“An Ideal Mixing/Processing Tool for the Dispersion of Nano Particles and Formulation Studies”

James E. Kowalczyk*, MoinuddinMalik*

Dilhan Kalyon, Halil Gevgilili**, Cenker Demir**, Mark Mezger*** and Brett Reddingius*****

*Material Processing & Research, Inc.

**Stevens Institute of Technology HfMI

*** US Army ARDEC

14th JOCG Continuous Mixer and Extruder Users Group Meeting

Oct 11-12, 2006

Special challenge:

The ability to produce articles, including energetic grains, from nanometal powders (5-100 nm range) by encapsulating them into various binders including energetic binders

- **Need to conserve particle size**
- **Need to select proper wetting agents/binders**
- **Need to tailor the process to the rheological behavior of the dispersion**
- **Need to work at a relatively very small processing rate (some new materials cost as much as $50,000/lb, furthermore the safety issues with some of these materials is currently ambiguous (high cancer rates in mice with nano particles)**
- **Need to generate very small specimens**

This challenge stems from the difficulty of handling the nanometal particles

- **without introducing significant oxide layers on one hand, and**
- **without allowing the nano-particles to agglomerate into larger particles, on the other hand**

We have designed and built a remotely controllable mini-twin screw extruder (smallest in the World)

- **We envision this extruder to be first directly incorporated into a bench-scale facility for wet-chemistry-based manufacturing and encapsulation of nano-particles.**
- **Research to support the objectives of the Army After Next (AAN) for highly lethal, more energetic, less sensitive explosives for use in smaller, lighter warheads for anti-armor and missile applications and their transition to the Army industrial Base.**

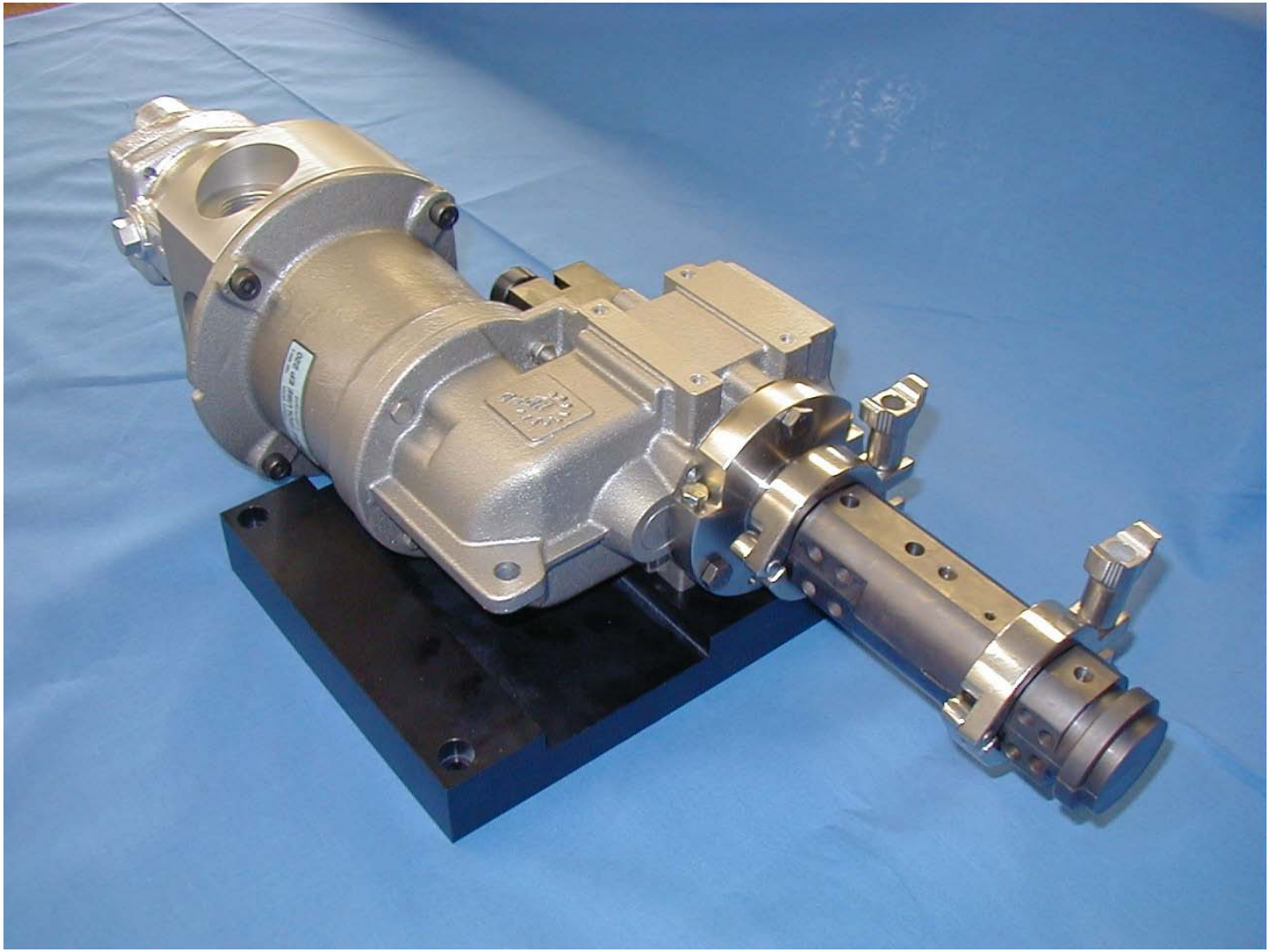


Figure 5

Fully-intermeshing corotating Twin Screw Extruder

- **Integrated slit die (dimensions: width of 10 mm, a gap of 1 mm and the length is 50.8 mm including the converging flow section and 36.8 mm long for the straight land section.**
- **Two temperature control zones, one at the die and the second for the extruder**

