Truly Flexible Manufacturing Technologies for the Processing of Energetic Formulations*

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Conventional batch and continuous processing methods for energetics generally suffer from their lack of flexibility. Here a new truly-flexible continuous processing method is to be discussed. This single manufacturing platform allows differing degrees of mixing, pressurization, and conveying capabilities that can be tailored specifically for a given formulation upon selection from a library of drives, mixing/pressurization hardware, and with the configuration finalized using mathematical modeling.

** The full paper will be submitted to Journal of Energetic Materials*

I. Introduction: Continuous processing of energetics

The production of energetics in the United States has been largely confined to batch technologies. Interest in continuous production has been increasing (1), but this interest has been limited to development over the past several years. As technology has improved in many areas, a renewed interest in continuous processing of energetic materials has gained momentum in the U.S. Some of the major advantages to be derived from continuous processing are: higher production capacities, while maintaining lower in-process inventories; the economics of lower installation and operational costs; more uniform product quality, and lower personnel contact with hazardous materials.

Continuous processing technologies are also inherently safer than the batch processing methodologies because of the small amount of the energetic material found in the confines of the processor at any given time during the manufacture. For example, at the same production rate on a per hour basis, while a 1000 gal mixer would contain typically about 700 gallons of energetic material, the twin screw extrusion system would have only 10s of pounds within the confines of the extruder. Furthermore, the drag-based pressurization in the twin screw extruder allows for processing of extremely viscous mixtures without the potential for overburdening the drive, which typifies batch mixing. The ability to easily mix and convey highly filled mixtures leads to the utility of this equipment for increasing solids loading in composite explosives and propellants.

Continuous processing technologies are also more amenable to the control of the properties of the propellant since the surface to volume ratio of the continuous processor is orders of magnitude higher than the typical batch processors used in conventional processing, thus making it easier to achieve relatively homogeneous temperatures and degree of distributive and dispersive mixing during manufacture. Furthermore, continuous processing technologies like the twin screw extrusion process have the added benefit of flexibility since the screw and barrel sections are generally modular and interchangeable, thus providing a myriad of geometries for processing of energetics, with each screw and barrel configuration representing a different processor. Twin screw processors are so flexible that they have been used to process energetic materials with processing viscosities from 2,000 poise to 1,250,000 poise. This range of materials cannot be processed effectively in any single batch mixer.

However, the opportunities offered by the continuous processes like the twin screw extrusion process come at the price of necessitating extensive characterization and simulation tasks prior to the processing of the first pound of the propellant (2-33). Such characterization uses specialized techniques to characterize the rheological behavior, including the characterization of the viscoplasticity and the wall slip behavior of the energetic material, thermal properties, decomposition characteristics, the conditions under which the particles or the binder of the energetic suspension migrates or filters out, means to keep the air out, development of surface irregularities, analysis for the degree of mixedness of the ingredients as part of the characterization of the energetic formulation (2-33). This is especially necessary considering that unlike other industries, which also utilize continuous processors, trial-and-error procedures should not be used in the energetics industry. The wrong marriage of geometry, operating conditions and material properties will invariably result in an incident that is likely to generate significant losses.

Some of the preliminary characterization tasks which are necessary in the development of a continuous processing technology for the manufacture of energetic formulations include thermo-gravimetric analysis, TGA, and differential scanning calorimetry, DSC, analysis, the characterization of various material functions, including temperature and wall shear stress dependent shear viscosity, extrudate swell, flow instability and wall slip behavior of the energetic formulation, the simulation of the coupled flow and heat transfer occurring in the twin screw extruder, the design of the shaping geometry including the die, and validation experiments generally using thermal imaging and well-instrumented twin screw extruders. Such characterization and simulation tasks are essential to the safe processing of energetic materials, which can be accomplished without the use of precarious trial-and-error procedures.

II. Operation and Safety Features

Designing mixing equipment that answers the needs of today's energetic material producers requires an in-depth understanding of the safety and performance problems facing this industry. The hardware needs to be designed on the basis of various criteria that recognize the safety concerns without compromising performance. Here two extruders will be used as examples for illustrating the integration of safety into design and manufacture of continuous processing machinery. These two extruders are interesting because the first one (ME7.5) is, to our knowledge, the smallest twin screw extruder in the world, designed specifically for the incorporation and processing of nanoparticles in energetics formulations (5) and the second is the Universal, which is the most flexible manufacturing platform for energetic materials, with the ability to be converted into a single or twin screw, co-rotating or counter-rotating extruder (6). The intermesh between the two screws in the twin screw extruder mode can be altered to any degree of intermesh ranging from the tangential to the fully-intermeshing mode (Universal Mixer/Extruder). Furthermore, the barrel is splittable with a hydraulic mechanism which can sustain internal pressures of up to 5,000 psi and the sensor, feed, vacuum locations, as well as the total length of the extruder, can be altered to fit the processing task at hand.

Safety becomes even a greater concern with the necessity to process without organic solvents, using binders that are themselves energetic and with nanoparticles with their very high surface to volume ratios. The decomposition characteristics of some of the relatively new materials are also a cause for worry.

