN ACTIVITIES: NEW LOCATION

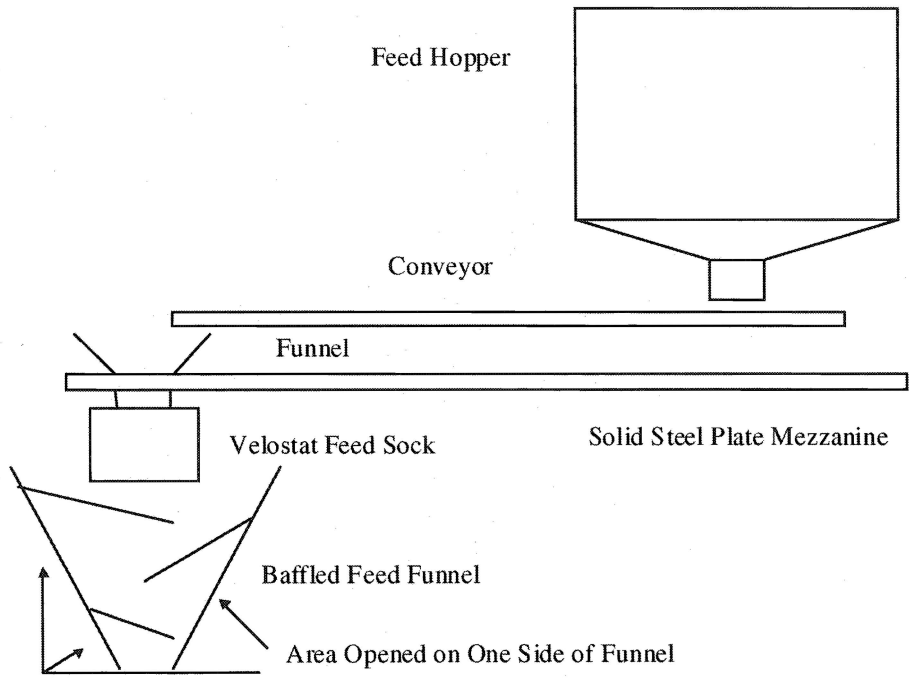


- M-241 selected due to remote location, dedicated control bunker

Extrusion Facility Installation

FEED SYSTEM ISOLATION STUDIES

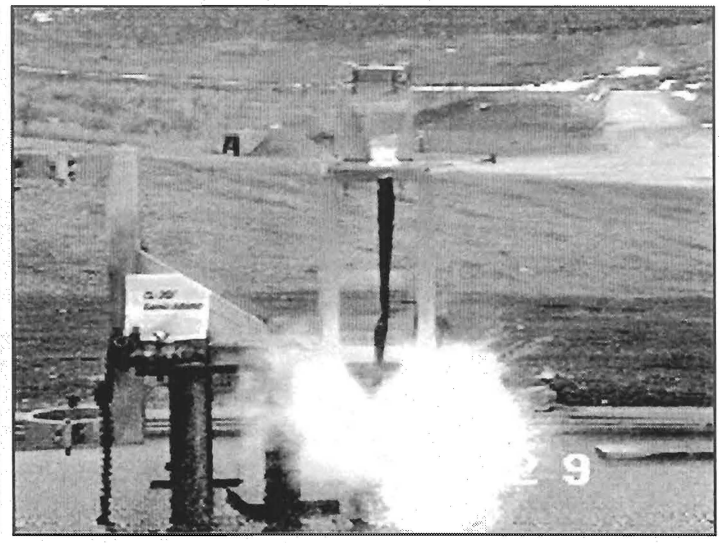
- System design employed a conveyor, solid steel plate mezzanine, Velostat® feed sock, and baffled funnel. Thiokol has a patent pending on this approach



Extrusion Facility Installation

FEED SYSTEM ISOLATION STUDIES

- Complete system was tested using live materials



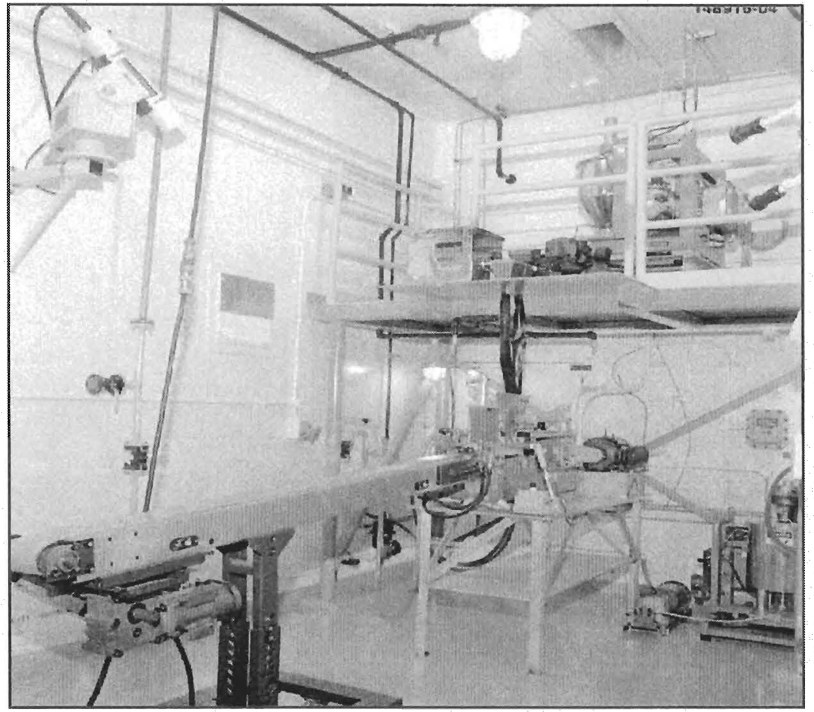
- Fire did not propagate to feed hopper

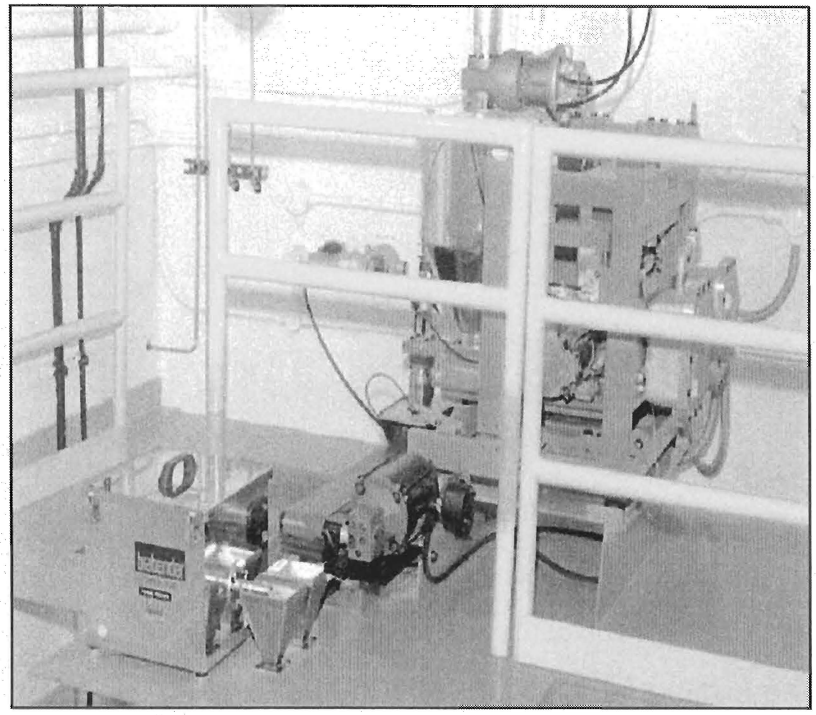
Extrusion Facility Installation

NEW CONTROL SYSTEM AND INSTRUMENTATION

- **Computer-based control and data acquisiton**
  - Allen Bradley hardware and software utilizing data highway
  - RSTrend for data acquisition and display of processing data trends
    - Temperatures and pressure
    - Extruder torque and screw speed
  - RSView for process control
    - Operation of all process equipment
    - Instantaneous display of set points and actual performance
- **Army TIME program installed real-time network for data transfer**
- **Instrumentation was also improved**
  - Added IR thermocouple to independently measure extrudate temperatures
  - Direct coupled torque and rpm instrumentation to drive shaft

Extrusion Facility Installation



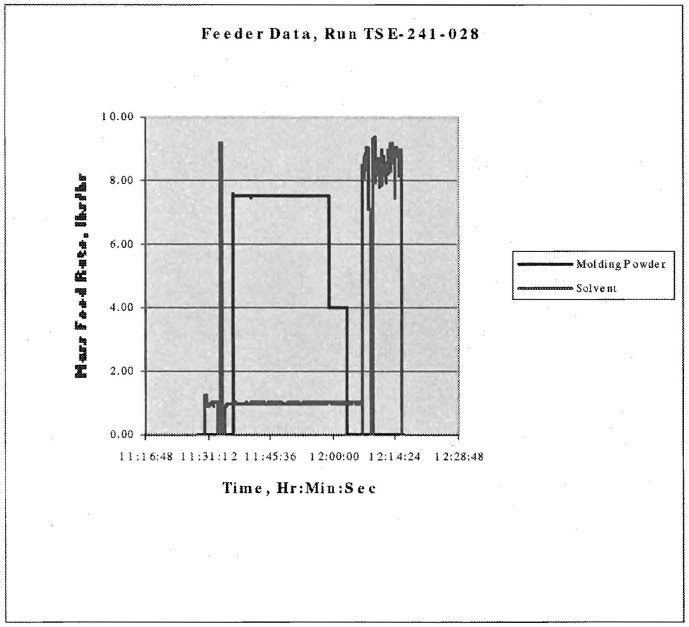


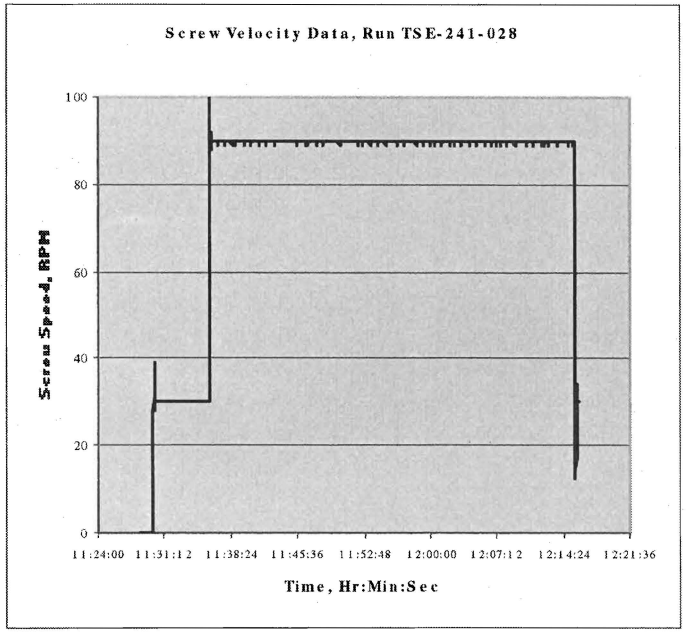
- M-241 facility was completed and operational in March 1998

M-241 Extrusion Facility Processing

PROCESS PARAMETER DATA

- **Facility has been used to complete over 60 runs of energetic material**
  - Longest run time is 64 hours
  - Approximately 1,500 lb of material total has been processed
- **Processing data is analyzed and stored**

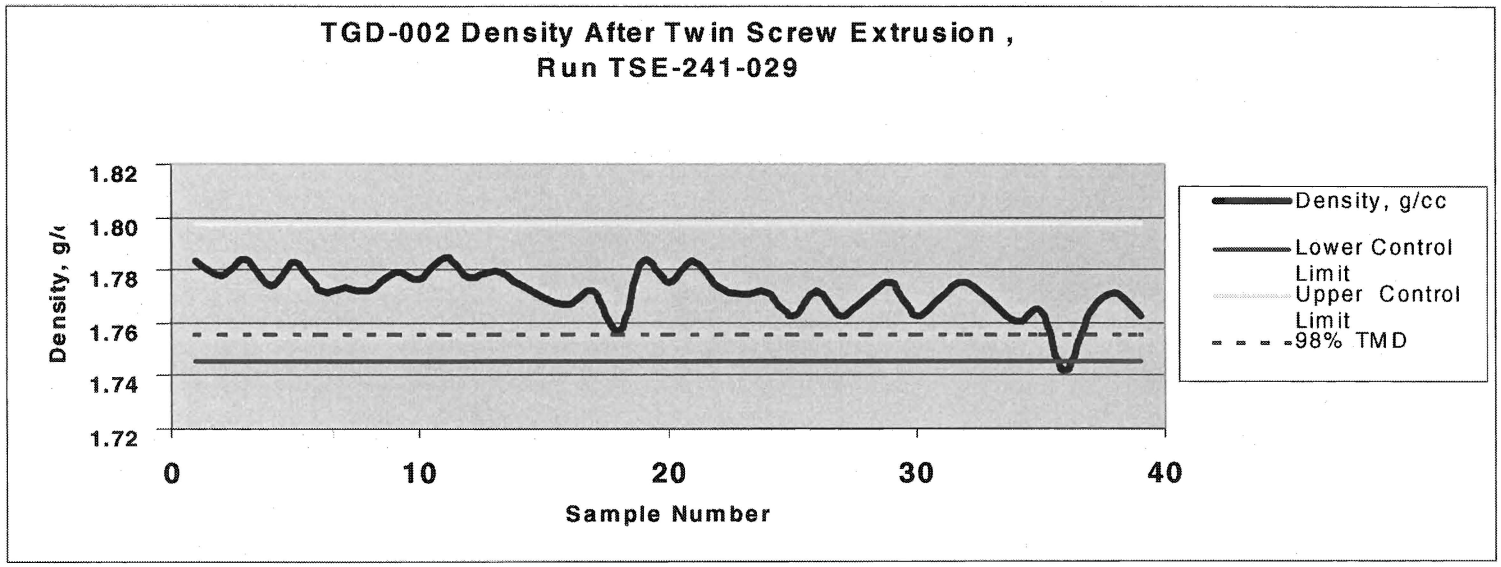




M-241 Extrusion Facility Processing

EXTRUDATE QUALITY

- **Most commonly used measures of extrudate quality are density and surface finish**
  - Surface finish is a qualitative measurement; however, poor surface finish can indicate low density
  - High density extrudate has been achieved with vacuum application during long run times



Summary

- **M-241 19mm TSE facility has successfully been used to process energetic materials**
  - Approximately 1,500 lb total
  - Longest run time was 64 hours
- **Facility design was improved through past experience**
  - LAAP 58mm TSE
  - M-56 19mm TSE
- **Facility capable of producing a wide range of energetic materials**
  - Gun propellants
  - Explosives
  - Pyrotechnics

Processing Energetics Through the Totally Integrated Manufacturing Enterprise (TIME) Network

US ARMY, TACOM-ARDEC

Presented by:

Dave Gilson

For the JOCG CMEUG

May 12, 1999

Outline

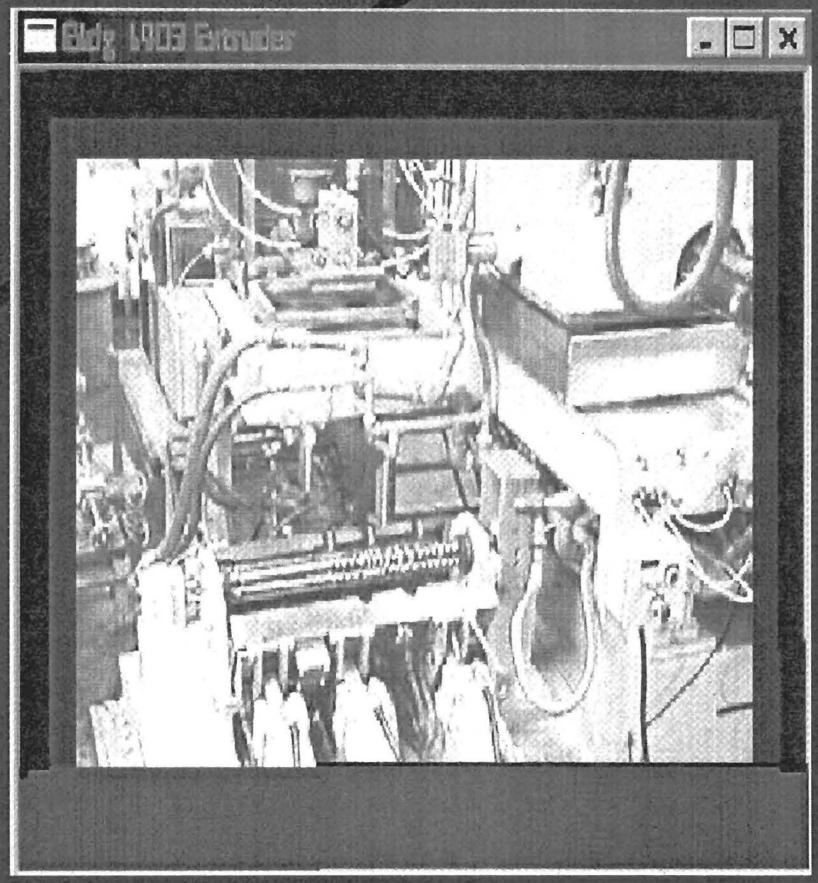
- Background
- Description
- Objectives
- Network Diagram
- Application of TIME
- Benefits of the Virtual Factory
- Future Objectives
- Summary