Figure 6

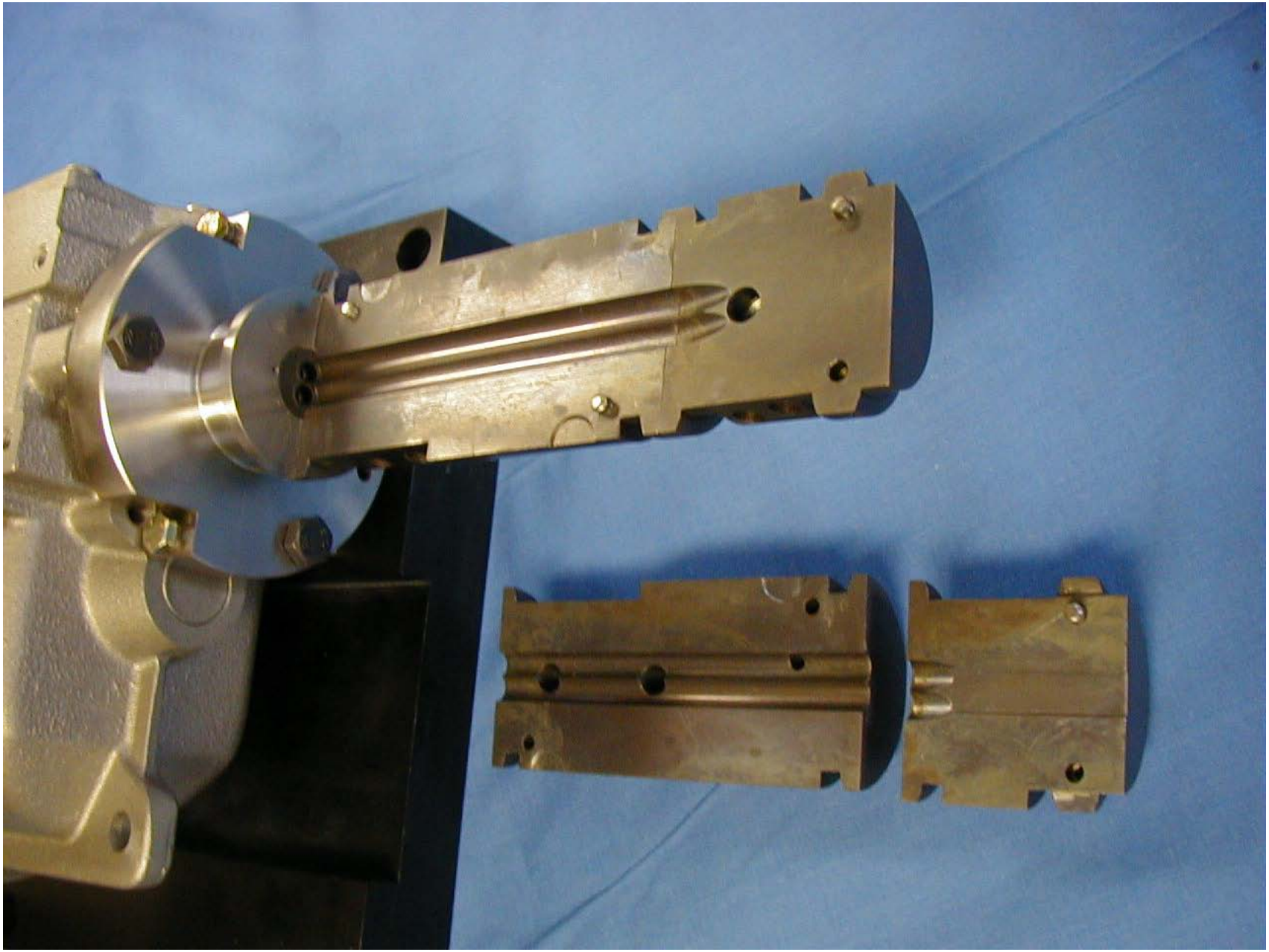
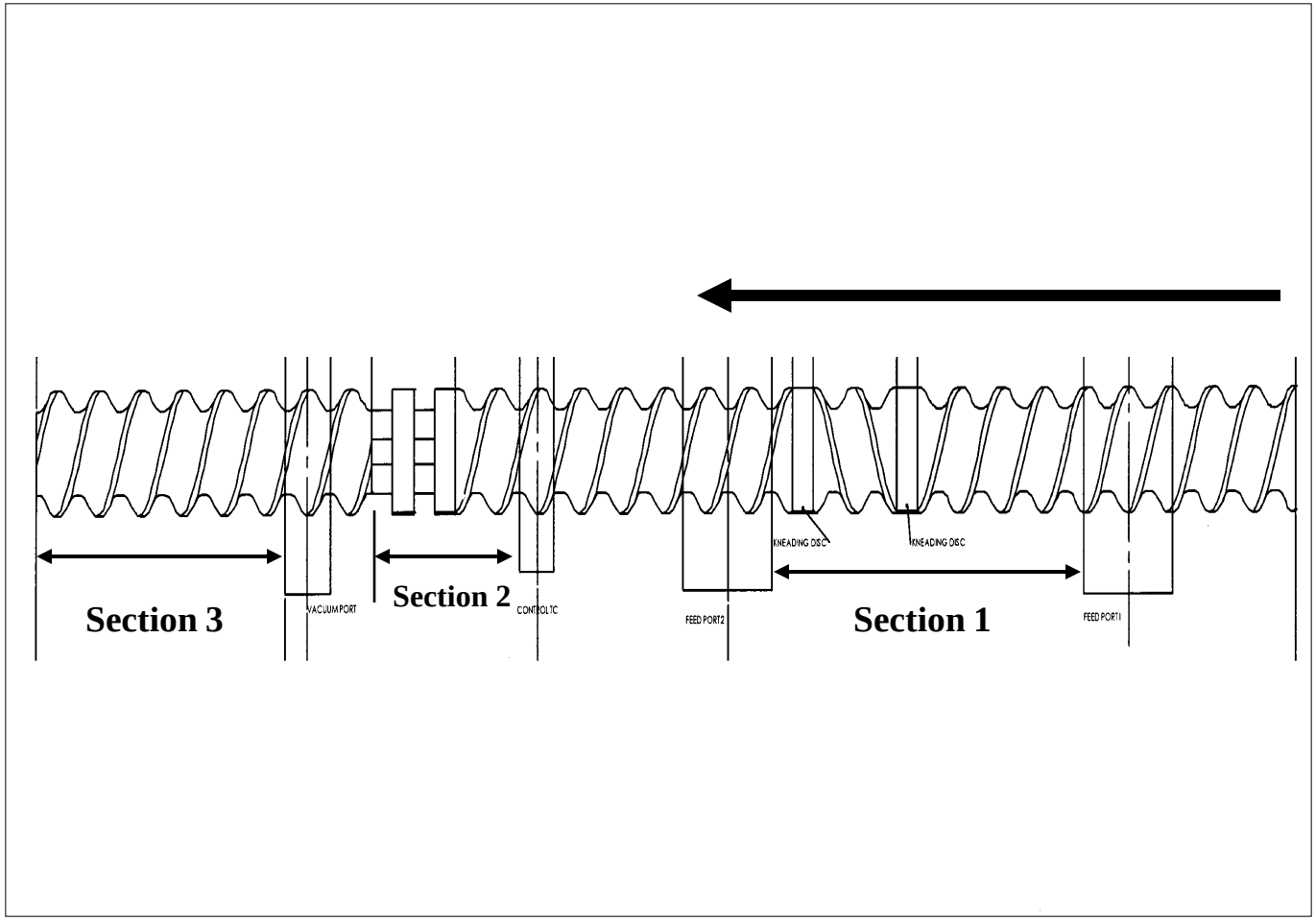