Design Development and Safety Issues

In general, the following summarizes how the extruders work and how the principal design of the geometries is made. Product ingredients enter the machine through various feed-ports. The product passes to the conveying screws, where it is carried from the drive end to the mixing section. A custom tailored agitator section (generally material specific and designed using mathematical modeling employing 3-D Finite Element Method) then alternates product mixing and thermal conditioning until the product is fully homogenized and reaches the desired temperature. The product then moves to the vent section for devolatilization. Next the mixture moves into the discharge section where pressure is developed to extrude the product through a die to develop a continuous geometric form.

These unique pieces of processing machinery, though based on twin screw polymer processing compounders, were specifically designed for use in the continuous manufacture of energetic materials. Therefore, they differ from polymer processing machinery commonly used in the marketplace today in a considerable number of ways. Aside from the ability to properly mix and extrude a variety of energetic materials, the prime consideration in design was, and is, safety. The energetics industry should not tolerate the use of trial-and-error procedures since as noted earlier any wrong marriage of the geometries, operating conditions and material properties will lead into an incident with possible significant loss of property. Thus, the ability to predict the coupled flow and heat transfer to occur in the extruder at the design stage introduces a significant advantage (7).

Mini twin screw extruder ME 7.5 for processing of nanoenergetics:

The ME7.5 (Figures 1, 2) was designed using mathematical modeling and was built initially for the distribution and coating of nanoparticles (5). The twin screws have a diameter of 7.5 mm (Figure 1), are co-rotating, fully intermeshing and self-wiping with an L/D of 15/1. The design is based on the mathematical modeling of the flow and heat transfer occurring in the extruder for the formulation to be processed and for which the screw configuration is developed and thus relies right from the beginning on the knowledge of the detailed thermo-mechanical history that the target formulation is to be exposed during processing. The entire processing operating condition space can be probed to determine if there are precarious conditions under which the energetic formulation will develop stagnant "dead" zones, hot spots, which will generate temperatures which are greater than the decomposition temperature of the energetic material, and pipe line flows which will give rise to the passage of the material through the extruder without intermixing with the rest of the ingredients.

The degree of fill in the extruder is also determined as a function of the possible operating conditions. This is especially crucial since for a given set of operating conditions the energetic material should not back-up into the feed hoppers or into the vacuum port. Thus, overall the mathematical modeling of the processing operation for a target formulation allows the design of the machine to be tailored and precarious geometry/operating condition combinations can be eliminated at the design stage. The ability of the extruder to process the given formulation is also fully assessed to recognize what the maximum and the minimum flow rates are to be and what the distributions of residence time, shear rate, pressure and temperature are to be in the twin screw extruder.

The ability to select the screw configuration on the basis of mathematical modeling of the process for a target formulation is again important, especially considering that most safety departments will not allow the utilization of segmented shafts and will insist on the use of solid screw agitators. Also for the 7.5 mm twin screw extruder shown in Figure 1 the screws are so small as to not permit the utilization of slip on elements and the entire screw needs to be machined in one piece. The utilization of the simulation technologies allows the designing of the proper screw configuration right from the beginning, thus providing not only safety of knowing to what thermo-mechanical history that the energetic material is to be exposed but also savings in cost and schedules by eliminating the trial and error necessary otherwise for the screw design. The ability to model a particular product in a twin screw also enables the researcher to explore optimum solvent conditions for both processing and safety. Selecting the lowest solvent concentration for safe processing reduces the equipment and energy required to dry the product.

It is also important to recognize that the thermo-mechanical history that the energetic material will be exposed to in the extruder will also depend on the geometry of the die to be used. The flow and temperature history in the extruder is directly affected by the presence of the die since the extruder will generate only the pressure necessary to overcome the pressure drop at the die. The pressurization rate and thus the degree of fill will be a function of the die used. Thus, the mathematical problem needs to be solved as a coupled flow and heat transfer problem covering both the extruder and the die. One of the die designs used for the mini is shown in Figure 2. This is a rectangular slit die designed to produce rectangular extrudates, which can then be used for the testing of the nanoenergetics grain. The design of the die is an integral part of the design of the extruder for the processing of the energetics formulations. The change in the shape of the flow channel in the die will generate a different extruded grain shape.

Universal Extrusion Platform:

The Universal extrusion platform (MU 40) is a multi-flex platform which can be operated as a single screw extruder, fully intermeshing co-rotating twin screw extruder, fully intermeshing counter rotating twin screw extruder, tangential co-rotating twin screw extruder, or tangential counter rotating twin screw extruder (Figure 3). One of the configurations involves twin screws with a nominal diameter of 40mm and the length of the machine is variable in 5/1 L/D sections up to 40/1 L/D maximum. However, depending on the nature of the application the extruder can be custom designed for other sizes also. The ability to determine conditions, which are precarious, and their weeding out at the design stage on the basis of the use of 3-D FEM solution of the coupled flow and heat transfer, is again one essential feature of the screw design and selection of the operating conditions for a given energetic formulation.