Background

- TIME: Totally Integrated Munitions Enterprise
  - Integration of Government, Industry, & Academia as a Virtual Enterprise for Munitions
  - Integration:
    - Level 1: Shop Floor Operations
    - Level 2: Business Processes
    - Level 3: Site-to-Site
- Creation of a Virtual IPT
  - Government: TACOM-ARDEC, Picatinny Arsenal, NJ
  - Industry: Thiokol Propulsion, Brigham City, UT
  - Academia: Stevens Institute of Technology, Hoboken, NJ

Description

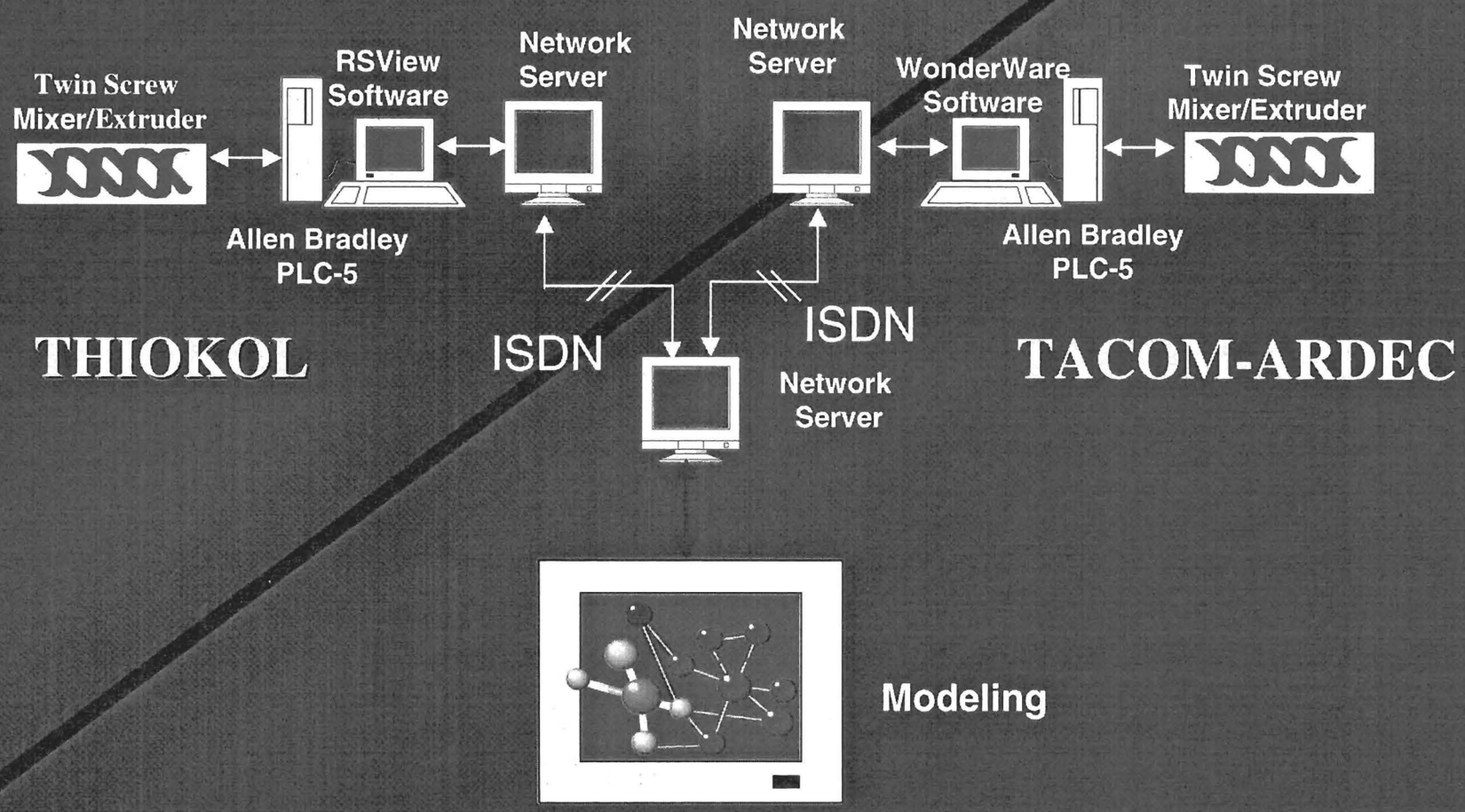
- Link Floor Operations at 3 Sites
  - Real-Time
    - Video
    - Data
  - Face-To-Face (VCON)
  - MeetingPoint



Objectives

- Establish Real-Time Video & Data Links to
  - Demonstrate Feasibility of Application of TIME Concept to Energetics Industry
  - Establish Application of Real-Time Process Modeling
  - Develop Lab Scale Processing
    - 19 mm Extruder: 2-10 lbs/hr
  - Demonstrate Pilot Scale Processing (Prototyping)
    - 40 mm Extruder: 10-35 lbs/hr
- Current Participants:
  - TACOM-ARDEC
  - Thiokol
  - Stevens Institute of Technology

Wide Area Network Connection



STEVENS INSTITUTE OF TECHNOLOGY

Application of TIME

Successful Wide Area Network Demonstration on 21 Oct 98

- PAX-2A Explosive Processing
- TACOM-ARDEC & Thiokol Extruders Network Broadcast Live Video & Data
- Real-Time Modeling by Stevens
- Final Product within 1% of Formulation

Benefits of Virtual IPT

- Realization of Integrated Process Team (IPT) Advantages w/out Co-Location of Personnel
- Shortened Development Time of Energetics
  - Demonstrated with PAX-2A Processing on a Twin Screw Mixer/Extruder at multiple sites
- Enhanced Communication and Integration of Participants
  - Connecting Government, Academia, Industry
  - Enabling Rapid Technology Development/Transfer
- Flexible Capacity to Manufacture Energetic Materials

Future Objectives

- Integration of Production Scale Equipment (Thiokol's 58 mm Extruder)
- Upgrade from ISDN to T1 Lines
- Utilize the TIME Network for a Multi-Base Propellant Formulation
- Expand TIME Network:
  - Include Level 2 Processes
  - Additional Partners & Industries
  - Model Based Controls

Twin Screw Processing of Gas Generant for Air Bag Applications at Autoliv ASP

Gary Smith

Manager, Process Development

10th Meeting of the JOCG Continuous Mixer & Extruder Users Group

11 - 12 May 1999

Autoliv

Topics

Overview of Airbags

- History and Statistics
- Principles of Operation
- Current Applications of Extruded Pyrotechnics
- Future Directions for Airbags
- Web Sites for Additional Information
- Airbags Now Video
- Paris Auto show Video

Autoliv Extruded Pyrotechnics

- Types
- Volumes

Autoliv Extruder Types

Autoliv Extrusion Processes

- Safety Considerations
- Critical Extrusion Process Parameters
- Manufacturing Processes Overview

Autoliv

Overview of Airbags

History and Statistics

- Airbags first installed on limited basis in early 1970's
- Airbags mandated by law approximately 20 years later
- Airbags can inflate up to 200 mph to meet automotive crash requirements
- Over 85 million cars and light trucks possess driver airbags
- 3.3 million driver airbag deployments through December 1998
- 600,000 passenger airbag deployments through December 1998
- NHTSA estimates over 4,100 lives saved with airbags as of April 1999

Principles of Operation

Current Applications for Extruded Pyrotechnics

- Driver Pyrotechnic
- Driver Smart Pyrotechnic
- Passenger Pyrotechnic
- Passenger Hybrid
- Passenger Smart Pyrotechnic
- Passenger Smart Hybrid
- Side Impact Pyrotechnic

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Overview of Airbags (cont.)

Future Directions

- Higher gas conversion - Smaller, fewer components, cheaper
- Gentler airbags
- Multi-level performance inflators
- Seat belt pre-tensioners and load limiters
- Seat belt condition monitoring
- Seat position / Seatback angle sensors
- Occupant weight detectors - Segmented pad, load cell, strain gauge, averaging method
- Occupant position - Ultrasonic, infrared, capacitance, radar
- Steering wheel position
- Tags on rear facing infant seats
- Improved crash sensors - Collision severity and vector
- Crash warning and avoidance - Radar
- Roll-over protection - Inflatable curtain
- Side-Impact Airbags - ITS, Thorax, Head
- Knee bolster
- Inflatable Carpet

Autoliv

Overview of Airbags (cont.)

Web Sites for Additional Information

- http://www.autoliv.com
- http://www/highwaysafety.org
- http://www.nhtsa.dot.gov

Airbags Now Video

Paris Auto show Video

Autoliv

Autoliv Extruded Pyrotechnics

Types

- Honeycomb PVC Propellant
- Single Perf. Or solid grain Guar Gum Propellant

Volume

- Extruded Products

	CY'96	CY'97	CY'98	CY'99
Quantity (lbs)	56,000	163,000	281,000	700,000

Autoliv

Autoliv Extruder Types

	Description	Rates (lb / hr)	Die Ports
Haake	25mm Single Screw	8	1
APV	40mm Twin Screw (SE-1600)	90	Honeycomb
B & P	50mm Twin Screw	70	1 to 2
B & P	65mm Twin Screw	200	3 to 4
B & P	100mm Twin Screw	600	14

Autoliv

Autoliv Extrusion Processes

Safety Considerations

- Minimize personnel exposure
- Minimize quantity of explosive
- Minimize number of people
- Minimize processing time
- Monitor screw to screw and screw to barrel clearances
- Pin screws through coupler
- Screen all raw materials for contaminants
- Processing start up
- Processing Equipment Interlocks
- Die design
- Materials of construction
- Proper alarm conditions and settings
    - Temperature, pressure, torque
- Loss of liquid or solid feed streams
- Solvent volatility
- Stagnant zones

Autoliv

Autoliv Extrusion Processes (Cont.)

Critical Extrusion Process Parameters (Measured by Manufacturing)

- Raw materials - purity, particle size, moisture
- Die head and barrel pressures
- Control temperatures
- Melt / material temperatures
- Motor load / torque
- Screw speed
- Clearances
- Screw segment sequence
- Solid feed rates
- Liquid feed rates
- Product sampling
- Cutter speed and number of blades

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Autoliv Extrusion Processes (Cont.)

Manufacturing Overview

- PVC Propellant
  - 40mm twin screw
- Water based propellant extrusion
  - 65mm twin screw
  - 100mm twin screw

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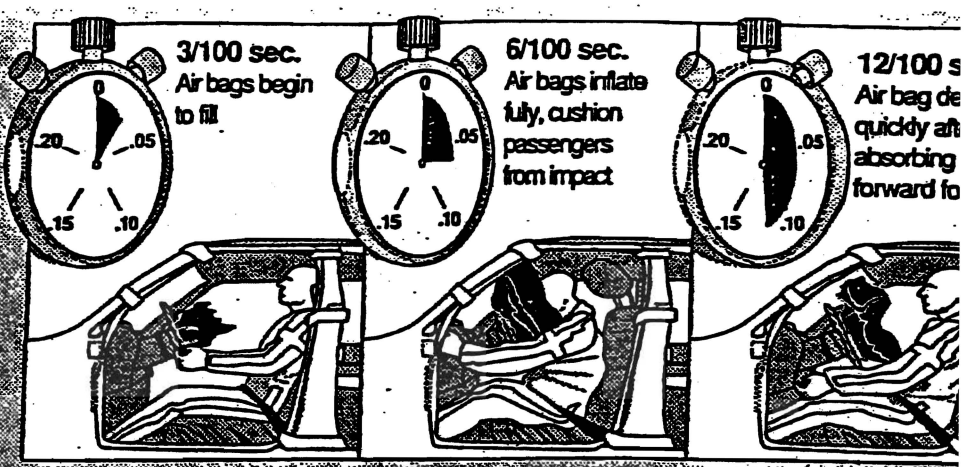
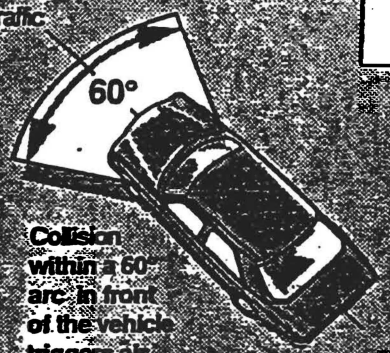
Air bags

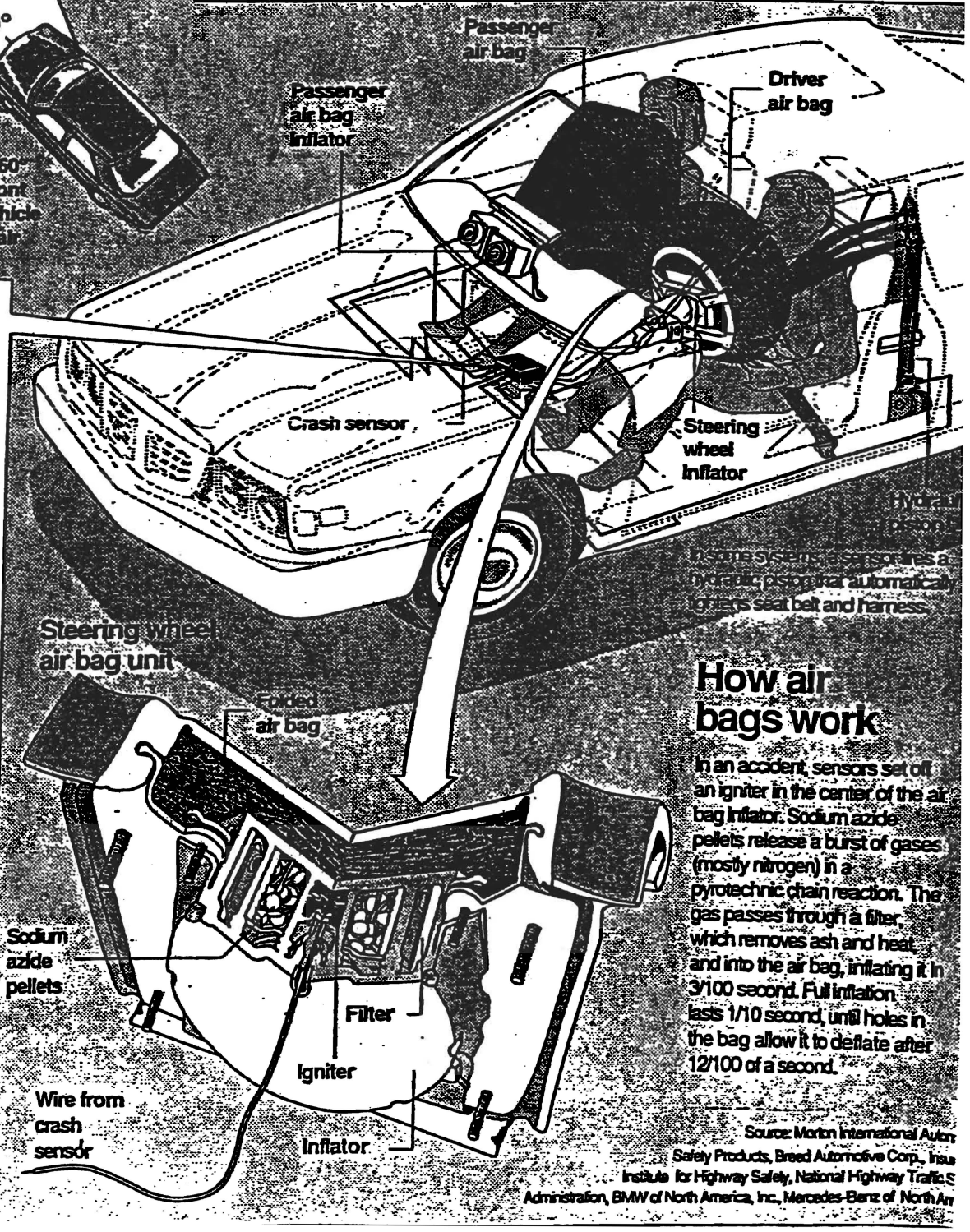
The U.S. Department of Transportation has required automakers to install passive restraints—either air bags, automatic seat belts or a combination, in all 1990-model cars.