Figure 7

Solid screws

- Three sealed mixing and vacuum zones
  - Fully-flighted conveying/plasticization section (Section I) sealed with a reverse fully-flighted element
  - Mixing of the nanoparticles into the matrix polymer (Section II) sealed with four pairs of 90 degree kneading disks.
  - Imposition of vacuum and devolatilization in between the die and the kneading disks (Section III)

Figure 8



Screw configuration designed for the extruder

Figure 9

Design through mathematical modeling of the extruder and die

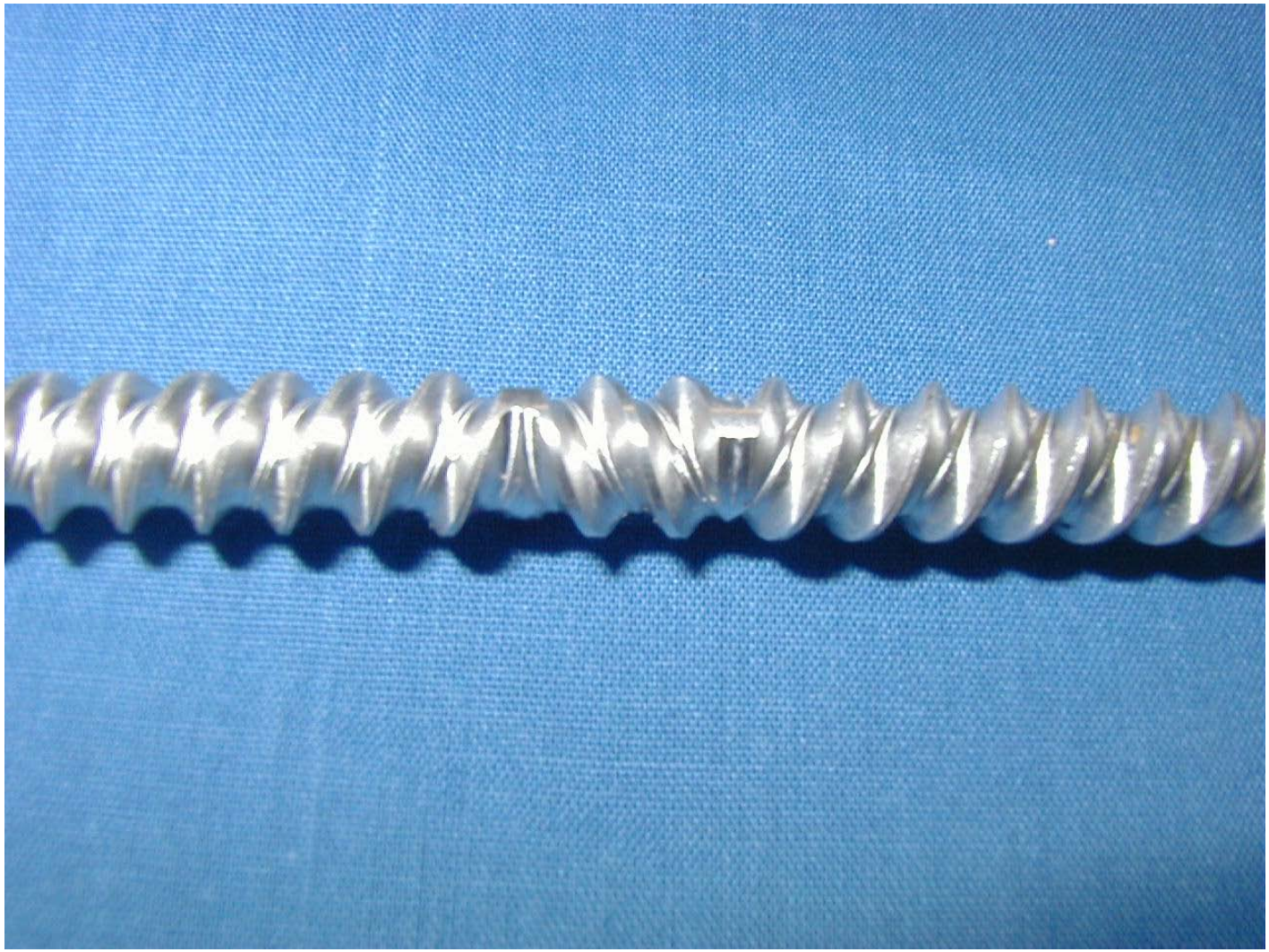
- **Use the rheological behavior of a typical ETPE propellant of interest to the Army (considered as a fast/slow burn gun propellant strand material), Hershel-Bulkley type generalized Newtonian fluid.**
- **Design on the basis of solution of the conservation equations with 3-D FEM.**

Figure 10

Mathematical modeling was utilized to design the three sections of the extruder and the die upon solution of the conservation equations for mass, momentum and energy conservation.