The specific requirement for the power capability can also be determined using mathematical modeling of the process for the target formulation. Closely allied with specific horsepower requirement is residence time of mixture within the process zone, for while total power calculations rely on screw speed, torque and available motor power. Typically residence times 30 seconds to 20 minutes are observed, dramatically improving safety over batch mixing operations.

Dispersive mixing implies the breaking apart and scattering of agglomerated materials, whether they are binder molecules or solid particles. Distributive mixing implies the even apportioning of each phase and ingredient of the final product. In the twin screw mixer, studies have indicated that appropriate mixing normally occurs within fifteen percent of the available processing area, the remainder of which becomes available for forwarding, shearing, deaeration and consolidating the mixture.

The ability to devolatilize and/or deaerate an energetic formulation is of considerable importance due to the degenerative effects of such entrapments on mechanical properties and sensitivity. Comparative studies show that because of the low volume to surface area ratio and the rapid exposure of fresh mixture to the vacuum source, deaeration and devolatilization of mixtures are more complete in this type of equipment than in batch type processors. This enhances the safety of the process, especially considering for example the possible adiabatic compression of the air to result in catastrophic losses.

Specific Safety Issues and Corresponding Design Features

Issue: Temperature control

One of the primary safety issues related to processing energetic materials is the requirement for precise control of the temperature of the energetic material. The most critical parameter for safely processing energetic materials is the ability to control the temperature of the product, both the apparent mass (or bulk temperature), as well as localized temperature due to shear energy, stress etc.

The twin screw mixer converts the mechanical energy of the rotating screws into heat through shear forces generated within carefully maintained clearances between the screws and between each screw and the barrel housing. The viscous energy dissipation is directly related to the deformation rate and the shear viscosity of the mixture (scales with the square of the deformation rate and directly proportional to the local shear viscosity). The shear viscosity of the mixture changes with increasing degree of mixedness of the ingredients of the formulation and the total specific energy input. As the shear viscosity of the energetic formulation being processed is also a strong function of temperature, it is essential to control temperature at various sections of the barrel. These sections include the feed zones, mixing zones, deaeration zone, discharge zone, and, when used, the extrusion zone and die head assembly.

The temperature of the energetic formulation should not be allowed to reach the decomposition temperature of the formulation. Since there is no way one can measure the formation of the hot spot in the extruder during the processing operation one needs to predict and eliminate conditions which lead to hot spot temperatures which reach the decomposition temperature of the formulation, following the simulation route outlined earlier. Modeling can accurately predict high temperatures and where they will occur. The highest temperatures occur in the midst of mix where thermocouples cannot measure the temperature. However, rpm, torque and temperatures measurement along the barrel and in the die can be used to determine if there is an excursion into conditions that may result in dangerously high temperatures.

In addition to optimizing the agitator profile for a specific process, these mixer designs incorporate the largest possible liquid media channels for the thermal conditioning of the process by providing the greatest possible surface area to volume ratio within the barrel envelope. Independent multiple zone controls and in process monitoring instrumentation are employed at strategic locations through out the length of the barrel and in the die. In addition to utilizing these sensors to monitor and control process temperature, they can be used for emergency equipment shut down and to trigger a high response rate deluge system for the equipment. IR and UV detectors can also be installed as part of the equipment system for triggering a deluge system.

Another important contributor to control of heat build up addressed is the ability to control horsepower. A specially designed variable speed and torque control hydrostatic drive affords the flexibility to finitely control process energy input. Finally, on the Universal extruder the torque on the two shafts can be determined directly and separately using slip ring technologies (6). The torque per shaft is the shear stress integrated over the entire surface area of the screw and thus is intimately related to the degree of fill in the extruder and the shear stress generated by the energetic suspension. Alarms as well as torque related automatic disengagement mechanisms are incorporated into the design of the Universal as additional safeguards.

Issue: Control of product pressure

Pressure control is another area of special interest especially because many materials require pumping through a die assembly under large consolidation forces. Also, pressure increase in regions such as the feed zone or near shaft seals indicates undesirable conditions. Closely related to the critical characteristic of process temperature control is the requirement to keep the product within desired pressure limits, especially in the extruder portion of the unit.

Pressure control is required to eliminate the possibility of inter-granular shear and to preclude seal leakage due to loading created by product upsets (i.e. die blockage, feeder or pump failures etc.).

In addressing this safety issue, these mixers feature computerized optimization of the discharge screws that ensure design extrusion pressures are met with a minimum 50% safety margin on degree of backfill of the screws. During processing pressure transducers monitor internal barrel pressure at critical points and will trigger shut down of the unit if preset safety limits are exceeded. As with the temperature sensors, the pressure sensors can be used to trigger a deluge system.

Issue: Electrical Discharge

The potential of ignition due to electrical discharge is another area of concern when processing potentially hazardous material such as energetics due to the electrical sensitivity of many of the formulations. In addressing this potential safety issue, the design of these systems is such that all electrical components in the mixer area are either fully explosion proof to Class I group D and Class II groups EF&G standards or are intrinsically safe as per the National Electric Code. By definition from the NEC, intrinsically-safe equipment and wiring shall not be capable of releasing sufficient electrical or thermal energy under normal or abnormal conditions to cause ignition of flammable or combustible atmospheric mixture in its most easily-ignitable concentration.