- The force of almost all collisions is spent within 1/8 second. An air bag inflates in less than 1/25 second, then deflates.
- More than half of severe traffic injuries and deaths are due to frontal collisions. Air bags are designed specifically to protect passengers in frontal or front-angle collisions.
- Advanced safety system cannot protect in all crash situations. Severe injury or fatality is possible even when all systems work properly.





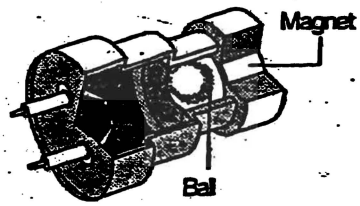
Collision within a 60° arc in front of the vehicle triggers air bags.



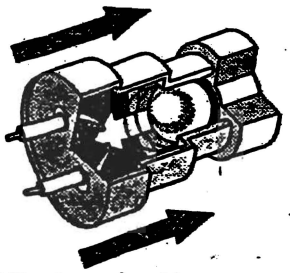
Inertial switch

Simple, dependable switch triggers air bag and seat belts.

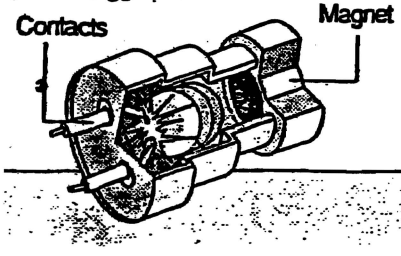
Gold-plated ball sits inside a sealed cylinder, held in place by air pressure and a magnet.



Inertia from rapid deceleration after impact breaks magnet's hold, throws ball forward.



Ball flies forward, making electrical contact within 1/600 second. Fender-bender collisions up to 15 mph do not set off trigger.



How air bags work

In an accident, sensors set off an igniter in the center of the air bag inflator. Sodium azide pellets release a burst of gases (mostly nitrogen) in a pyrotechnic chain reaction. The gas passes through a filter, which removes ash and heat, and into the air bag, inflating it in 3/100 second. Full inflation lasts 1/10 second, until holes in the bag allow it to deflate after 12/100 of a second.

Source: Morton International Autom Safety Products, Breed Automotive Corp., Insu Institute for Highway Safety, National Highway Traffic S Administration, BMW of North America, Inc., Mercedes-Benz of North Am

Autoliv's Safety Systems

1. COLLISION WARNING SYSTEM

A system which will detect – by use of radar – likely crashes, warn the driver, apply the brakes and alert the safety systems of the vehicle. The system will also include an Adaptive Cruise Control which will maintain the car at a constant distance from the vehicle ahead. A joint development project with CelsiusTech.

2. NEAR-ZONE SENSORS

Will help deploy the airbags and other safety systems of the vehicle earlier and in a more precise and accurate way, depending on the crash circumstances. Will also tell the driver the distance to other objects when parking. A joint development project with CelsiusTech.

3. ELECTRONIC CONTROL UNIT

A micro processor which determines, by use of a built-in electronic crash sensor, if and exactly when the seat belt pretensioners and the airbags should be deployed.

4. INFLATABLE CARPET (InCa)

An airbag that cushions the impact from an intruding floor panel on an occupant's feet, ankles and lower legs.

5. ITS (INFLATABLE TUBULAR STRUCTURE)

The world's first head airbag for side-impact protection. Introduced in 1997 in cooperation with BMW and the American company Simula.

6. STEERING WHEEL

Driver airbags are increasingly being integrated into the steering wheel. Autoliv is a leader in this development through its subsidiary *Isodelta* in France, one of Europe's leading steering wheel producers. In 1998, production of steering wheels also started in the United States.

7. DRIVER AIRBAG

Reduces driver fatalities in frontal crashes by 25% and the risk of facial injuries by 75% for belted drivers. Autoliv not only develops and assembles various airbag systems (full-size bags, face bags, gentle bags smart bags, etc.), but also produces all key components.

8. KNEE AIRBAG

First introduced in the world market in 1995 by Autoliv. Protects knees (and hip) and reduces the risk for submarining. Unlike conventional knee bolsters and paddings, knee airbags don't take away any leg room.

9. PASSENGER AIRBAG

Reduces fatalities in frontal crashes by approximately 20%. Autoliv has a complete offering of various systems (full-size bags, face bags, smart bags, etc.) and a comprehensive offering of airbag inflators, cushions and other key components.

10. INFLATABLE CURTAIN (IC)

Introduced in 1998 on Volvo, Mercedes, Toyota and Audi cars. With one *Inflatable Curtain* on each side of the vehicle, the heads of the driver and all passengers seated next to the sides are protected in side collisions and roll-over accidents. Manufactured using Autoliv's patented one-piece weaving technology.

11. ANTI-WHIPLASH SEAT (AWS)

Introduced in 1998 in the world market in the new Volvo car. Reduces significantly the risk for neck injuries in rear-end collisions.

12. SEAT FRAMES & SLIDING RAILS

Produced by Autoliv since 1996. Autoliv has developed a unique, stronger recliner for *Belt-In-Seat* Systems.

13. BELT-IN-SEAT (BIS)

Allows the shoulder belt to better wrap around the occupant's body, thereby increasing the efficiency of the belt system. Also contributes to keeping the clearance between the head and the roof in roll-overs.

14. INTEGRATED CHILD SEAT

Makes it possible for children to use the car's regular seat belt (which is better than a separate belt). Since the seat is integrated into the backrest of the rear seat, it does not take away any space when not used.

15. SAFETY REAR SEAT STRUCTURE

A backrest in the form of a strong, specially-designed aluminum structure which prevents luggage from penetrating into the passenger compartment in a frontal crash.

16. SEAT BELT BEAM

A macro-component with three pre-installed 3-point seat belt systems. The beam facilitates installation of a 3-point seat belt system in the mid-rear seat, especially in hatchbacks and station wagons. Also provides passengers with improved side-impact protection.

17. TRUNK BELT

A safety belt for the luggage compartment. Protects fragile goods and prevents luggage from penetrating into the passenger compartment in cars with foldable rear seats.

18. SEAT BELT SYSTEMS

Produced by Autoliv since 1956. Autoliv today has the most comprehensive offering and in-house production of all key components and new seat belt features (see page 12), such as:

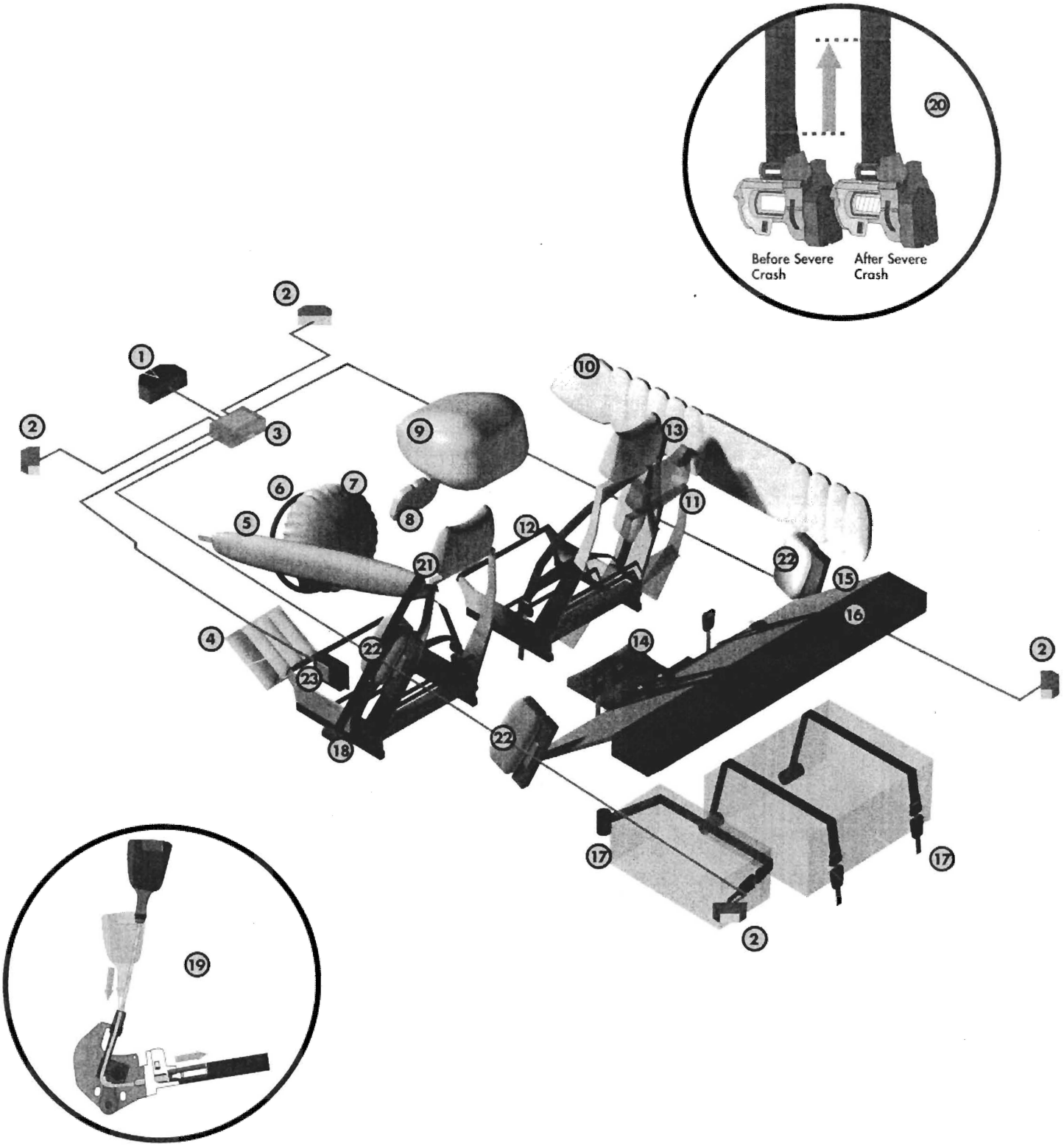
19. Pretensioners
20. Load limiters
21. Automatic height adjusters.

22. THORAX BAGS

Introduced in the world market by Autoliv and Volvo in 1994. Is as efficient in side impacts as a front airbag in frontal crashes.

23. SIDE-IMPACT SATELLITE

An electronic sensing system which determines if and when the different side-impact protection systems should be triggered.


Before Severe Crash
After Severe Crash
1
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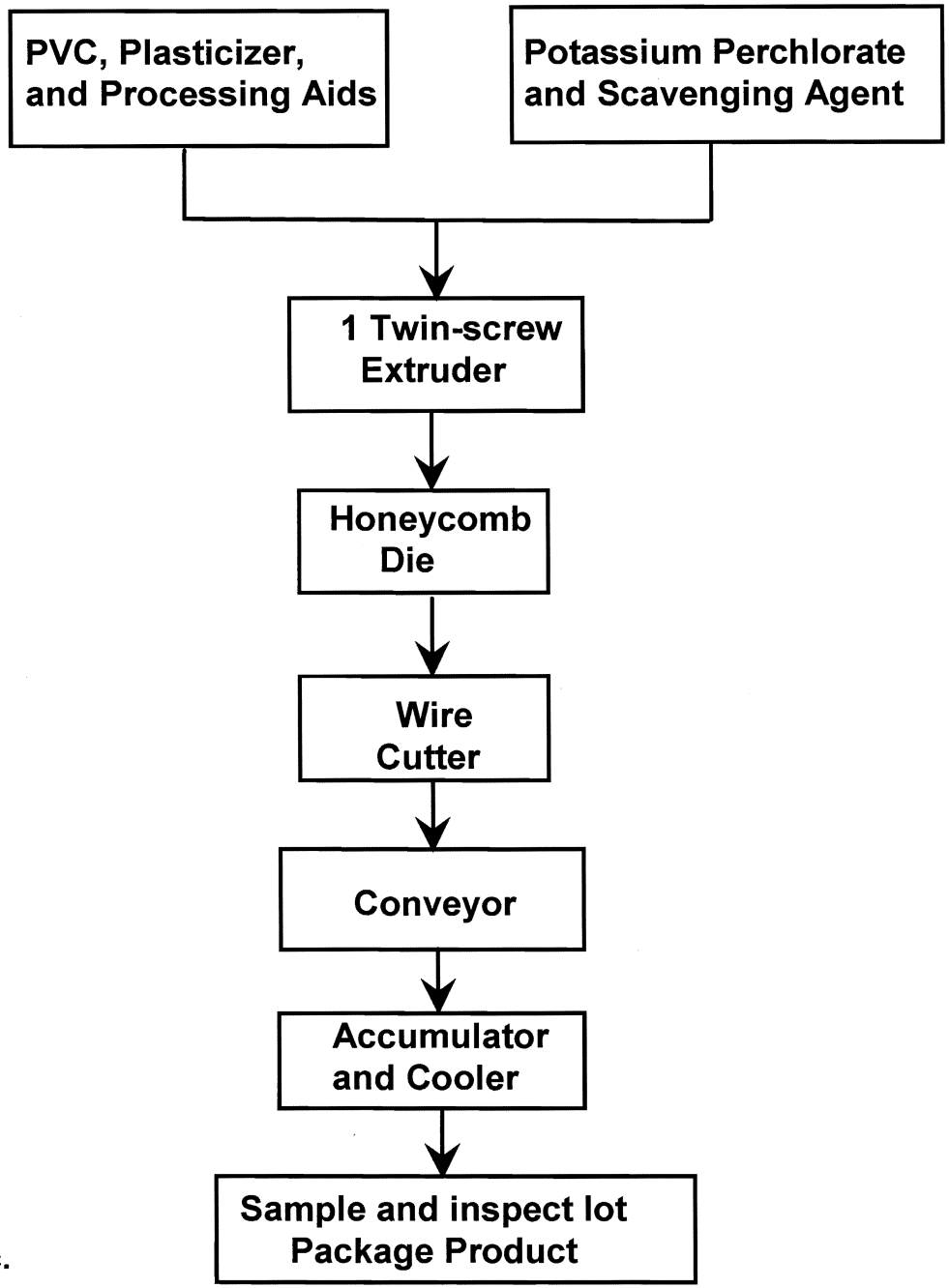
PVC HYBRID PROPELLANT MANUFACTURING FLOW



Blend PVC/DOA /PA's to form plastisol. Mix solid components with blender. Add ingredients with separate LIW feeders through feed funnel and injection port.

Disperse and mix ingredients, and bring to 325 F to form plastisol propellant.

Extrude plastisol propellant into desired shape through multi-perforated die.