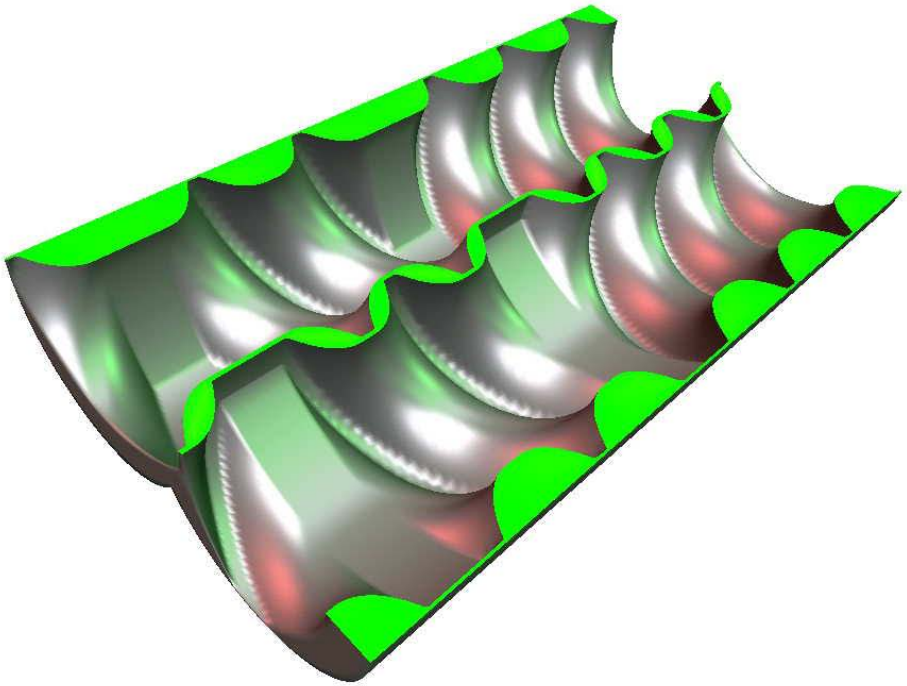
Figure 11

Design of section I



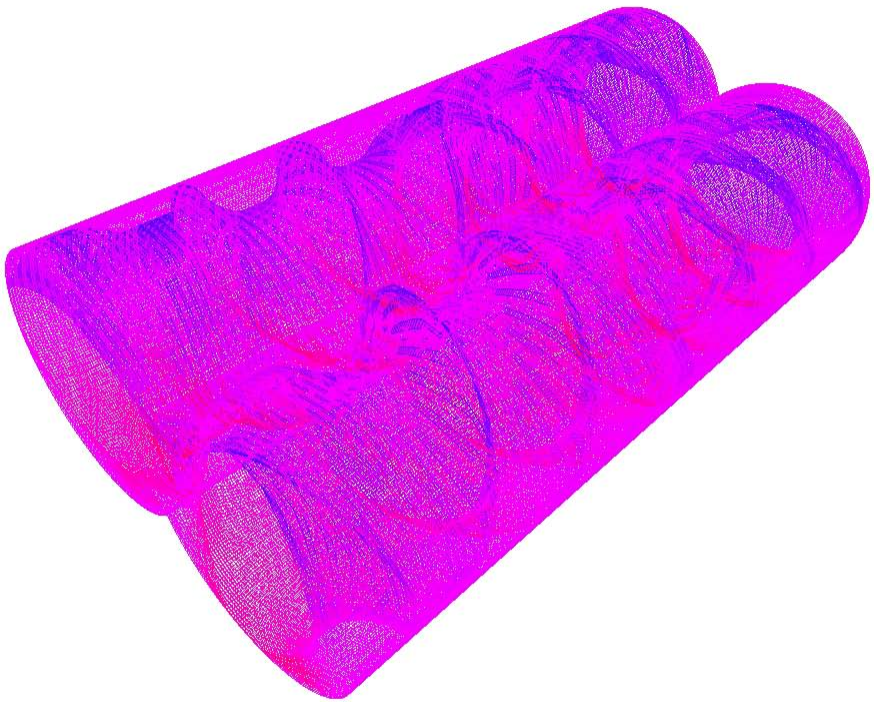
Reversing Feed Screw Section

Figure 13



Mini Extruder First Section Flow Channel Cutaway

Figure 14

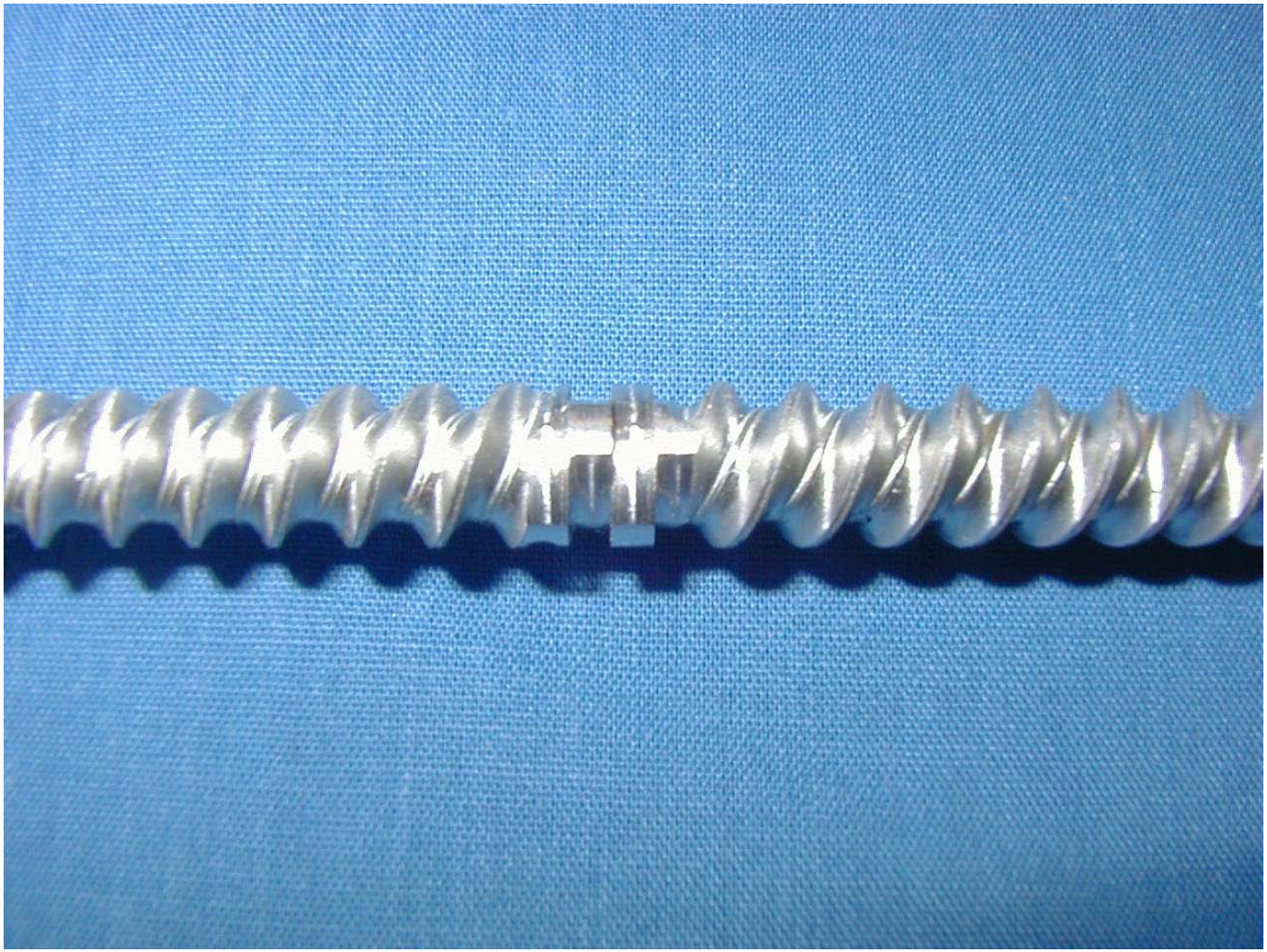