In the design of the two extrusion platforms hydrostatic drive systems were used, which puts the primary electrical power source outside of the mixing area in a non-hazardous environment. The torque pick-up device on the drive motor needs to be intrinsically safe, as are all the temperature and pressure transducers on the mixer proper. In our designs the hydrostatic power unit utilizes a special polyolesther synthetic fire retardant fluid that is classified as non-hazardous. The drive is designed to be unidirectional to prevent the inadvertent start up of the machine in the reverse direction.

Thermal conditioning for the hardware is accommodated via liquid media circulated through cooling channels in the barrels. The media conditioning systems are remotely located from the process in a non-hazardous area.

Additionally, the ME7.5 machine is small enough that the entire machine and the ancillary components supporting the process can be placed in a hood to optimize safety. A typical processing rate for this unit is around 10 to 100 grams per hour. The equipment is supplied with grounding lugs to dissipate static electric charge before it can build up on the machinery.

Issue: Metal-to-metal contact

Frictional ignition of the product due to dynamic metal contact is a concern for the processing of energetic materials. Experience has shown that the hydrodynamic film of the product will support the screws off of the barrel surface in most applications. However, under certain conditions, the film can break down and metal contact can occur (especially for formulations which lack elasticity as manifested by a relatively low first normal stress difference upon rheological characterization). When this happens, it is desirable to have the barrels and screws manufactured from materials that prevent adhesive wear. For example, for the ME 7.5 and the Universal, the screws and barrels of these units are manufactured from a through-hardened stainless alloy that is specifically designed to resist wear and corrosion. They are then treated with ferritic nitro carburizing that changes the surface of the metal for several thousands of an inch in depth, imparting a greater hardness to the metal at the surface and reducing its coefficient of friction. This reduces the possibility of any metal-to-metal pick up and greatly reduces the energy generation from any contact of the screws to the barrel bores.

Screw speed is a concern because of the possibility of reaching tip speeds exceeding the friction sensitivity of energetic oxidizers. Additionally, at a given torque and throughput rate, the localized heat transfer capacities of energetic binders and plasticizers may be process-self limiting factors. Because reliable prediction of such hazard levels is difficult, it is generally assumed best to operate at the lowest possible screw speed necessary to achieve the mixing and consolidation of phases required by mechanical property constraints. However, maintaining the shear required to maximize binder-filler interaction at any screw speed occurs when the mixture is processed at maximum torque. Consequently, at the design stage a hydrostatic drive unit is selected as the optimal drive for meeting these requirements.

The thrust bearing and seal assembly for the ME7.5 machine is manufactured from a carbon fiber reinforced electrically conductive polymer and precludes any metal contact with the agitator shafts at the drive end of the machine. The agitators are supported at the drive end by sealed anti-friction bearings, which are located outside of the seal area. The 40mm Universal has thrust bearings mounted in the gearbox, remote from the barrel assembly and are separated from the process material by seals and a considerable air gap. The agitators are supported on the drive end by anti-friction bearings mounted in the gearbox.

Issue: Maintenance

Another safety consideration is maintainability and good housekeeping characteristics of the processing machinery for energetics manufacture. In addressing the particular use for any equipment, it is obvious that cleanliness is an essential part of the safety consideration. In addition safe maintainability at the design stage has to be considered a high priority as it is evident that personnel will be required to clean and disassemble the unit after shutdown from live material runs, or if a system fault occurs.

Addressing this issue, the following design features were incorporated into our designs. In the fully-intermeshing mode, the agitators in the mixing and extrusion area are fully conjugal; that is, an arc on one agitator fully describes the surface of the mating agitator through a 360-degree revolution. Hence, the agitators completely wipe each other and the barrel bores within the clearances prescribed. From a process standpoint, this assures that the surfaces are completely regenerated of product, but possibly as important, it means that the machine is internally self cleaning, and will purge itself of product when run after the purge feeders are shut off. There should be no stagnation areas in the machine, orifice plugs and other devices sometimes used in polymer processing to restrict flow are avoided in the design of extruders for energetics manufacture to ensure that no voids or pockets are present which could entrap material in the unit.

The mini extruder designed for the processing of nanoenergetics, ME7.5, features a horizontally split barrel and die assembly, which is held together by quick release clamps. The barrel and die assembly also separate vertically for ease of cleaning. The barrel separates from the drive assembly via the same quick release clamps. The agitators can be removed from the machine by the use of a release mechanism that does not require the use of tools (Figure 1). The drive sleeve which holds the agitator shafts in the machine is slid back after it is released outside of the process area via a spring loaded detent system. When this sleeve is moved, it releases the agitator shafts from the radial and thrust bearing assembly and they can then be removed from the machine. This process can be accomplished without the need for special tools in the product area.

The 40mm Universal features barrel sections that are both horizontally and vertically split (Figure 3). The barrel can be separated at the horizontal split line and the entire upper barrel section opened via the hydraulic power unit for ease of cleaning and maintenance. This also allows for easy access to change out and re-configure the agitator assemblies. The barrel sections are electrolysis nickel-plated on the exterior surfaces to aid in cleaning and to prevent corrosion of the components.