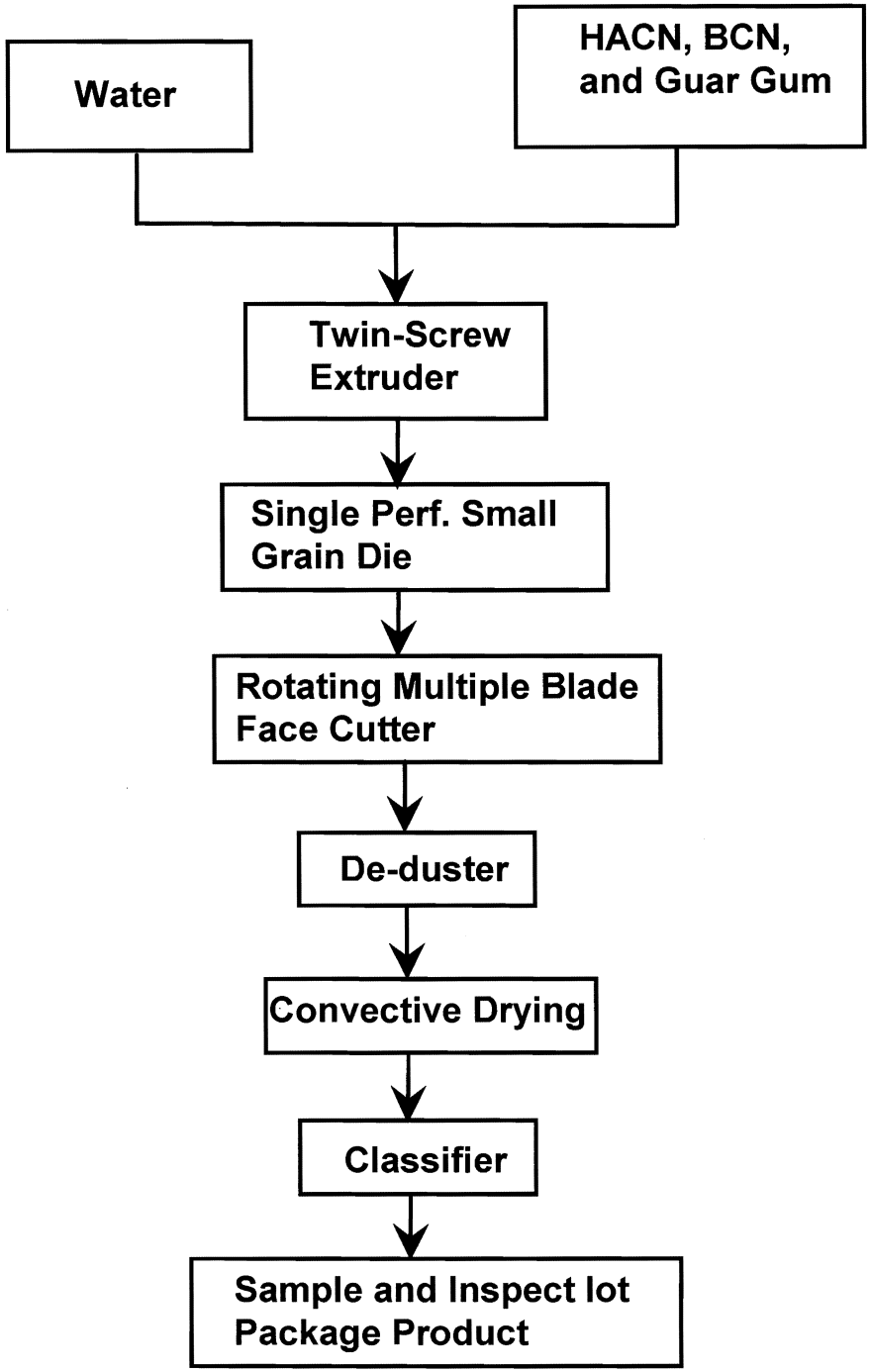
Cut propellant into easily managed length.

Transport propellant through blast wall into collection bay.

Shuttle propellant onto cooling conveyor system.

Inspect and test propellant sub-scale properties.
Prepare for shipment to customers

GUAR GUM PROPELLANT MANUFACTURING FLOW



Blend solid ingredients with Forberg blender. Add solid blend with LIW feeder through feed funnel. Meter in water through injection port.

Disperse and mix ingredients, chill product to maintain desired product temperature.

Extrude water based generant or advanced solid fuel into desired shape

Cut propellant into specified length

Transport product across de-duster

Dry product per planning to meet specification requirements

Classify product

Test propellant sub-scale properties
Prepare for shipment to customers

Autoliv ASP, Inc.
12 May 1997

NEXPLO
BOFORS

EXPLOSION IN PROPELLANT PRODUCTION PLANT

Dr. Per Lagerkvist
NEXPLO Bofors AB
SE-691 86 Karlskoga, Sweden

INTRODUCTION

NEXPLO Bofors is manufacturing LOVA propellant by continuous extrusion in a co-rotating twin-screw extruder. The plant for continuous extrusion of LOVA propellant was built in 1992. The process is outlined in Figure 1 and consists of two separate steps, the first is the precipitation process and the second the extrusion of the propellant. In the precipitation process (Figure 2) all the propellant ingredients are dissolved in a solvent. The solution is then pumped to a stream-driven ejector where the product, the so-called LOVA powder paste, is precipitated at the same time as the solvent is recovered. The LOVA powder paste is obtained in a water slurry, which is filtered and then the powder paste is dried. The dried powder paste is a well desensitized RDX, which is used in the extrusion process.

The plant for extrusion of LOVA propellant is installed in three bunkers (Figure 3). The extruder is located in one bunker on the ground floor. This is fed by a gravity feeder, which is located in a bunker on the floor above directly over the extruder. The gravity feeder is refilled by the use of a vacuum transporter. The LOVA powder paste is extracted from a bunker in another building. This process equipment arrangement provides the highest degree of personnel safety. This was also experience in both our accidents as there were no injuries as a result of the explosions, although the material damages were severe.

The most difficult problem in continuous extrusion is to feed the ingredients into the extruder in a controlled manner in the correct proportions to each other. This problem has been solved by the use of the precipitated powder, which gives only one solid ingredients to feed. The LOVA powder paste is mixed with solvent in the extruder and is then extruded to propellant strands of desired dimensions. The propellant strands are then cut into the required lengths and they are then dried, sieved and graphited before being packed.

The work in our extruder has mostly been done with slightly modified M 39 LOVA formulation, a typical formulation is shown in table 1.

DESCRIPTION OF THE ACCIDENTS

Two accidents have occurred in our twin-screw extruder since the start of the LOVA propellant manufacturing 1992. Both accidents involved pilot lot production of a new type of a triplebase nitramine propellant.

The first one occurred on 26th February 1997. In that run the ballistic variation of propellant made from different powder paste batches was tested. The explosion occurred when the extrusion was restarted with powder paste batch number 2. The experiment was run with a little bit different procedure than normal production. In the normal procedure the extruder is completely emptied between different batches. In this case the change was done in the following way. The extruder was stopped with about 1,5 kilo of solvent wetted powder paste left in the extruder after batch number 1. The feeder system was run until no powder paste from batch no. 1 was left. Batch no. 2 was then introduced into the feeder. The whole system was then restarted and after about one minute the explosion occurred. The hydraulic pressure was normal just until the explosion, when it increased rapidly. The pressure in the die head was a little bit higher than normal during that run with a drastic increase just before the explosion.

The ignition of the powder paste resulted in a detonation in the extruder, where all explosive materials reacted. The detonation did not , however, spread to the feeder or the die, where unreacted powder paste was found. About 1,5 kilo of the nitramine powder paste participated in the explosion. The twin-screw extruder was extensively damaged with both the barrel and the screw elements completely destroyed. The bunker, which is calculated to withstand a detonation of 3 kilo TNT, was not affected and the light blow-out wall at the back of the bunker functioned as predicted.

The second accident occurred on 14th May 1998. In that run the purpose was to produce a pilot lot of propellant for test shooting. The production had been going on continuously since the day before. There had been some problem with the feeding system. The powder paste had a tendency to stick to the wall of the feeding pipe between the gravity feeder and the extruder. After some hours of running the powder on the wall suddenly got loose (a "fall"), which led to a momentary increase in the flow of the powder paste into the extruder. This was immediately followed by a slight increase in the hydraulic pressure and after two minutes an increase in the pressure and temperature in the die. Then all temperatures and pressures went back to normal value.

At the accident there was a "fall" followed by a slight increase in the hydraulic pressure and after two minutes an increase both in the pressure and the temperature in the die. The limiting values on both parameters in the die were reach, but the shutdown function was not quick enough to prevent an accident. The ignition of the explosive materials most probably occurred in the first kneading zone. The result of the second

explosion was almost the same as in the first accident with severe damaged on the extruder but no effect on the bunker and no injuries.

DISCUSSION

The investigation of the first explosion has shown that the solvent flow failed to come to the extruder. This was due to blocked injection tubes. The tubes were blocked with powder paste during standstill between the running. This was not observed and the alarm/shutdown function did not work. Thus dry powder paste from batch no. 2 was feeded into the extruder, while the wetted powder paste from batch no. 1 was extruded through the extruder and the die. When the dry powder paste reached the die the material flow in the extruder stops. This resulted in an extensive kneading of the material with a quick temperature raise of the powder paste. The ignition occurs in the first kneading zone with a detonation as a result.

The conclusions from the first accident were the following. The flow-gauge has been replaced by new types and new types of solvent pumps have also been installed. The extrusion process is today controlled by a PLC-system, which means that every running is controlled by a recipe. The recipe contains all process parameters as well as the alarm/shutdown function for each type of propellant.

In the investigation of the second accident the faultree analysis method has been used. The explosion has been divided into five main events, which are summarised in table 2. The investigation has not been finished yet, but the analysis has shown that there could be several different causes for that accident. The following conclusions have so far been drawing from that explosion. The most probably reason is that the "fall" led secondary to either a mechanical breakdown or a quick temperature increase in kneading zone 1.

The investigation of the second accident has led to the following leaning points.
The computer system is very reliable.

There is a problem with static electricity in feeding LOVA powder paste from a feeder situated in a separate room above the extruder. The free fall of the powder paste in the interconnection tube between the gravity feeder and the extruder will introduce a problem. The electric energy is, however, not strong enough to ignite the powder, but the powder will stick to the wall due to the static electricity. This could lead to the problem we have experienced with the suddenly occurrence of "falls".

It is very important to choose the right material in the barrel and in the liners as well as the screw elements.

The temperature measurement is very difficult in an extruder. Usually the temperature is measured with a thermocouple where the measuring probe is at the surface of the barrel. This arrangement could in fact lead to a measuring only of the barrel temperature not of the powder paste. It is also very important that there is a flow of material at the thermocouple not a stagnation point.

The problem with foreign material is well documented in the investigation. This has led to extremely stringent instruction for handling LOVA powder paste. That goes all the way from the precipitation line to the extruder line.

The properties of different powder paste have also been investigated. The rheological properties have been measured and the dependence on the process parameters in different parts of the extruder have been studied.

The production of LOVA propellant in our extruder will in the future be based only on well established process design. The different types of propellants have its own recipe with the running parameters and the limiting value close to each other, which leaves only a very narrow space for process operation.

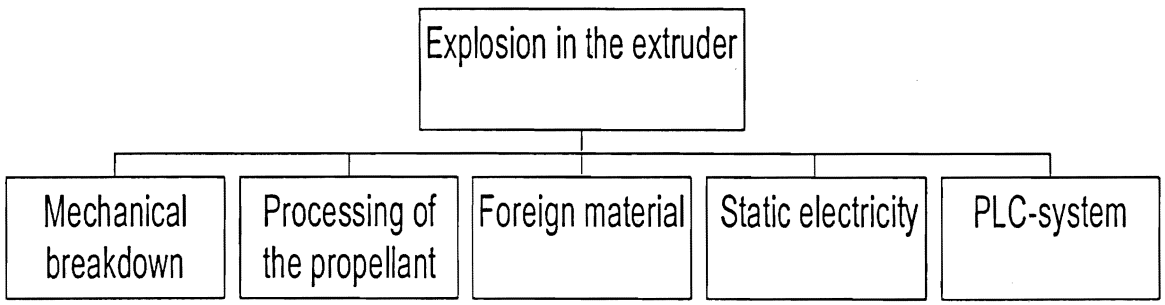
TABLE 1

LOVA FORMULATION, NL 001

Ingredients	Weight %
RDX	76
Cellulose Acetate Butyrate	12
Tri Butyl Citrate	7,6
Nitrocellulose	4
Ethyl Centralite	0,4

TABLE 2

FAULTREE ANALYSIS



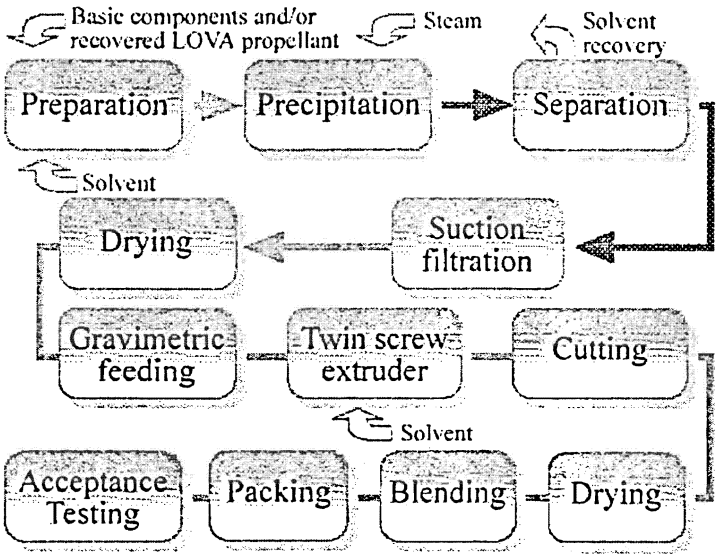
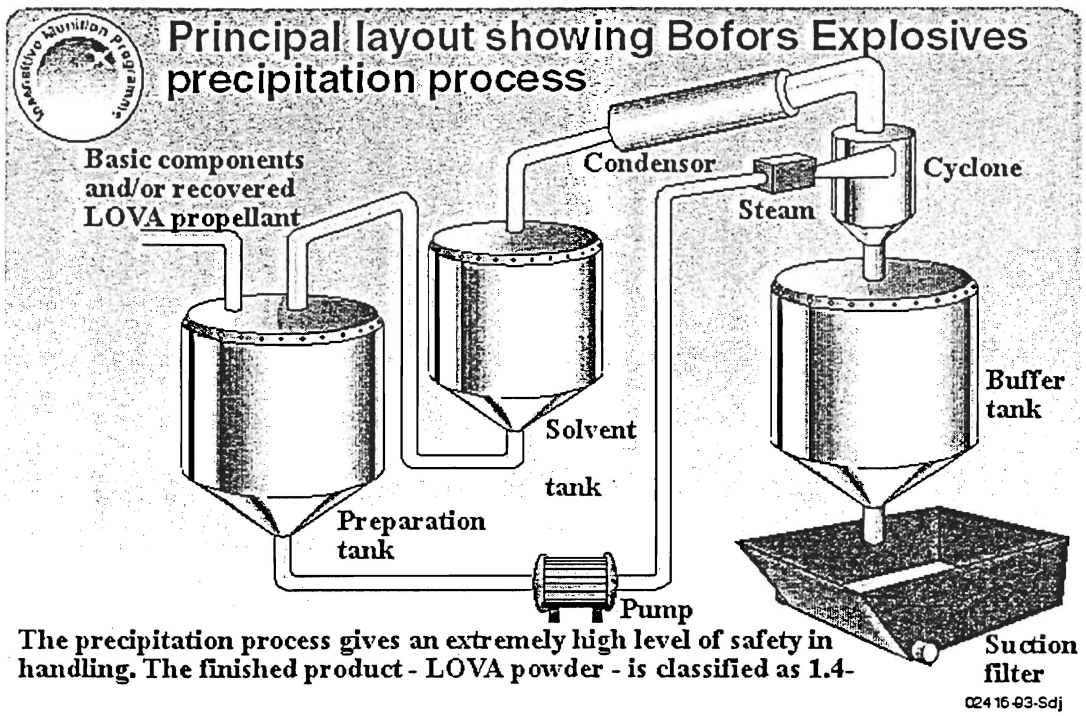
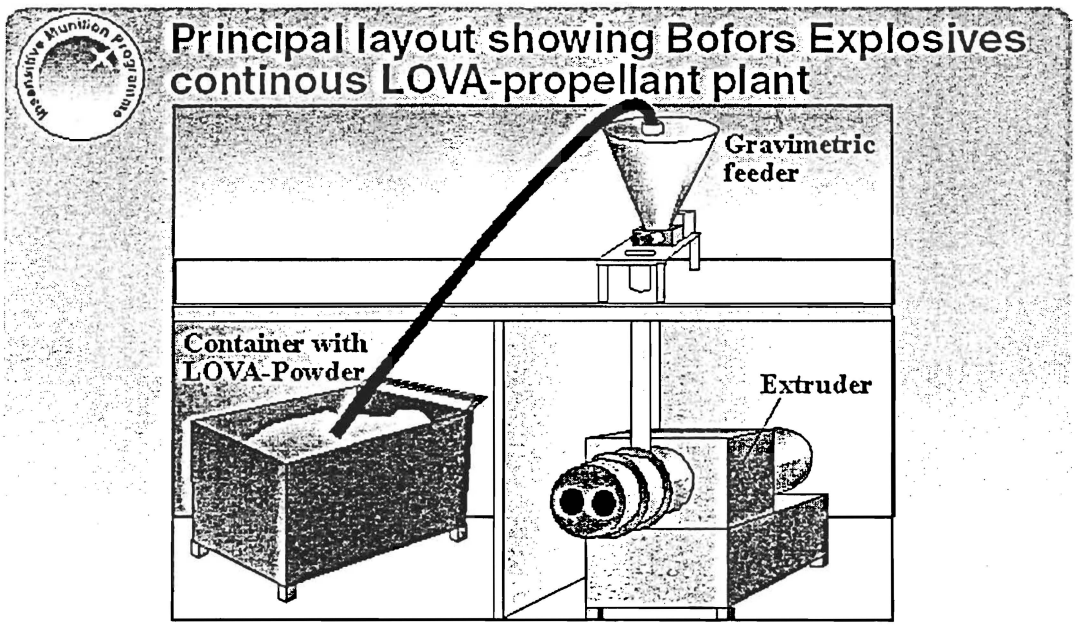

Basic components and/or recovered LOVA propellant
Steam
Solvent recovery
Preparation
Precipitation
Separation
Solvent
Drying
Suction filtration
Gravimetric feeding
Twin screw extruder
Cutting
Solvent
Acceptance Testing
Packing
Blending
Drying

Figure 1



The precipitation process gives an extremely high level of safety in handling. The finished product - LOVA powder - is classified as 1.4-

Figure 2



LOVA powder gives an extremely safe extruder process.
Both the LOVA Powder and the produced LOVA Propellant are classified as 1.4-S.