Mini Extruder First Section Flow Channel: FEM Mesh

Figure 15

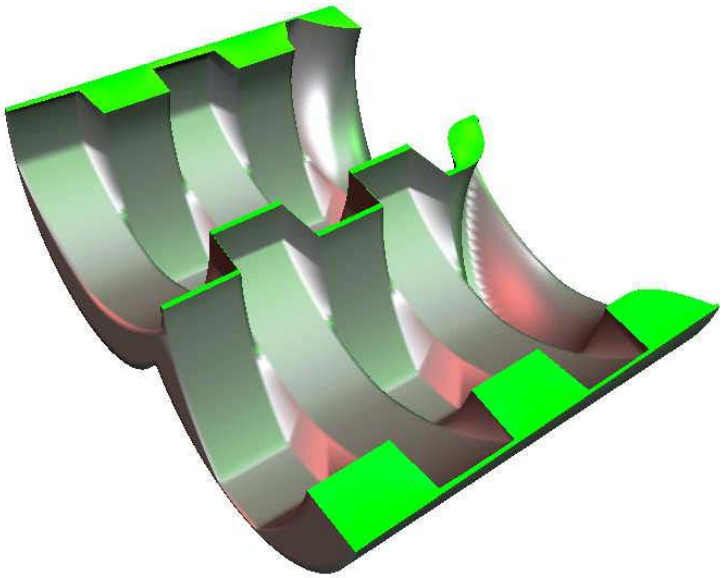
Mini TSE Section 2 Design

Figure 16



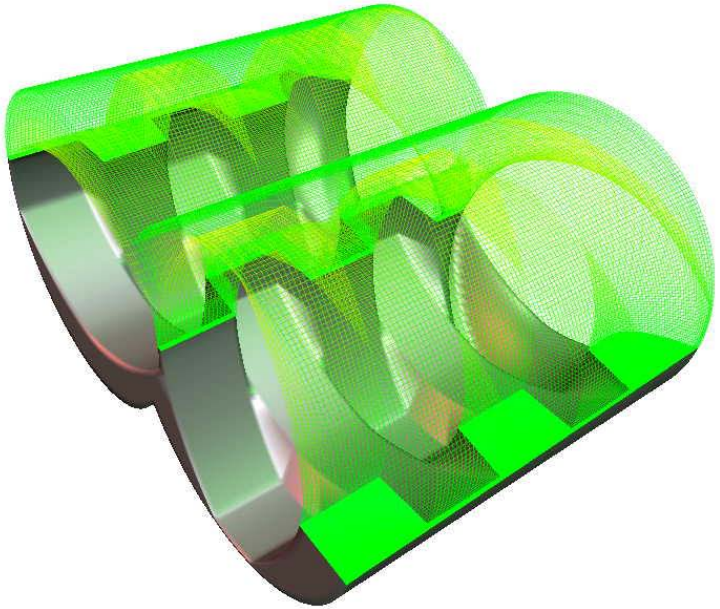
Paddle Block Section

Figure 17



Mini Extruder Second Section Flow Channel Cutaway

Figure 18



Mini Extruder Second Section Flow Channel Transparent Model