Additionally, the ability to access the equipment via the split barrels on both designs affords the operators to perform dead stops. This is a process that allows for important observations and data gathering when the machinery is stopped and disassembled during steady state operation. This information can be used to validate the predictions of a model if one is being used. Typically, when running inert formulations, this allows the operator to peer into the process zone in search of otherwise difficult to determine information, such as stagnation, metal-to-metal contact points, leakage paths, and percent fill of various portions of the process zones. This information can also be used during development to verify data predetermined from the 3D computer models.

To eliminate cracks and crevices, the ME7.5 agitators are a monolithic design. They are precision tooled from solid bar stock, thereby yielding better torsional and bending strengths versus segmented designs for these small diameters. The solid agitators are designed and custom built for each application via computer modeling.

While slip-on elements are highly suited for testing a wide variety of formulations, the equipment designed to process a given energetic mixture need not offer such flexibility, because once process parameters are established for a steady state operation, no adjustments should be necessary. Furthermore a fixed agitator configuration may be used for mixtures requiring different treatments through control of such variables as temperature, screw speed, torque and residence time. This conclusion is based, not only on mathematical modeling, but also on data gathered by observation when dead stops are performed during operation.

Issue: Process control and Data Acquisition

Acquisition of process data and control of the process as well as safety parameters are critical features of the continuous processing equipment design. Both extrusion platforms are operated via a state-of-the art open architecture PC based system that includes full instrumentation to monitor and control zone temperatures and screw speed, monitor product temperatures, process pressures and screw torque. The software allows remote operation of the units as well as remote data collection; either wireless or via the Internet. All of the safety features are programmed with automatic shut down for over temperature, over pressure or over torque of the system. Mechanical safety back up for over torque in the form of a quick response, high flow relief valve is an integral part of the hydrostatic drive system on both units.

III. Conclusions: The prime design consideration for the development of extrusion technologies for processing of energetic formulations is safety. All the features mentioned in this manuscript lend themselves to the safe application and operation of extrusion equipment for these types of applications. However, as indicated, the geometry and the operating conditions need to be selected for a given energetic formulation. The initial characterizations of rheological and thermal properties are essential for the mathematical modeling of the process to understand the thermo-mechanical history that the energetic material will be exposed to during the processing stage.

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V. References:

1. Fair, D., "Application of science based manufacturing to continuous processing of energetic materials", submitted to Journal of Energetic Materials (2006).

2. Kalyon, D., "Overview of the rheological behavior and characterization of energetic formulations: Ramifications on safety and product quality", Journal of Energetic Materials, 24, 3, 213- 246 (2006).

3. Kalyon, D., H. Gevgilili, J. Kowalczyk, S. Prickett and C. Murphy, "Use of adjustable-gap on-line and off-line slit rheometers for the characterization of the wall slip and shear viscosity behavior of energetic formulations", Journal of Energetic Materials, 24, 3, 175-194 (2006).

4. Kalyon, D., H. S. Tang and B. Karuv, "Squeeze flow rheometry for rheological characterization of energetic formulations", Journal of Energetic Materials, 24, 3 195- 212 (2006).

5. S. Ozkan, H. Gevgilili, D. Kalyon, J. Kowalczyk and M. Mezger, "Twin screw extrusion of nano-alumina based simulants of energetic formulations involving gel based binders", submitted to Journal of Energetic Materials, (2006).

6. http://www.mprus.com

7. M. Malik, H. Gevgilili, D. Kalyon, M. Fair and D. Fair, "Model based selection of operating conditions for twin screw extrusion based processing of PAX III formulation", submitted to Journal of Energetic Materials (2006).

8. D. Kalyon, "Apparent Slip and Viscoplasticity of Concentrated Suspensions", J. Rheology, 49, 3, 621-640 (2005).

9. M. Malik and D. Kalyon, "Three-dimensional Finite Element Simulation of Processing of Generalized Newtonian Fluids in Counter-rotating and Tangential Twin Screw Extruder and Die Combination", Int. Polym. Processing, 20, 398- 409 (2005).

10. M. Erol and D. Kalyon, "Assessment of the Degree of Mixedness of Filled Polymers: Effects of Processing Histories in Batch Mixer and Co-Rotating and Counter-rotating Twin Screw Extruders", International Polymer Processing, 20, 228-237 (2005).

11. D. Kalyon, D. Dalwadi, M. Erol, E. Birinci and C. Tsenoglu "Rheological Behavior of Concentrated Suspensions as affected by the Dynamics of the Mixing Process", Rheologica Acta, On-line, 1435 (2005).

12. Allende M. and D. Kalyon, "Assessment of Particle-Migration Effects in Pressure-Driven Viscometric Flows", J. Rheol. 44, 79-90 (2000).

13. Aral B. and D. M. Kalyon, "Effects of Temperature and Surface Roughness on Time-Dependent Development of Wall Slip in Torsional Flow of Concentrated Suspensions," J. Rheol. 38, 957-972 (1994).

14. Aral B. and D. M. Kalyon, "Viscoelastic Material Functions of Noncolloidal Suspensions with Spherical Particles," J. Rheol. 41, 599-620 (1997).