02417-93-Sdj

Figure 3

A METHOD OF ISOLATING ENERGETIC MATERIAL FEED SYSTEMS FROM EXTRUSION PROCESSES

Prepared by:

Mike Rose, Mike Harper, Steve Bradley, and Andy Haaland

Thiokol Propulsion

From Cordant Technologies

Outline

- Historical Background
- Objectives
- Design Review
- Simulated Extrusion Ignition Tests
- Conclusion

Historical Background

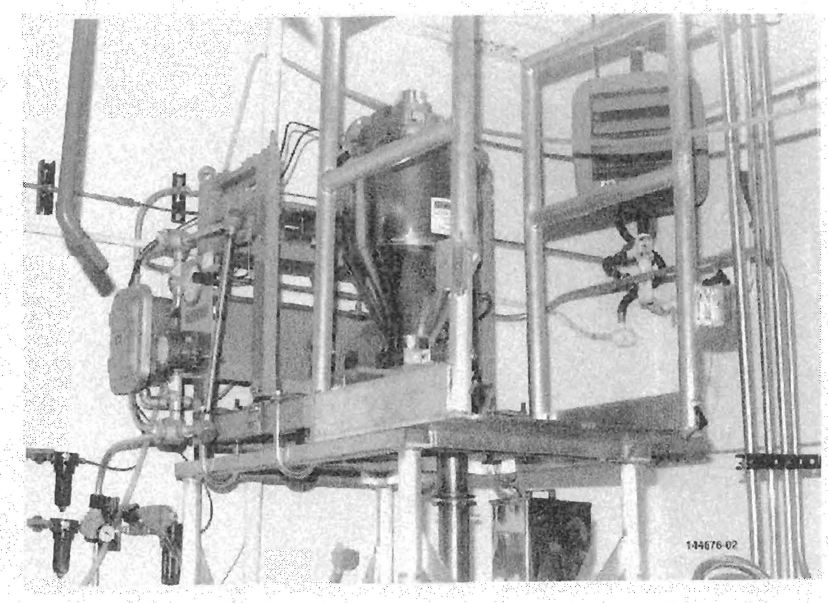
- **Twin screw extruder incident**
  - 19mm TSE
  - Extrudate was thermoplastic nitramine with a molding powder feedstock
- **Lessons learned**
  - Ignition began in the front end of the extruder and propagated through the extruder into the feed hopper
  - Majority of equipment and facility damage resulted from ignition of the material in the feed hopper
  - Blast plate effectively protected the extruder from the hopper explosion

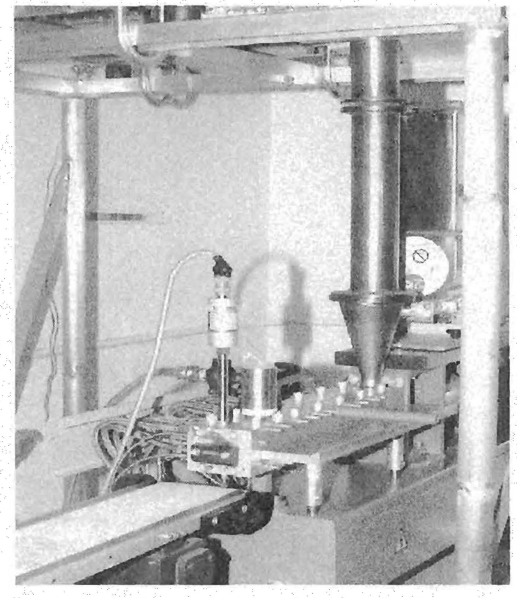
Thiokol Objectives

- **Minimize the risk for an incident**
  - Incorporate recommendations from M-56 incident investigation team and the energetic processing community
- **Minimize the potential for flame propagation**
  - Install fast acting deluge
  - Isolate feed system from the extruder
- **Maintain product quality**
  - Produce homogenous propellants and explosives

Design Review

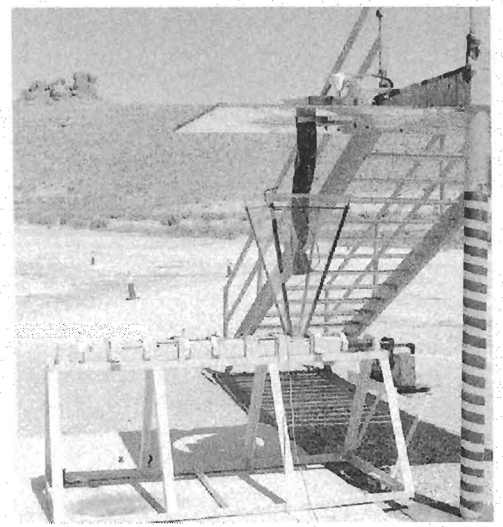
- **Feed system design for 19-mm prior to incident**
  - Blast plate on mezzanine for equipment protection
  - Loss in weight (LIW) feeder positioned on the mezzanine
  - Feed funnel attached to the extruder
  - Hard pipe connection between LIW feeder and feed funnel

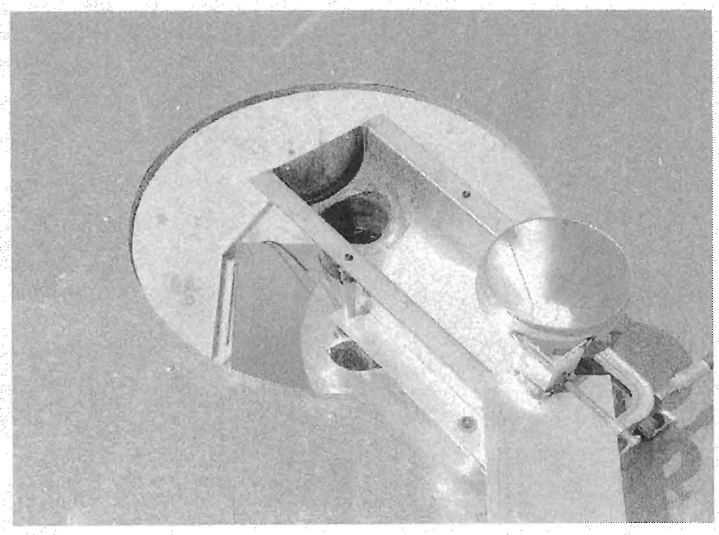




Design Review (Cont.)

- **Preliminary feed system design for 58-mm prior to M-56 incident**
  - LIW feeder positioned on the mezzanine
  - Feed funnel attached to the extruder
  - Soft sock connection between LIW feeder and feed funnel
  - Large access hole (~2 feet in diameter) to facilitate multiple solid feed streams
  - Vibrating tray to convey material from the hopper to the extruder





Simulated Extrusion Ignition Tests

- **Initial extrusion ignition test configuration**
  - Mock blast plate
  - Large opening in the blast plate (~ 2 feet in diameter)
  - Simulated vibrating tray and feed hopper
  - Soft sock connection between the vibrating tray and extruder
  - Extruder funnel for material containment
- **Test observations**
  - Design did not provide adequate feed system isolation
  - Extruder funnel channeled the flame engulfing the entire feed system

Simulated Extrusion Ignition Tests(Cont.)

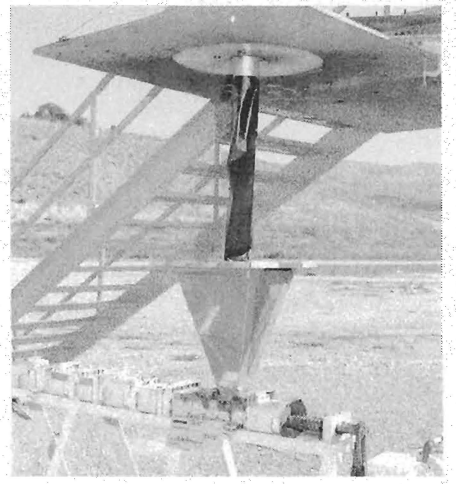
- **Design changes for the second extrusion ignition test**
  - Reduced opening in the mezzanine
  - A skeleton extruder funnel with blowout panels
- **Test observations**
  - Flames directed toward the feed system were significantly reduced
  - Flames still propagated through the feed sock igniting material in the vibrating tray and hopper

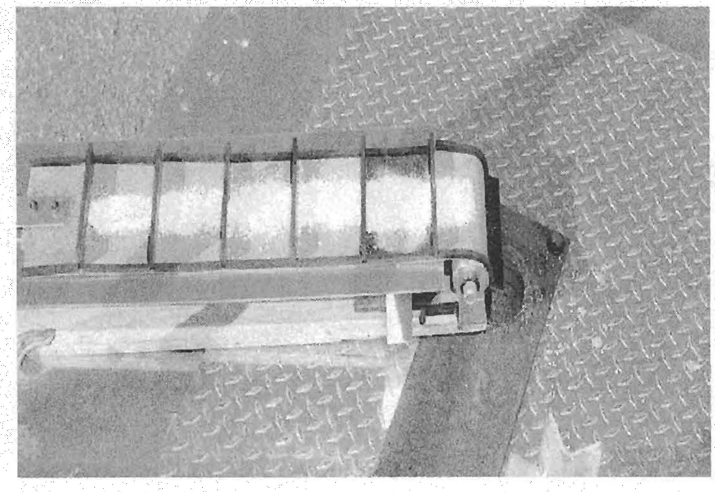
Simulated Extrusion Ignition Tests(Cont.)

- **Design changes for the third extrusion ignition test**
  - An open sided extruder funnel incorporating two baffles to direct flame away from the feed system
- **Test observations**
  - Design performed as expected. Flames were directed outward instead of upward
  - A small flash of flame propagated through the feed sock to the vibrating tray, however, material in the tray and hopper did not ignite

Simulated Extrusion Ignition Tests(Cont.)

- **Design changes for the fourth extrusion ignition test**
  - Replaced vibrating tray with a segmented conveyor belt
  - Added an additional baffle to the extruder funnel





- **Test observations**
  - Design performed as expected. Flames were directed outward instead of upward
  - No flame propagated to the feed system

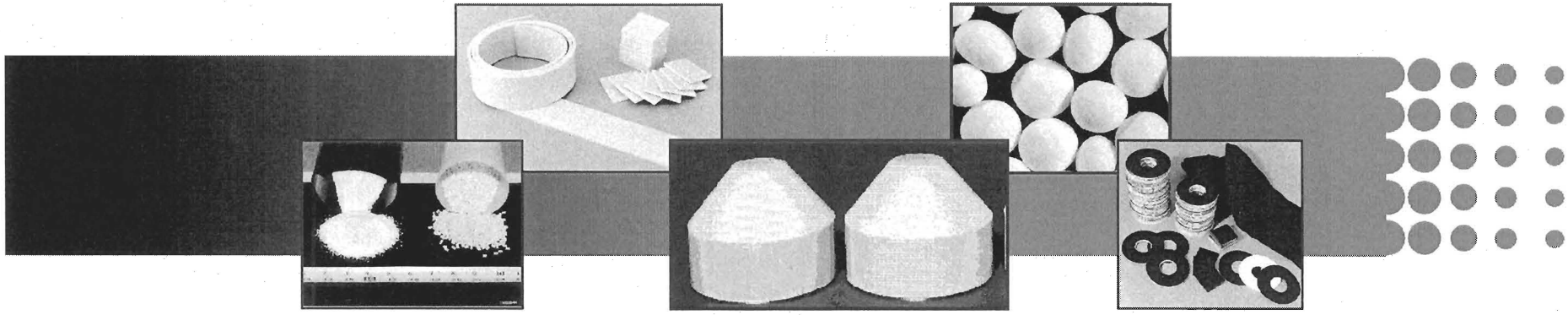
Conclusion

- Energetic material feed systems can be effectively isolated from the extrusion process

10TH JOCG CONTINUOUS MIXER AND EXTRUSION CONFERENCE

Molding Powder Production for Use in Extrusion

Prepared by:
Jeff Widener



Thiokol Propulsion™
From Cordant Technologies

May 11-12, 1999

990479.ppt

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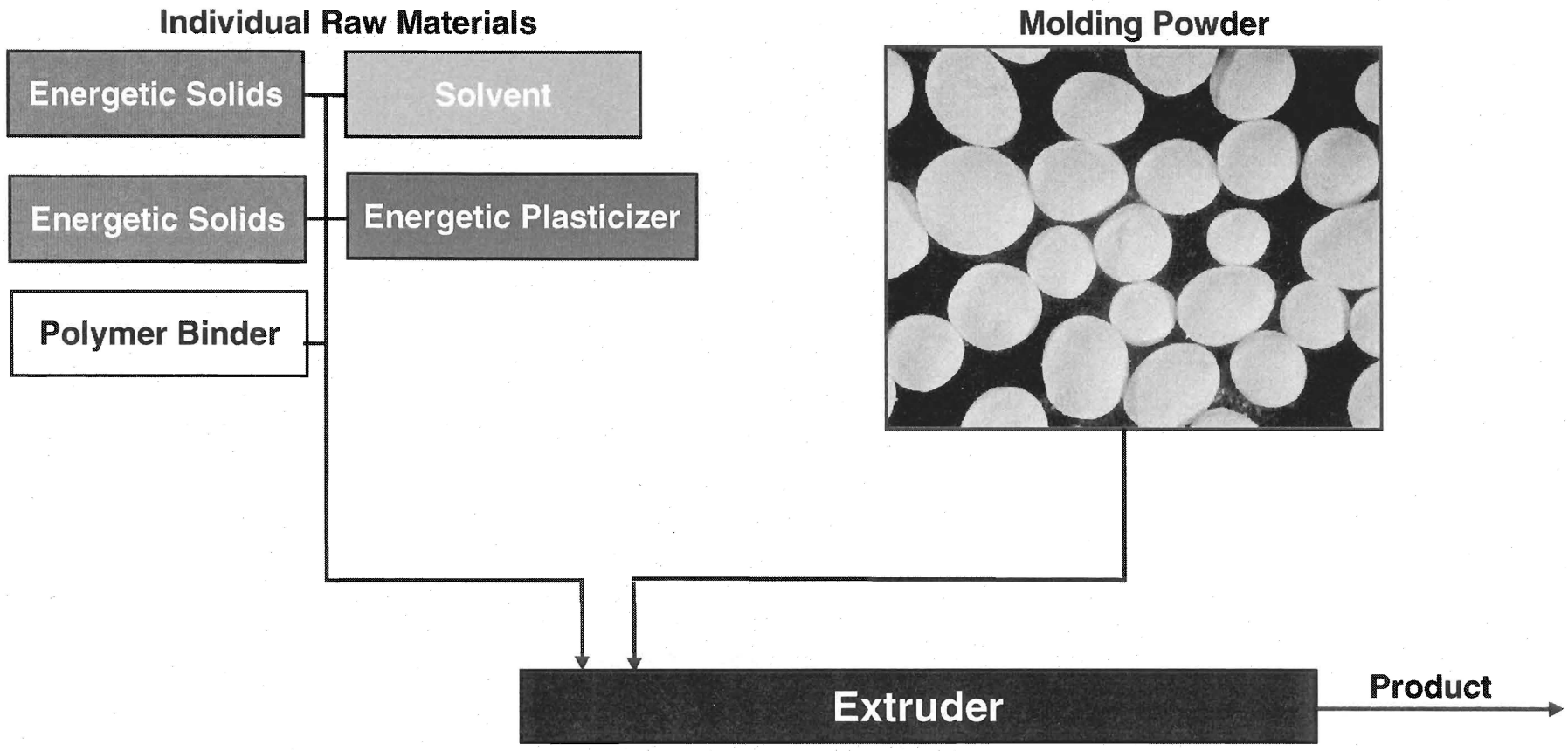
Discussion Topics

- **Why use molding powder as a feed for extrusion?**
  - Safety considerations
  - Process control
- **What are the current methods of molding powder production?**
  - Process
  - Target product