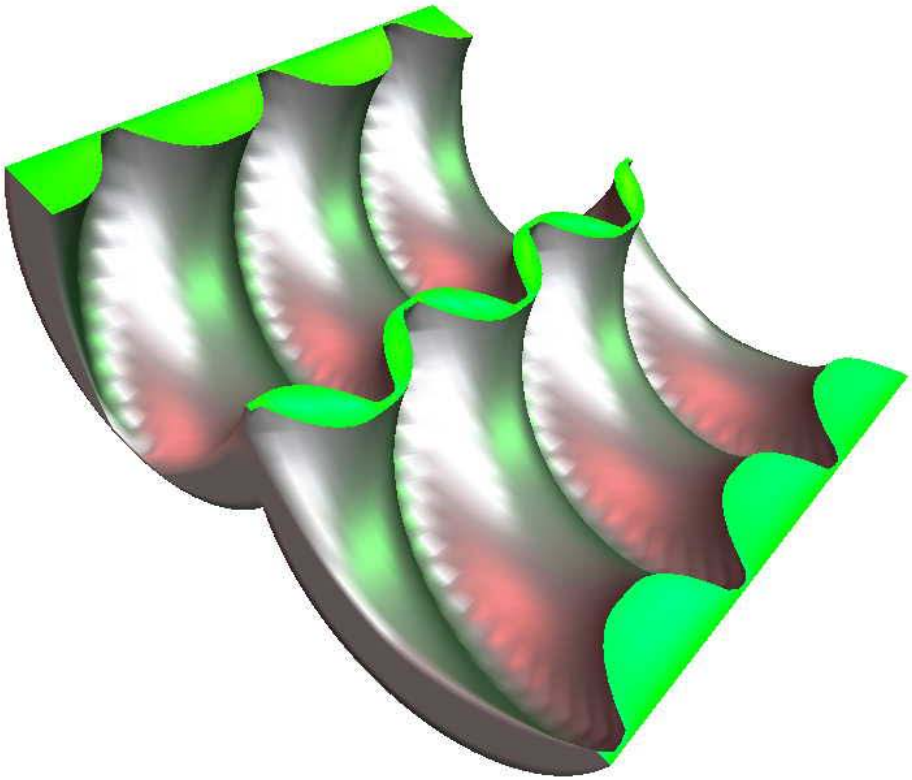
Figure 19

Mini TSE Section 3 Design

Figure 20

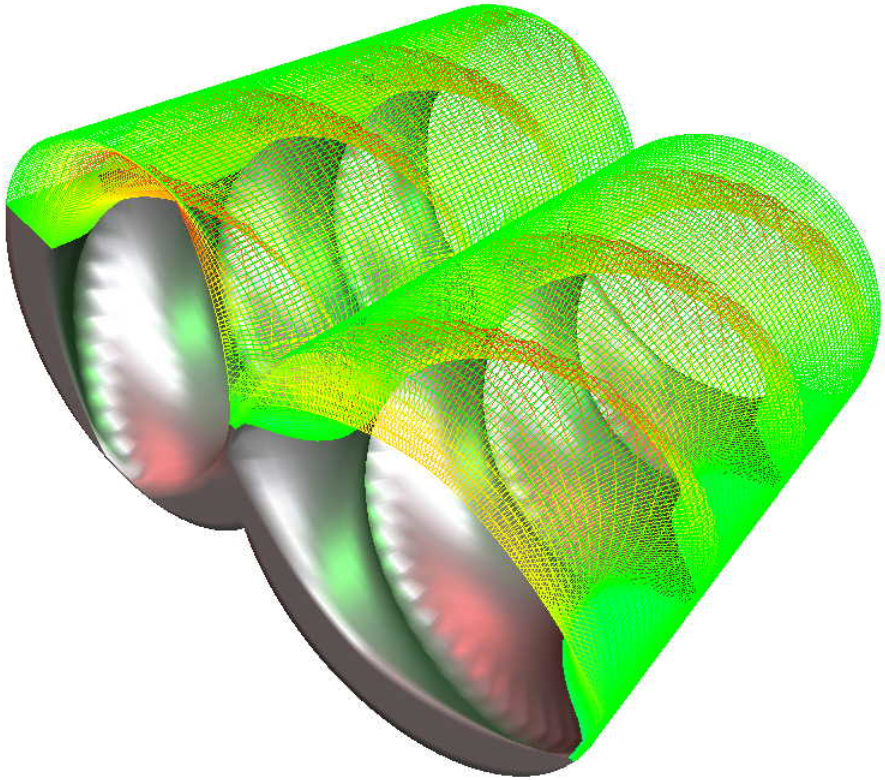
Extruder Screw Section

Figure 21



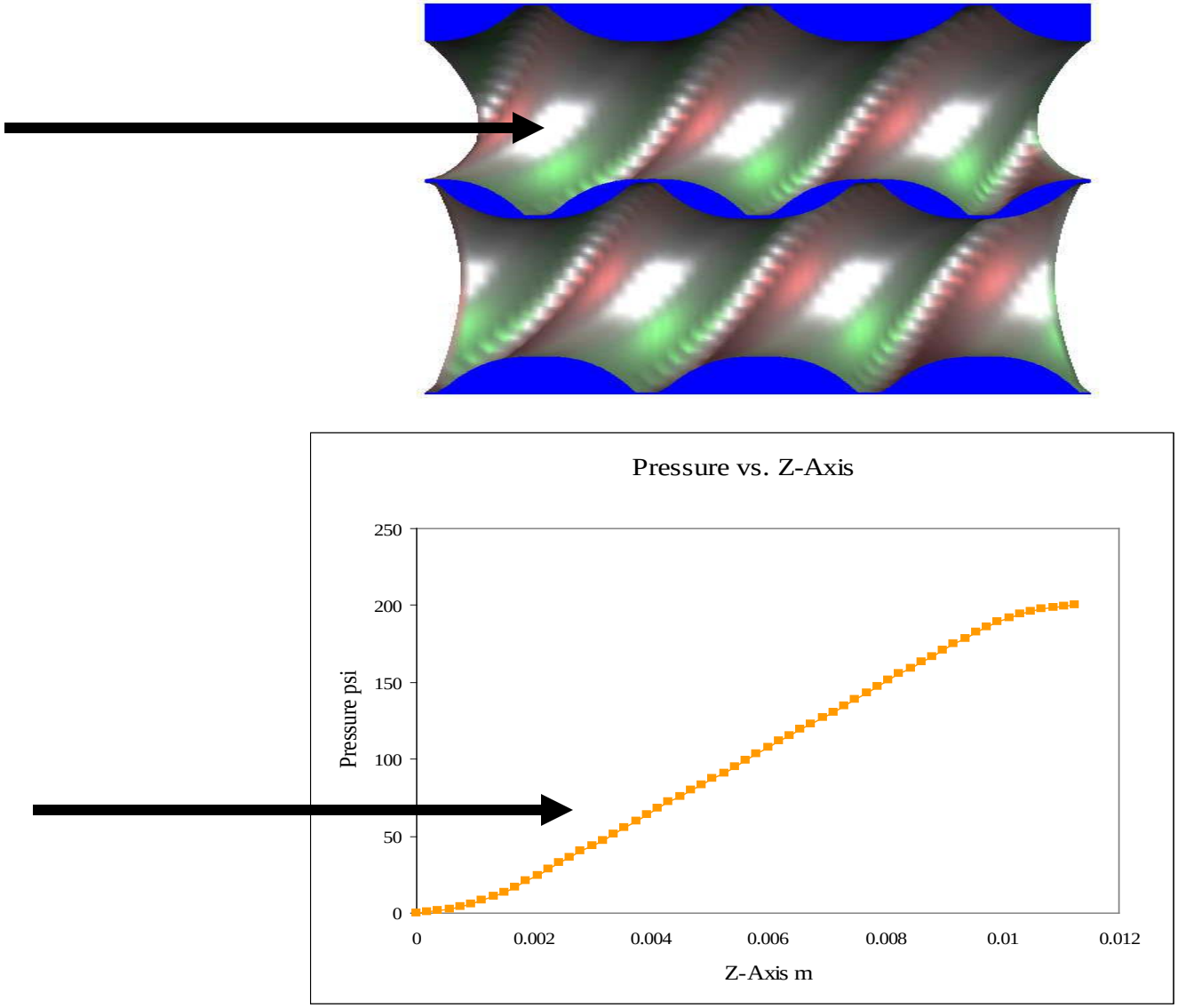
Mini Extruder Third Section Flow Channel Cutaway