15. Fiske, T., S. Railkar and D. M. Kalyon, "Effects of Segregation on the Packing of Spherical and Non-Spherical Particles," Powder Technology, 81 57-64 (1994).

16. Gevgilili, H. and D.M. Kalyon, "Step Strain Flow: Wall Slip and other Error Sources", J. Rheology, 45, 2, 467-475 (2001).

17. Kalyon, D. M., Yazici, R., Jacob, C., Aral, B. and Sinton, S. W., 1991a, "Effects of Air Entrainment on the Rheology of Concentrated Suspensions during Continuous Processing," *Polym. Eng. Sci., 31,* 1386-1392 .

18. Kalyon, D. M., Jacob, C. and Yaras, P., 1991b, "An Experimental Study of the Degree of Fill and Melt Densification in Fully-intermeshing, Co-rotating Twin Screw Extruders," *Plastics, Rubber and Composites Processing and Applications, 16* (3), 193-200.

19. Kalyon, D., P. Yaras, B. Aral and U. Yilmazer, "Rheological Behavior of Concentrated Suspensions: A Solid Rocket Fuel Simulant," J. Rheol. 37, 35-53 (1993).

20. Kalyon, D., "Review of Factors Affecting the Continuous Processing and Manufacturability of Highly Filled Suspensions," Journal of Materials Processing and Manufacturing Science, 2 159-187 (1993).

21. Kalyon, D. and H. S. Gokturk, "Adjustable Gap Rheometer," U.S. Patent # 5,277,058 (1994).

22. Kalyon, D. "Highly Filled Materials: Understanding the Generic Behavior of Highly Filled Materials Leads to Manufacturing Gains and New Technologies," CHEMTECH, 25, 22-30 (1995).

23. Kalyon, D. M., H. Gokturk and I. Boz "An Adjustable Gap In-Line Rheometer," SPE ANTEC Technical Papers, 43, 2283-2288 (1997).

24. Kalyon, D., A. Lawal, R. Yazici, P. Yaras and S. Railkar, "Mathematical Modeling and Experimental Studies of Twin Screw Extrusion of Filled Polymers", Polym. Eng. Sci. 39, 1139-1151 (1999).

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

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

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

28. Tang, H. and D. Kalyon, "Estimation of the parameters of Hershel-Bulkley fluid under wall slip using a combination of capillary and squeeze flow viscometers", Rheol. Acta, 43, 80-88 (2004).

29. Yaras, P., D. M. Kalyon, and U. Yilmazer, "Flow Instabilities in Capillary Flow of Concentrated Suspensions," Rheologica Acta, 33, 48-59 (1994).

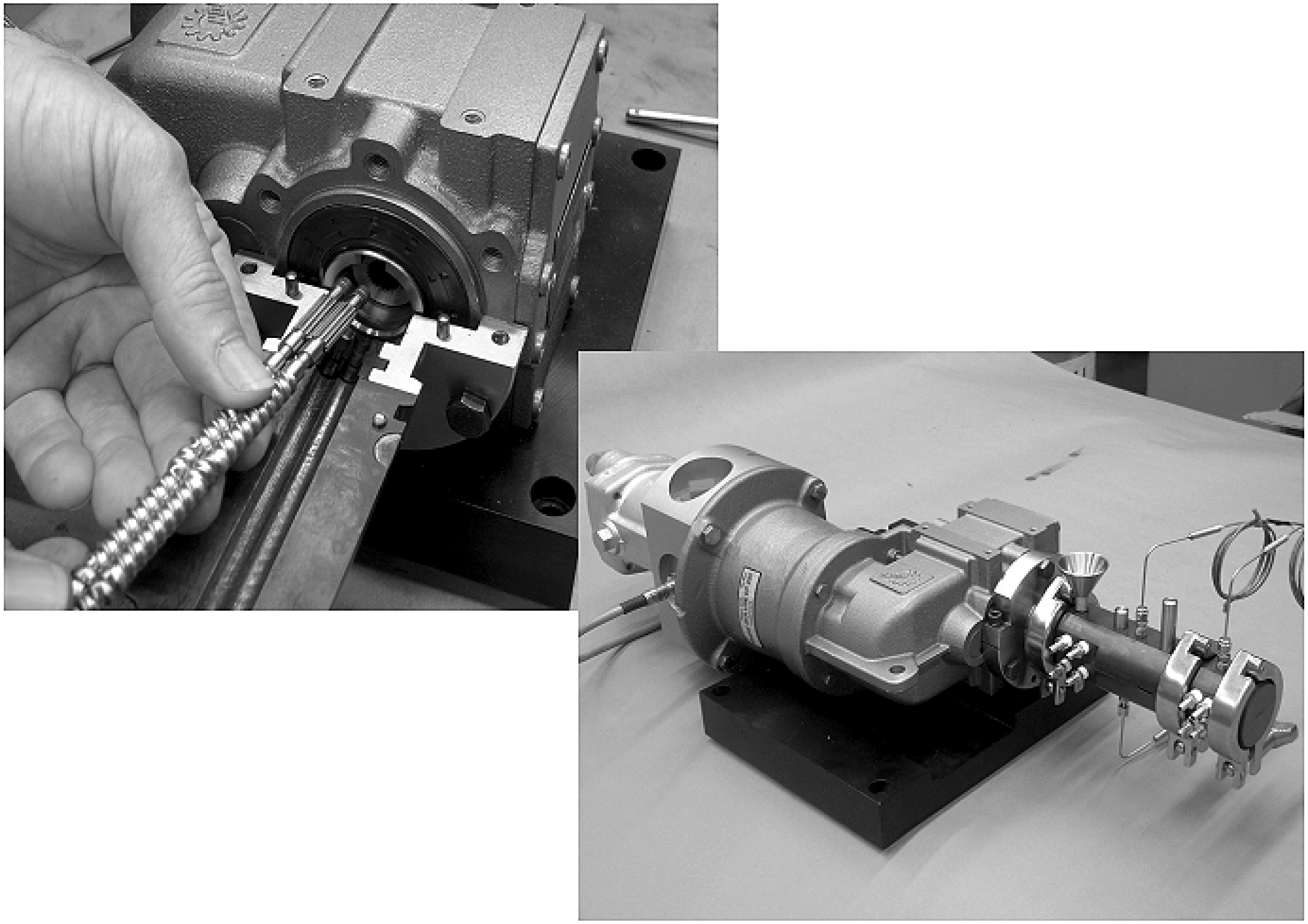
30. Yaras, P. "Rheological behavior and twin screw extrusion flow o non-colloidal concentrated suspensions", PhD Thesis, Stevens Institute of Technology, Hoboken, NJ (1996).