Safety Considerations

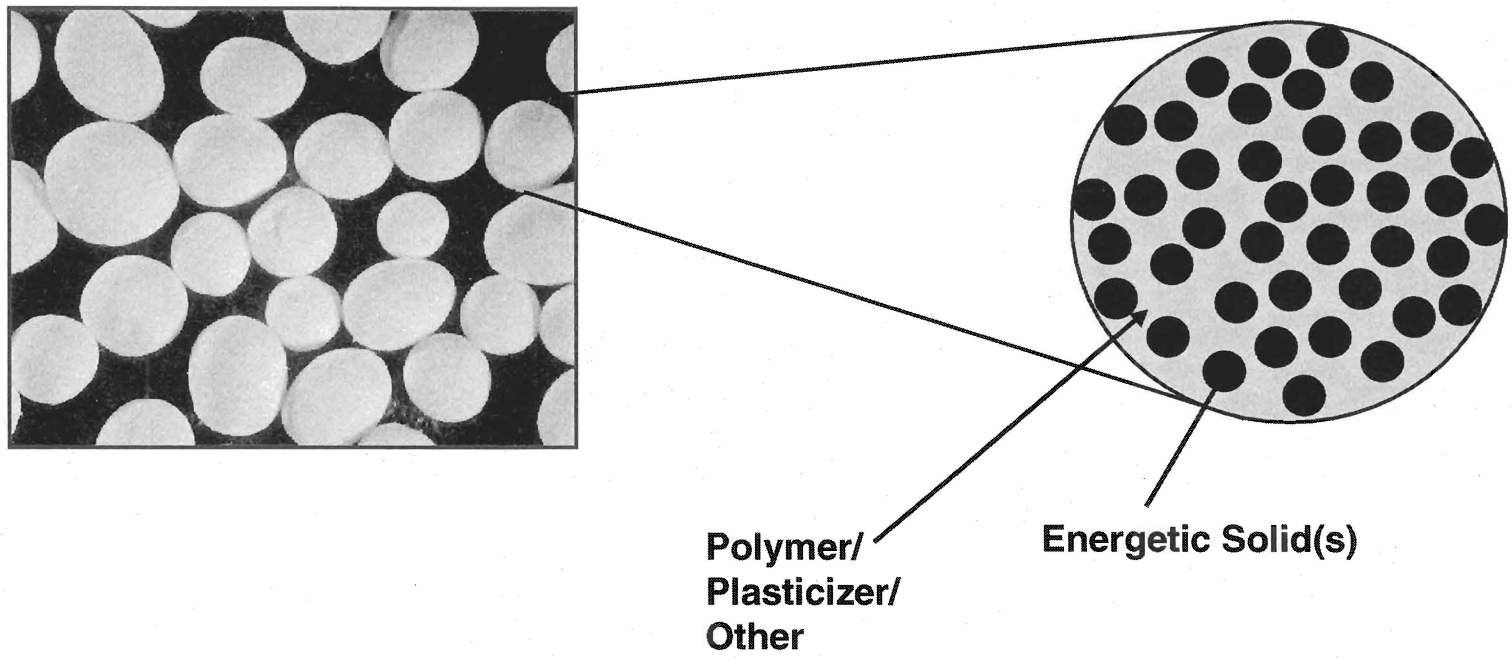
- **History**
  - In the past, individual components of energetic formulations have been successfully fed , mixed, and extruded. This includes dry energetic solids
- **Issues**
  - Pre-extrusion steps: Handling and loading of sensitive energetic solids may present ESD/other safety hazards
  - Extrusion: Feeding and consolidation of sensitive explosive ingredients brings with it impact and friction hazards, as the explosive is at the mercy of the machinery
  - Feeding raw materials: It can be difficult to keep tight control on multiple feed streams, causing variation in product composition/quality
- **Remedy**
  - Combining energetic solids with the polymer/other materials before extrusion to make molding powder effectively desensitizes the explosive, so that it may be processed in an easier, safer manner

Safety Considerations (cont)



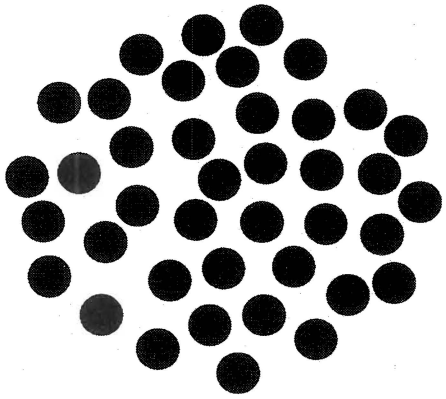
- The process material is only as safe as the least safe feed material

Safety Considerations (cont)

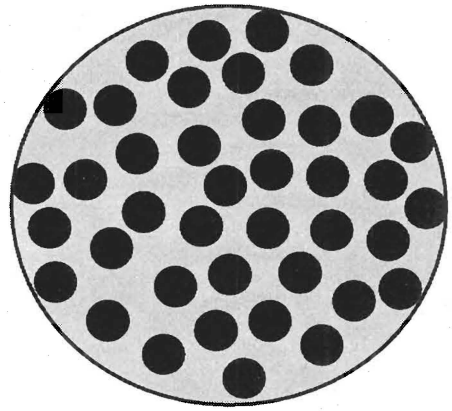


- Molding powder is less sensitive than the individual dry energetic solids because the polymer/plasticizer/other materials coat particles, desensitizing them to impact/ friction/ESD events

Safety Considerations (cont)



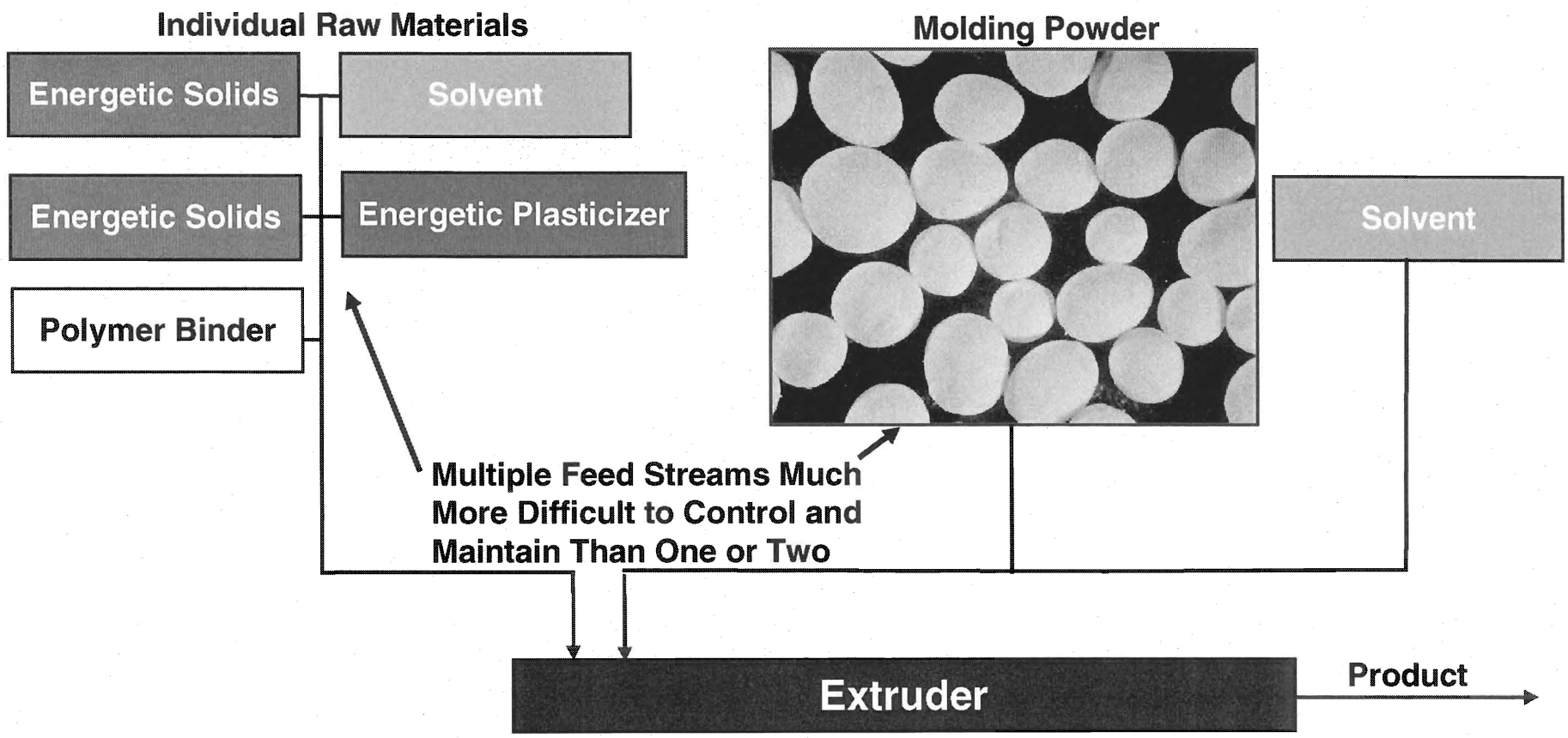
Nitramine

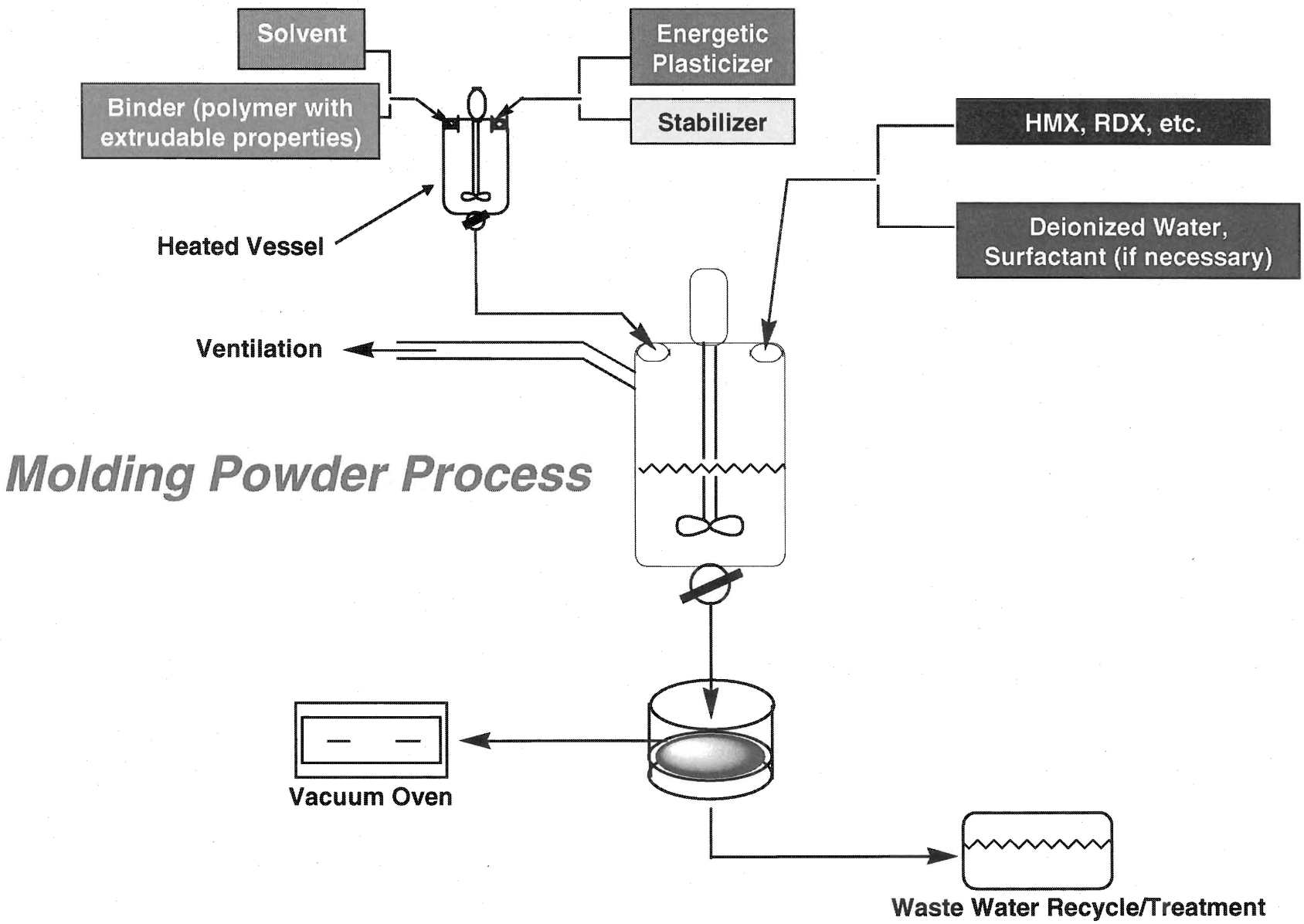


Nitramine (90%)/Binder-Plasticizer (10%) Molding Powder

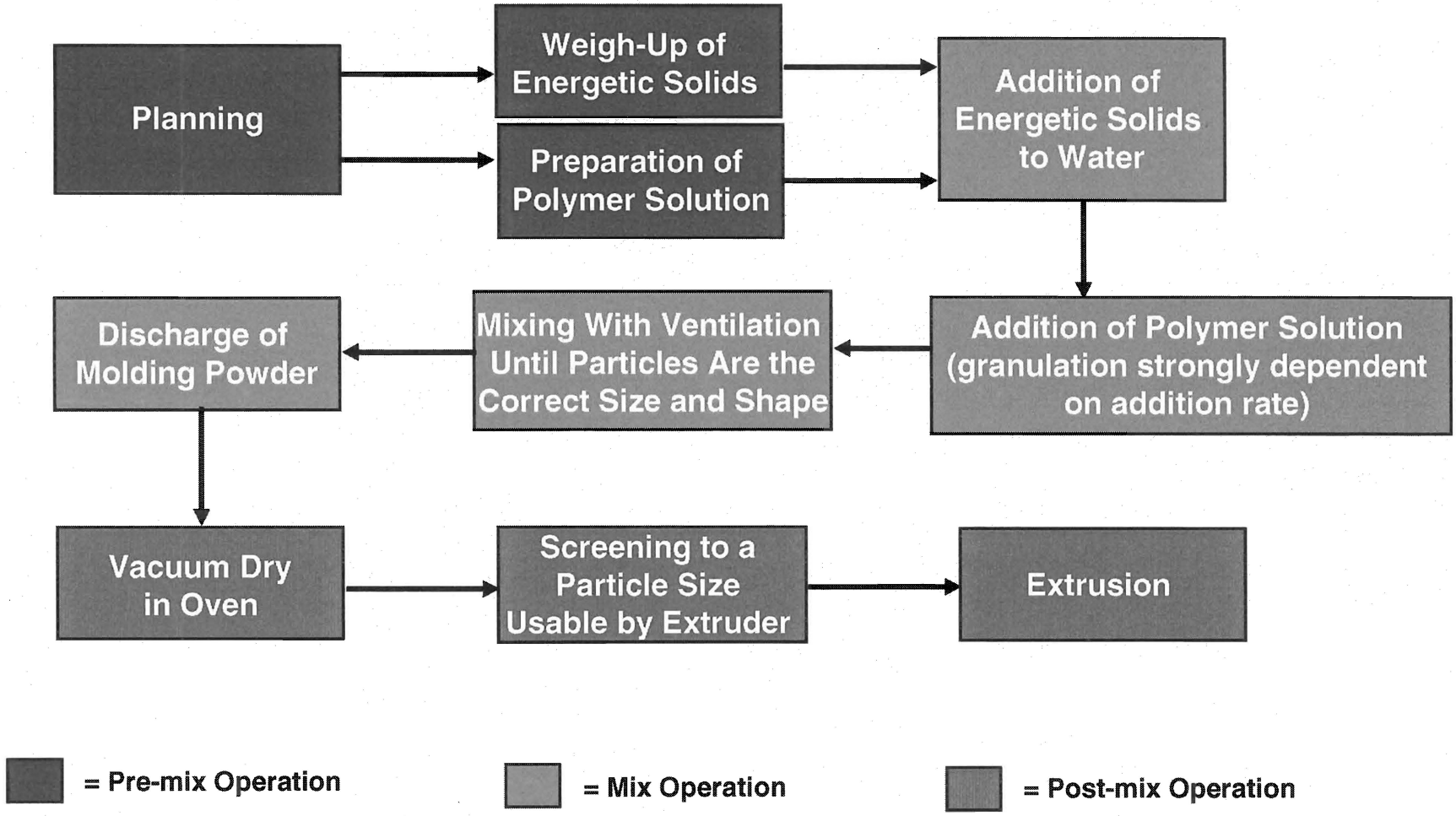
Material	ABL Impact (cm)	ABL Friction	TC ESD (J)	TC confined ESD (J)	TC Friction (lb)
Nitramine (100%)	1.1	50 lb at 2 ft/sec	1.26	0.05	18
Nitramine (90 %) Molding Powder	21	370 lb at 8 ft/sec	>8	8	>64