Figure 22



Mini Extruder Third Section Flow Channel
Transparent Model

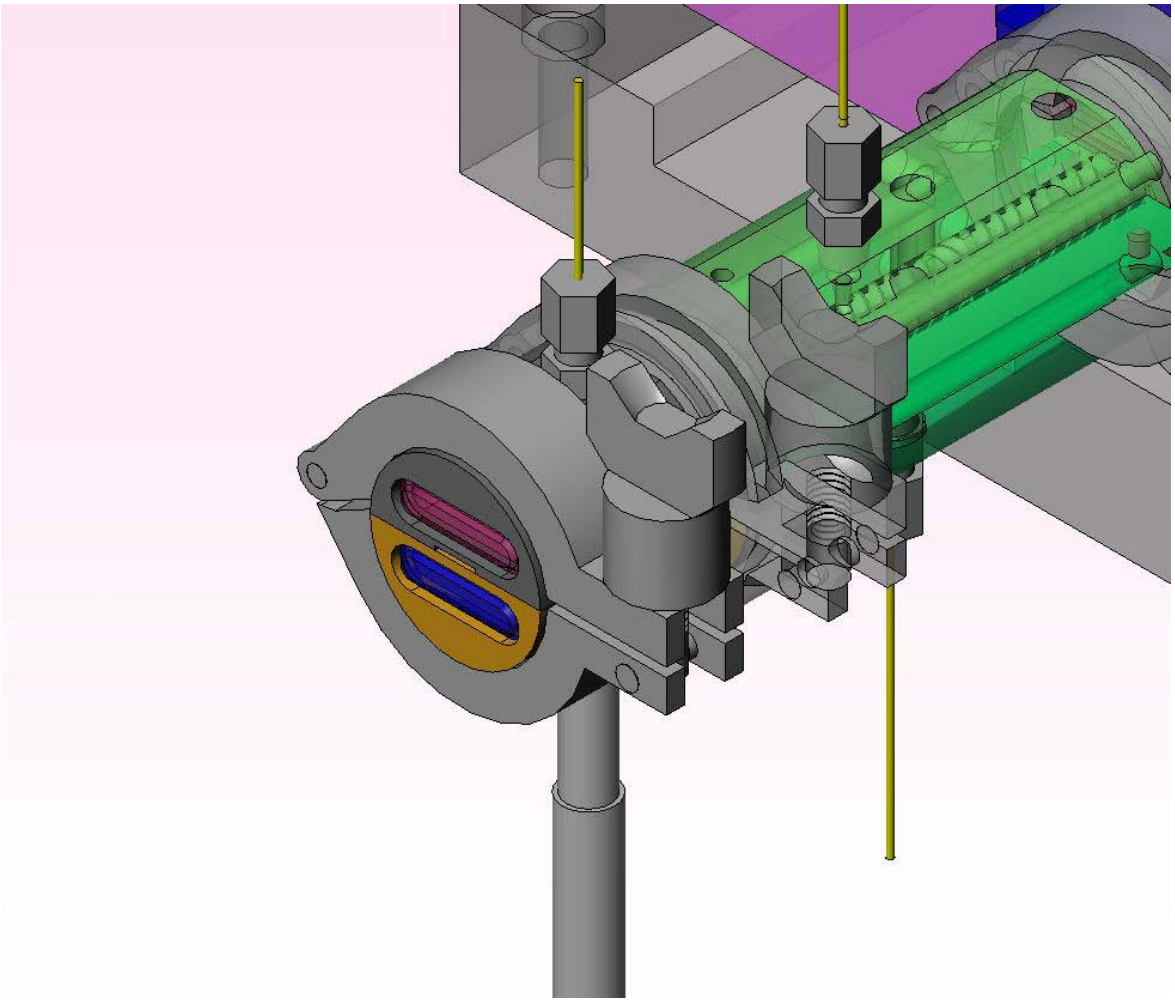
Figure 23



Mini TSE Third Section, 0.05 lb/hr, 105°C

Figure 24

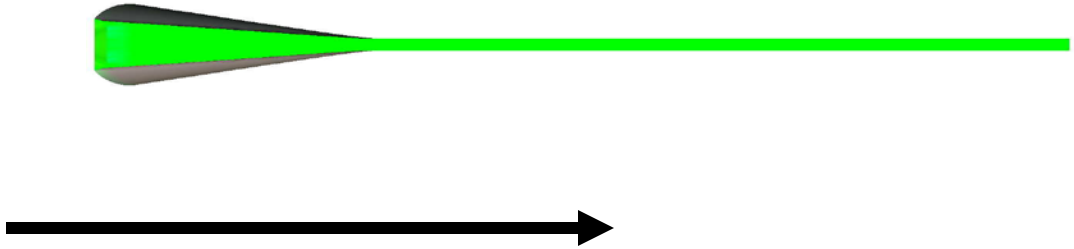
Mini TSE Die Results

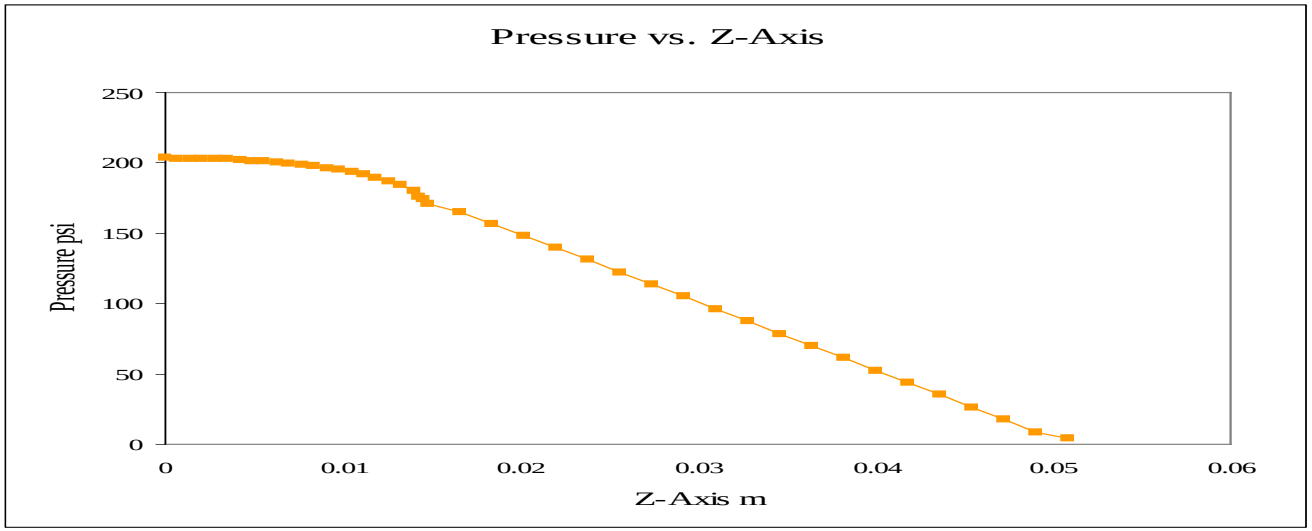


Slit die designed for the twin screw extruder

Figure 26

Mini Extruder Die Flow Channel FEM Mesh





Mini Extruder Die, 0.05 lb/hr, 200 psi,105°C

Figure 28

Safety: explosion proof equipment

- ¼ HP motor, hydrostatic, 0 to 1000 rpm, 100% explosion proof. Parallel shaft gear reducer, 11.66 to 1 reduction ratio, gives us a screw speed of 200 rpm.
- All of the thermocouples and the pressure transducer are explosion proof.