31. Yilmazer U., C. Gogos and D. M. Kalyon, "Mat Formation and Unstable Flows of Highly Filled Suspensions in Capillaries and Continuous Processors," Polymer Composites, 10, 242-248 (1989a).

32. Yilmazer U. and D. M. Kalyon, "Slip Effects in Capillary and Parallel Disk Torsional Flows of Highly Filled Suspensions," J. Rheol. 33, 1197-1212 (1989b).

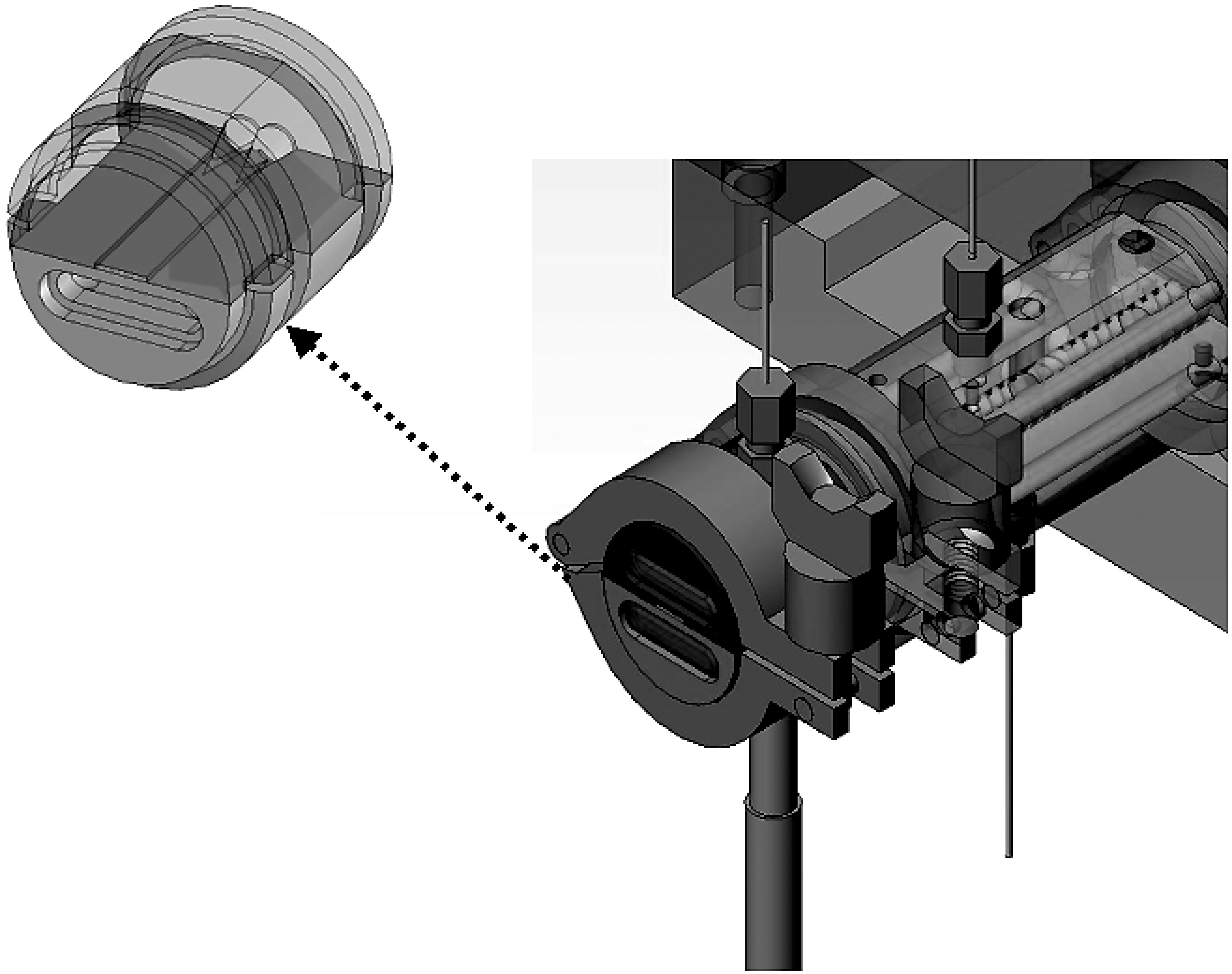
33. Yilmazer U., and D. M. Kalyon, "Dilatancy of Concentrated Suspensions with Newtonian Matrices," Polymer Composites, 12, 226-232 (1991).