Process Control

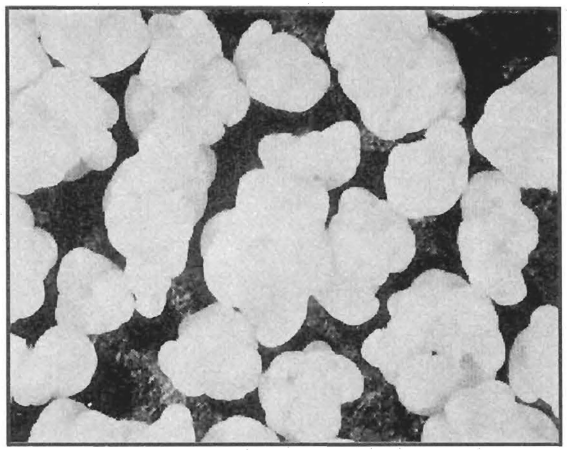



Solvent
Binder (polymer with extrudable properties)
Energetic Plasticizer
Stabilizer
HMX, RDX, etc.
Deionized Water, Surfactant (if necessary)
Heated Vessel
Ventilation
Molding Powder Process
Vacuum Oven
Waste Water Recycle/Treatment

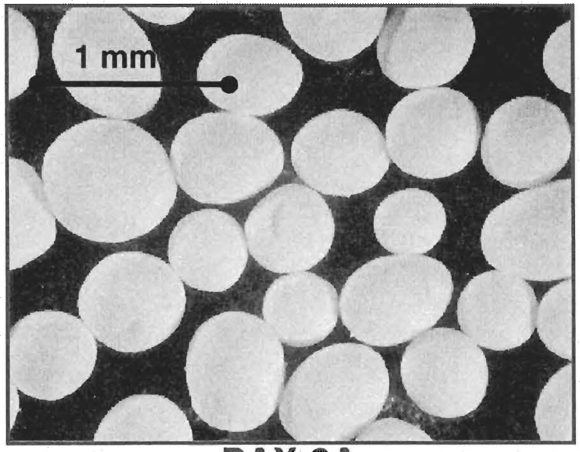
Molding Powder Process (cont)



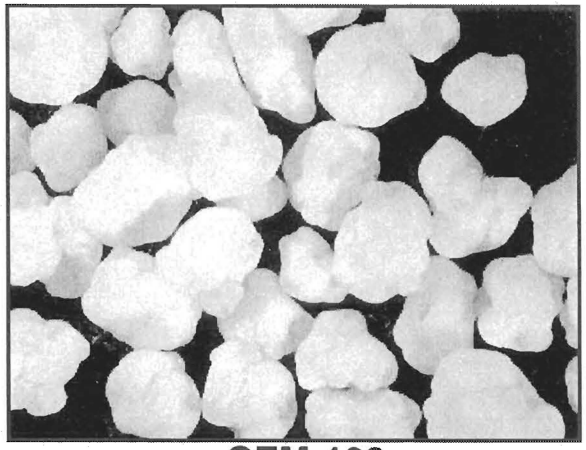
Below are several examples of molding powder. These are granules which have been made by coating explosive solids with a polymer binder in a water slurry reactor. Molding powder is used, among other things, as a feed for extrusion processing because of its safety properties, and because it greatly simplifies the extrusion process



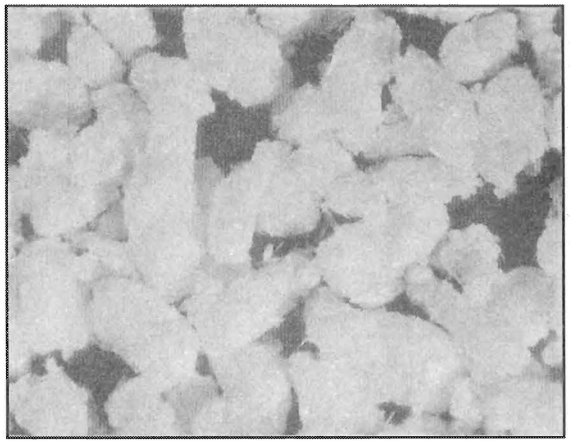
PAX-12



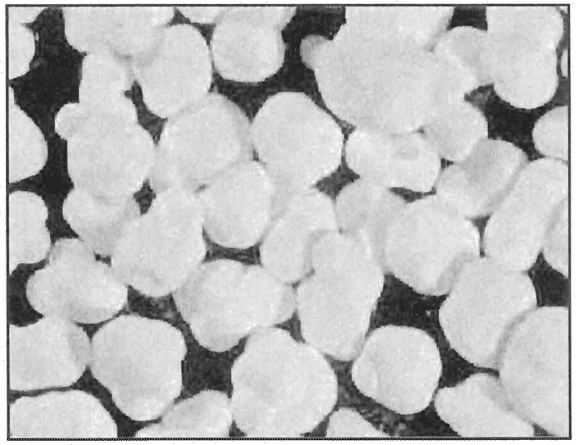
PAX-2A



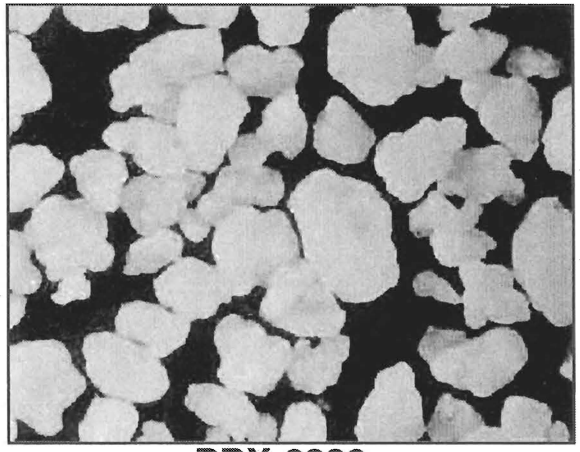
GEM-106



TGD-OO8



PBXN-5



PBX-0280

Summary

- **Molding powder is used as feed for extrusion in place of the individual ingredients for two reasons:**
  - Molding powder has much better safety properties than its energetic constituents alone
  - Molding powder consolidates all materials so that only one or two feed streams is necessary, making process control a much simpler matter
- **The molding powder process consists of:**
  - Dissolving the polymer (usually with heat) in a suitable solvent
  - Adding water, then nitramine, to the reactor while stirring
  - Adding polymer solution to the reactor with ventilation at a rate which forms particles of a consistent shape and size, and allowing particles to harden
  - Filtering off water
  - Drying material in a vacuum or convection oven
  - Screening to particle size usable in the extruder

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Injection Loading Technology :

An Automated Unit Operation for the Agile Manufacturing Facility

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CHALLENGE

- Navy policy mandates Insensitive Munitions
  - viscous thermosetting PBX loads for warheads
- Faster/Longer Range/Multiple Target/Smart Weapons
  - smaller payloads / submunition payloads
  - narrow flow channels for viscous IM energetics
- Economical Unit Costs with "Zero Defects"

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APPROACH

- NAVSEA IMAD/HE demonstrating I/L device
  - thermosetting castable PBXs (PBXN-107, PBXN-110, PBXC-304, etc.)
  - new PBXs (SMX-13, PBXIH-301, PBXIH-302, etc.)
- SERDP/CAME demonstration of pollution reduction
  - used computational tools to improve process design
  - used adaptive process control to sharply reduce BLU-97 reject rates
- NAVY MANTECH demonstration of economical process

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APPLICABILITY

- SUBMUNITIONS
- WARHEADS
- PROJECTILES
- EXPLOSIVE ROPE
- INITIATING SYSTEMS
- SMALL ROCKET MOTORS

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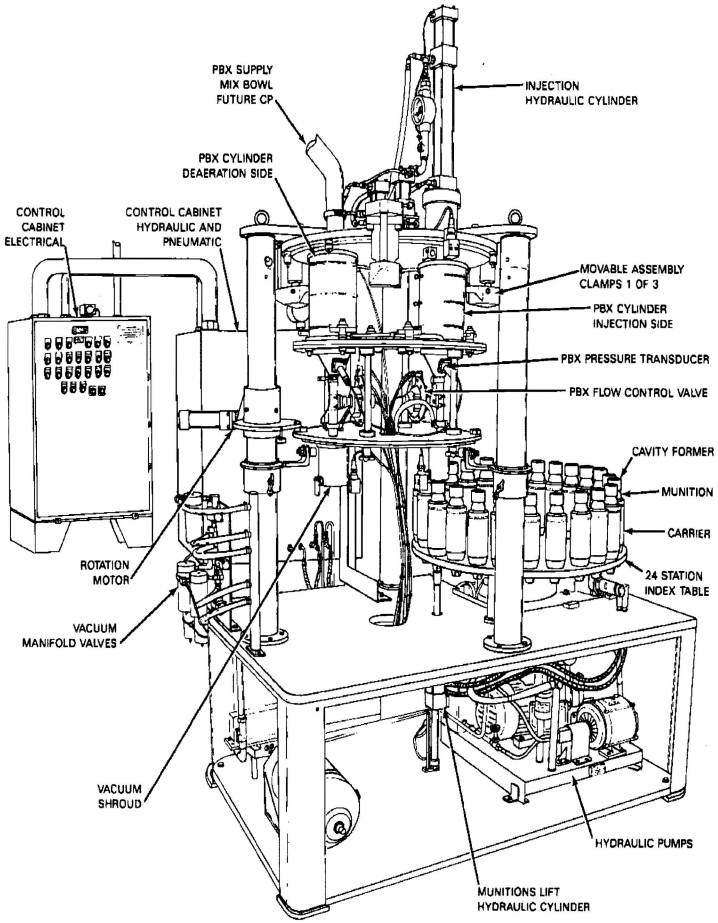

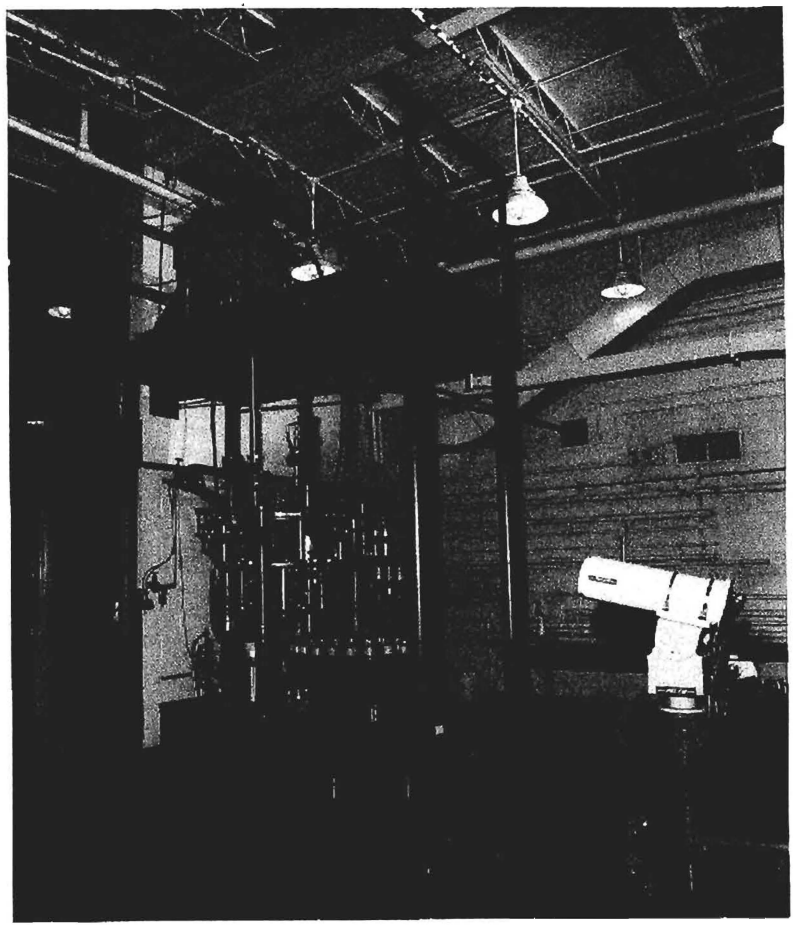

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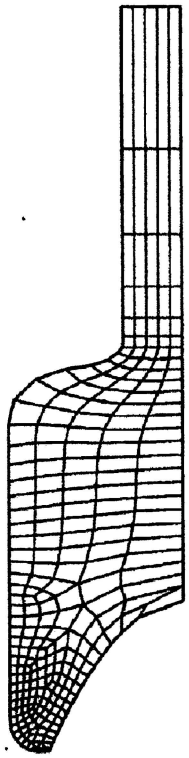

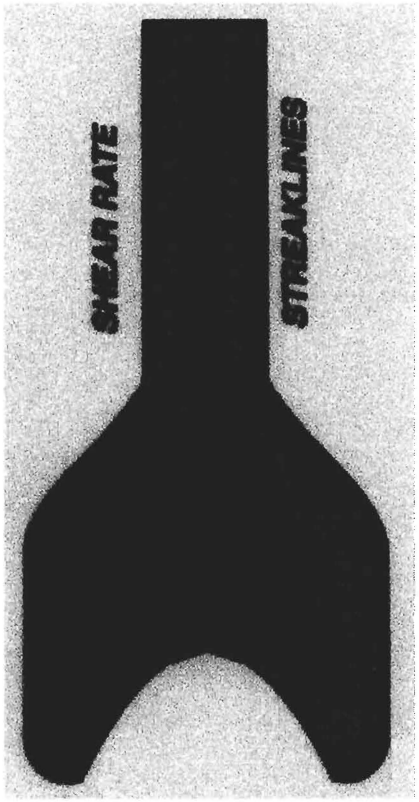

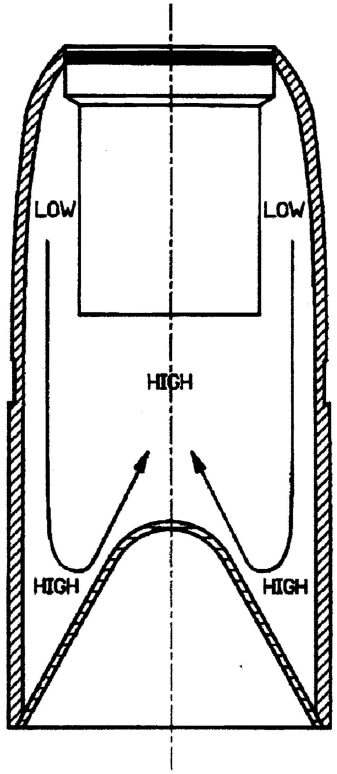

- Used data in FEM codes to determine the cause of density gradients in BLU-97 submunitions for JSOW
- Cooperation with SNL and LANL led to process design improvements that included the use of in-line static mixers for PBXN-107

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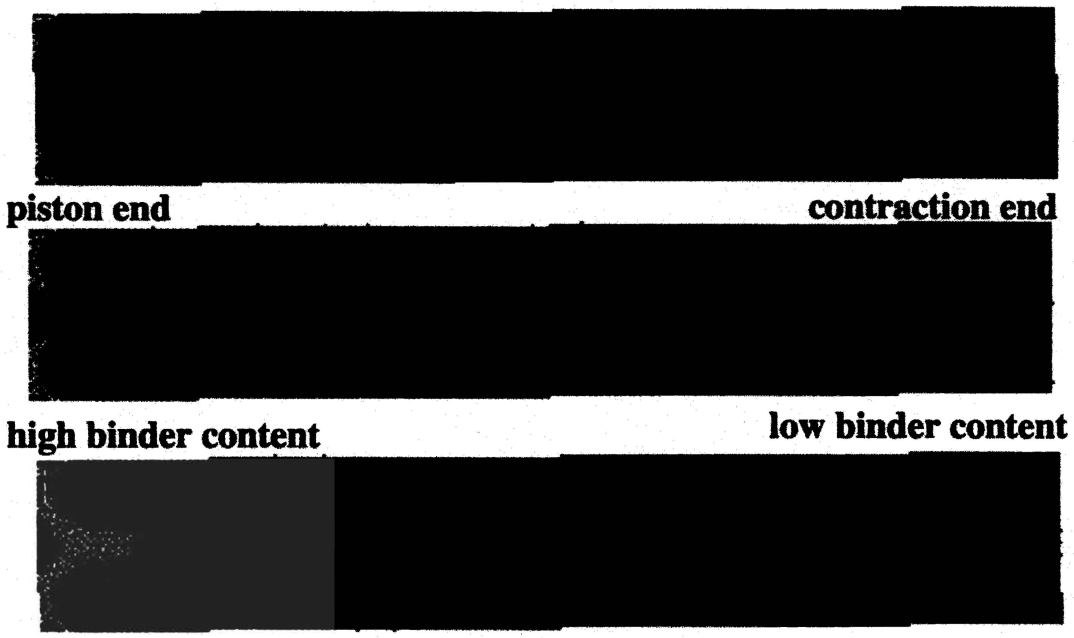