List of figures:



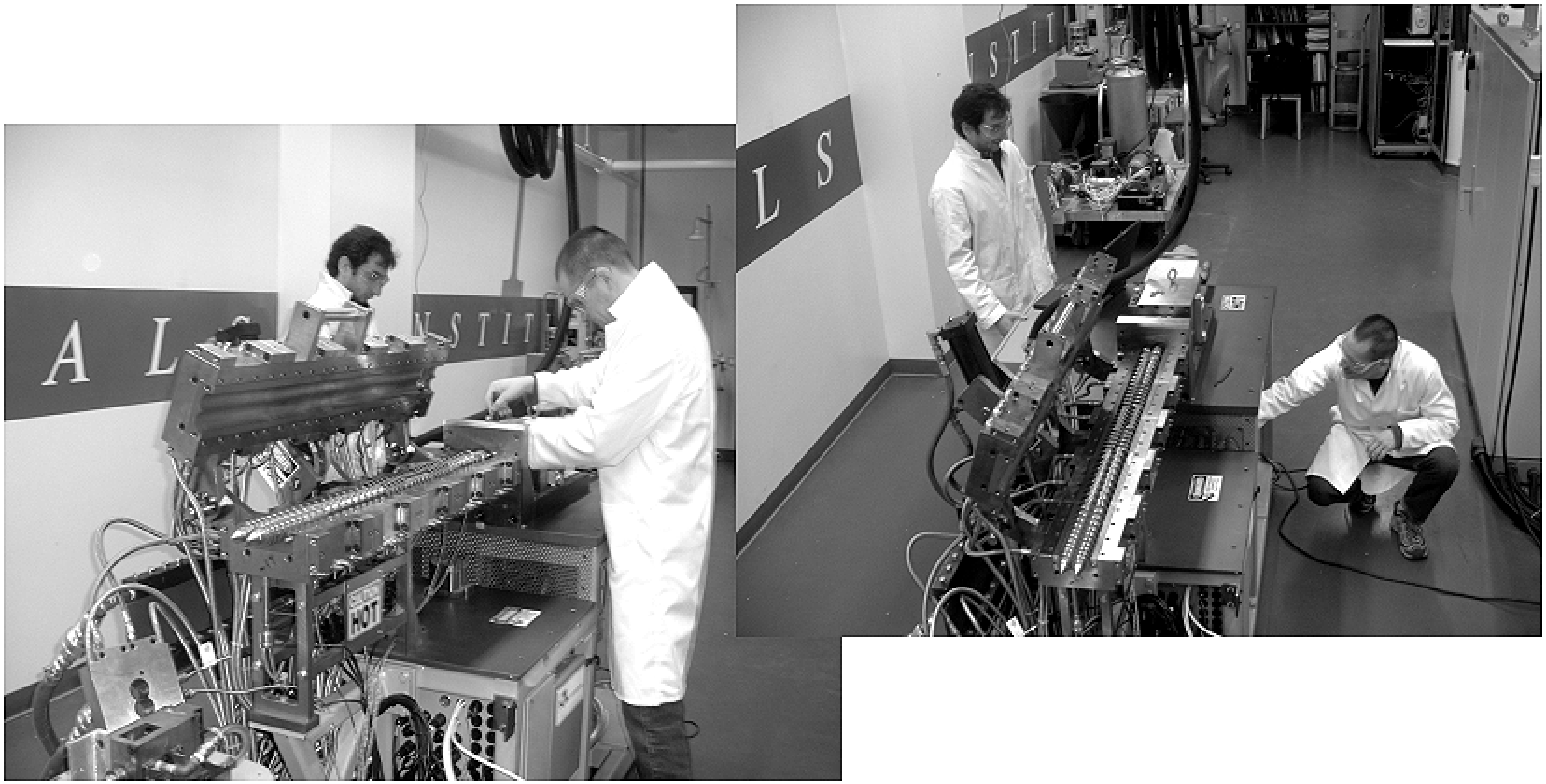
Mini extruder specifically designed for processing of nanoenergetics

Figure 1



Slit die of the mini twin screw extruder

Figure 2



Universal extrusion platform at the counter-rotating mode

Figure 3