- Magnetic Resonance Imaging (MRI) techniques have been used to assess the demixing of PBX
- MRI experiments are useful for PBX selection and formulation decisions
- MRI experiments are useful for process design improvements such as changing the contraction geometry to limit the extent of PBX demixing

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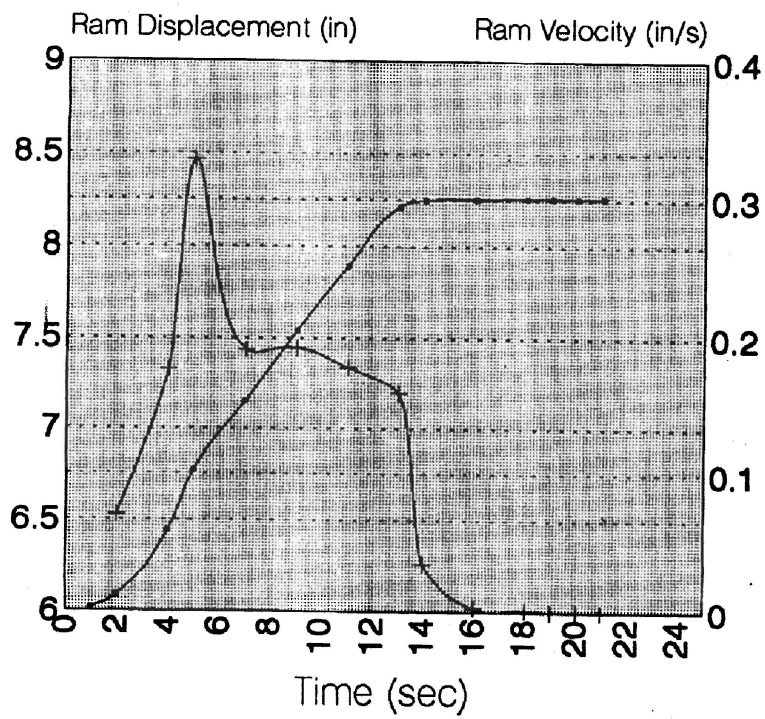

- After post-mortem X-ray inspection, we went back to our loading data and identified injection loading parameters necessary for producing properly filled and void free BLU-97 submunitions
- LANL and NSWCIHD developed adaptive process control algorithms for injection loading BLU-97 with PBXN- 107

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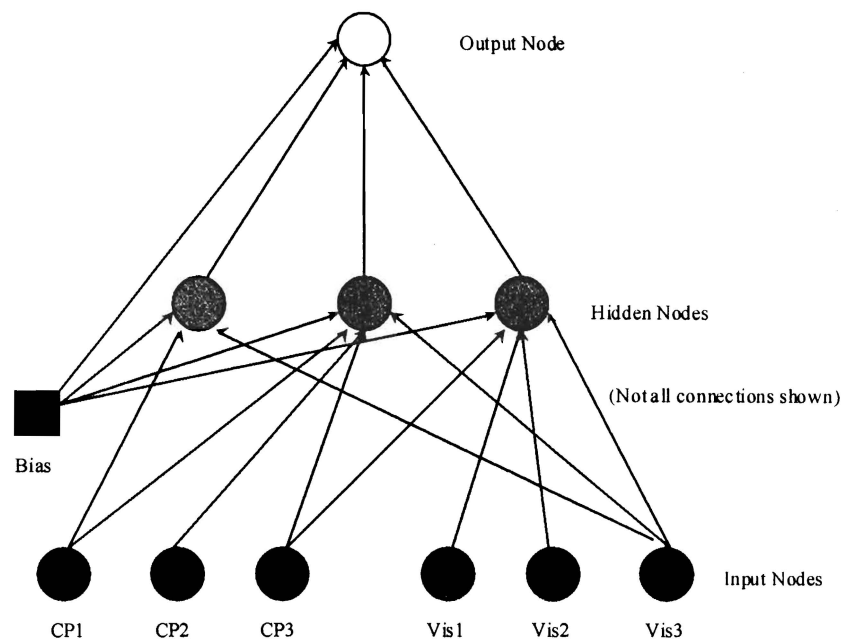
Chemical Engineering Principles

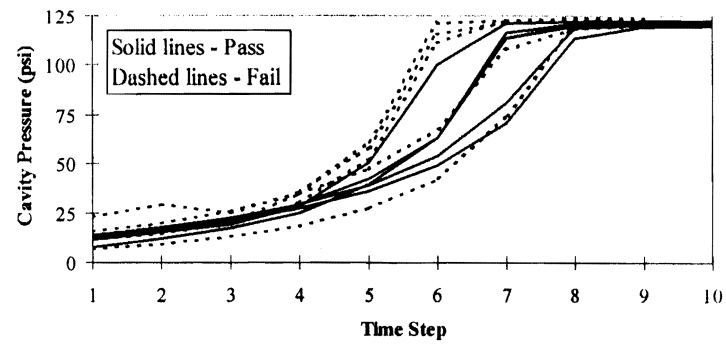
- Standard *backpropagation neural networks* were used by LANL to solve a pattern recognition problem for injection loading submunitions.
- The inputs to the network were the first N points of the time history data for one or more variables
- The output of the network was a single value in the range 0 to 1, with 0 representing a failed loading and 1 representing a passed loading. A value of 0.5 was used as a cut-off for the pass/fail choice.
- By analyzing the underlying equations relating the process variables and looking at correlations between the actual data, three significant variables emerge. These are cavity pressure (CP), ram velocity (RV), and viscosity (VIS).

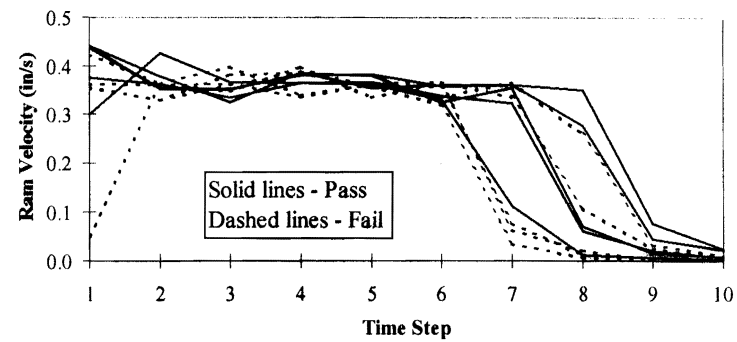


Neural network framework

Chemical Engineering Principles

- Initial attempts focused on using the entire time history of CP and RV.
- Later attempts focused on designing smaller networks that only looked at the first few time history data points
- The best networks had a 95% successful prediction rate using VIS, 90% using CP, and 75% using RV.
- Networks that used two of the significant variables were then tried. The networks using CP/RV and RV/VIS yielded 98% prediction rates while the CP/VIS network yielded a 100% prediction rate.
- The minimum number of hidden nodes that yield a 100% prediction rate was found to be three.
- The column labeled "Correlation" is a measure of how well the output of the network (a number between 0 and 1) correlated with the actual result (fail=0, pass=1).



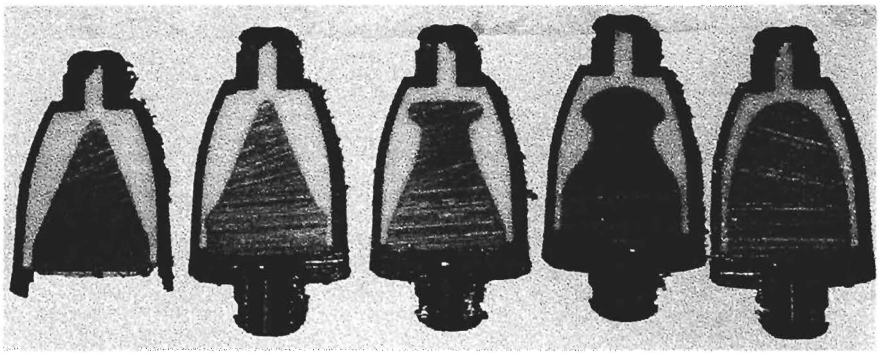


Results from CP/VIS neural networks of various sizes

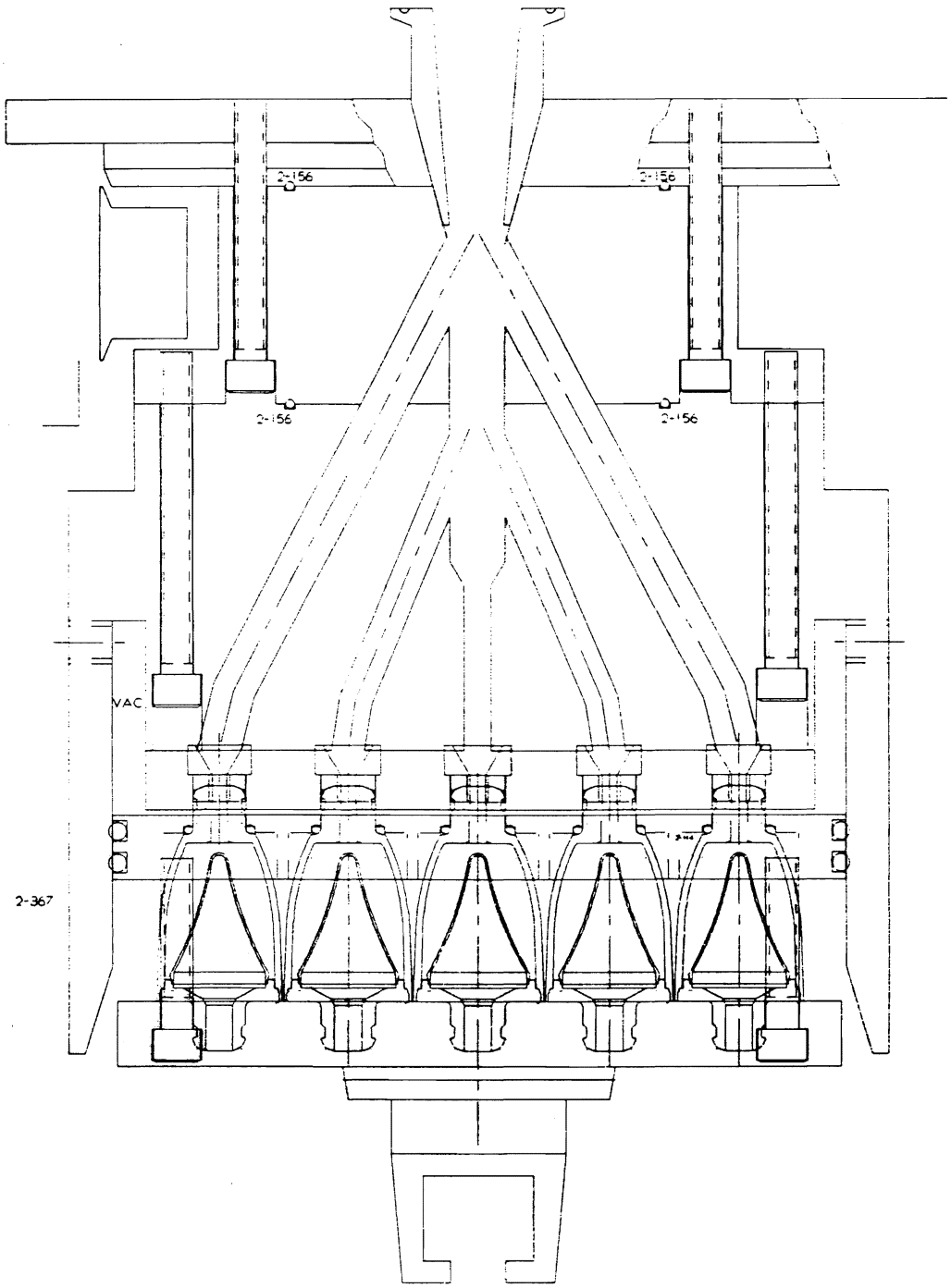
# Time Steps	# Hidden Nodes	Set	Correct	Total	Correlation
4	3	Training	34	34	0.973
		Test	11	11	0.927
		All	45	45	0.962
3	3	Training	33	34	0.917
		Test	10	11	0.793
		All	43	45	0.879
2	3	Training	33	34	0.844
		Test	10	11	0.929
		All	43	45	0.865

Potential Advantages of Injection Loading over Cast and Press Loading

- Load through small conduits (0.20 inch diameter)
- Load complex enclosed geometries
- Reduction of process steps, thereby reducing cost
- Potentially greater production rates



Longitudinal Sections of ESMB Submunitions with Various Liner Shapes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Roadmap for the Identification & Use of Computational Tools

NSWCIHD performs injection loading process design and process development tasks using empirical engineering methods.

↓

Resolution of process design and process development tasks require improved understanding before problems can be resolved.

↓

NSWCIHD utilizes computational fluid mechanics for process design modifications and mathematical models for adaptive process control algorithms.

→ ←

TTU performs experimentation to develop appropriate theoretical understanding of shear induced particle migration.

$$PBX\ composition = f(d\phi/dt) = f(a^2, \dot{\gamma}, \eta^{-1})$$

SNL performs experimentation to validate theory, and incorporates the data into fluid mechanics codes useful for process design.

$$PBX\ Flow = f(Q) = f(\dot{\gamma}, (a/R)^n) = f(\Delta P, \eta^{-1})$$

LMMS performs MRI experimentation in scaled models of NSWCIHD processes to validate SNL process design recommendations.

LANL performs numerical modeling of NSWCIHD process data to identify and recommend appropriate adaptive process control codes and techniques.

$$PBX\ load\ quality = f(d\phi/dt, Q) = f((d\rho/dt))$$

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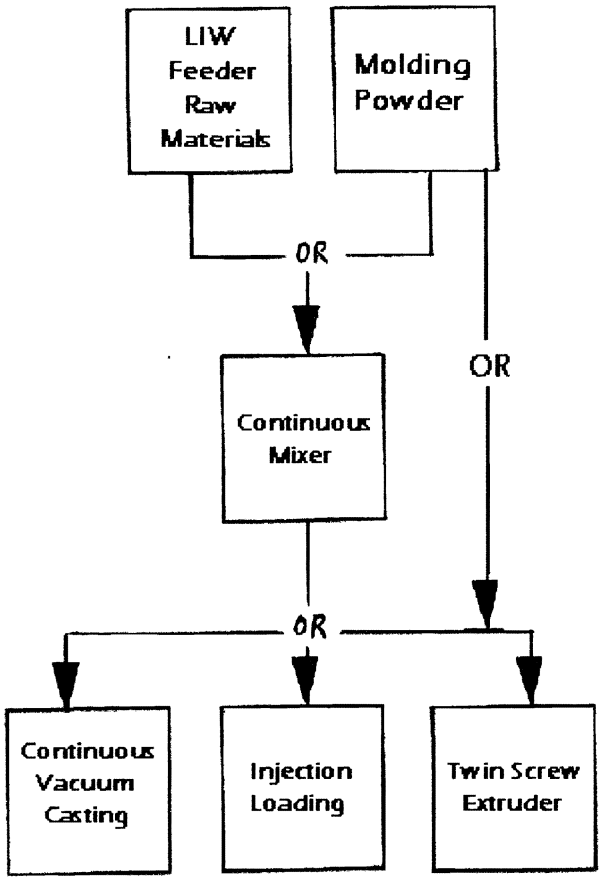
Agile Manufacturing includes Injection Loading



- Agile manufacturing facility for energetics could to be able to :

  [1] cast warheads with PBX, or

  [2] injection load submunitions with PBX, or

  [3] extrude composite rocket motors, or

  [4] produce gun propellant

- Agile manufacturing facility must reduce the traditional number of unit operations and have sufficient automation to be economical.

CONCLUSION

- I/L is a versatile unit operation
- I/L is automated and can use adaptive process control
  - perform momentum transport through narrow flow channels
  - reduce reject rates due to voids or mass transfer problems
- I/L is an economical processing technique
  - provides weapon designer more hardware fabrication options
  - features multiple loading per cycle