- No electrical heating, temperature control through the circulation of thermostatted oil.

Figure 29

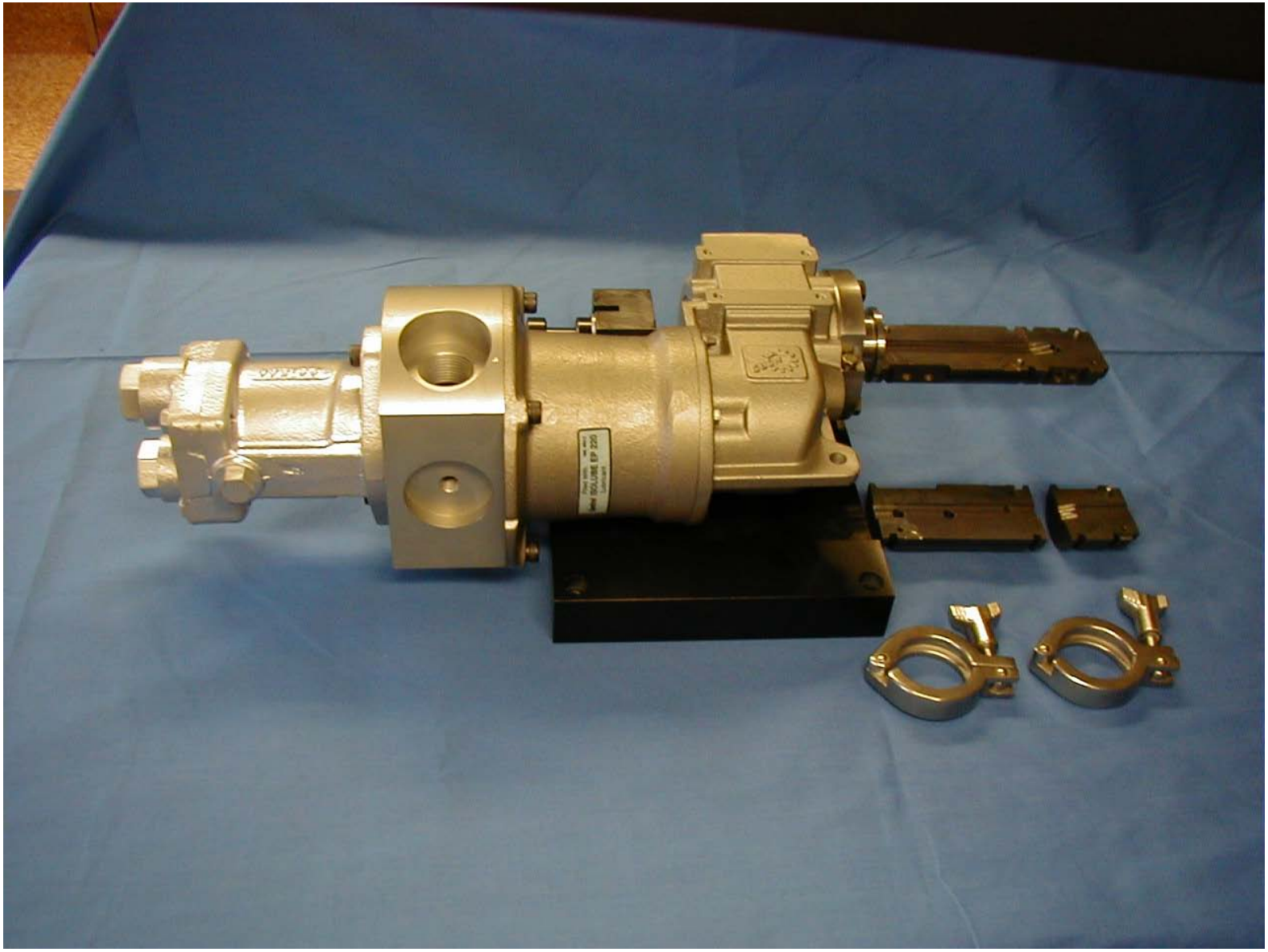


Figure 30

Critical dimensions:

1. **The bore diameter is 7.5 mm and the screw diameter is 7.38 mm. The clearance between the screw and the barrel is D/128 (± 60 microns)**
2. **The centerline distance is 6.27 mm and the screw root diameter is 5.04 mm. The tolerance on the screw diameter is ± .5″/1000″.**

Figure 31

Thrust bearing

1. **The thrust bearings are manufactured from Torlon.**
2. **The coefficient of friction for Torlon is very low.**
3. **With Torlon rusting problems will not occur.**

Figure 32

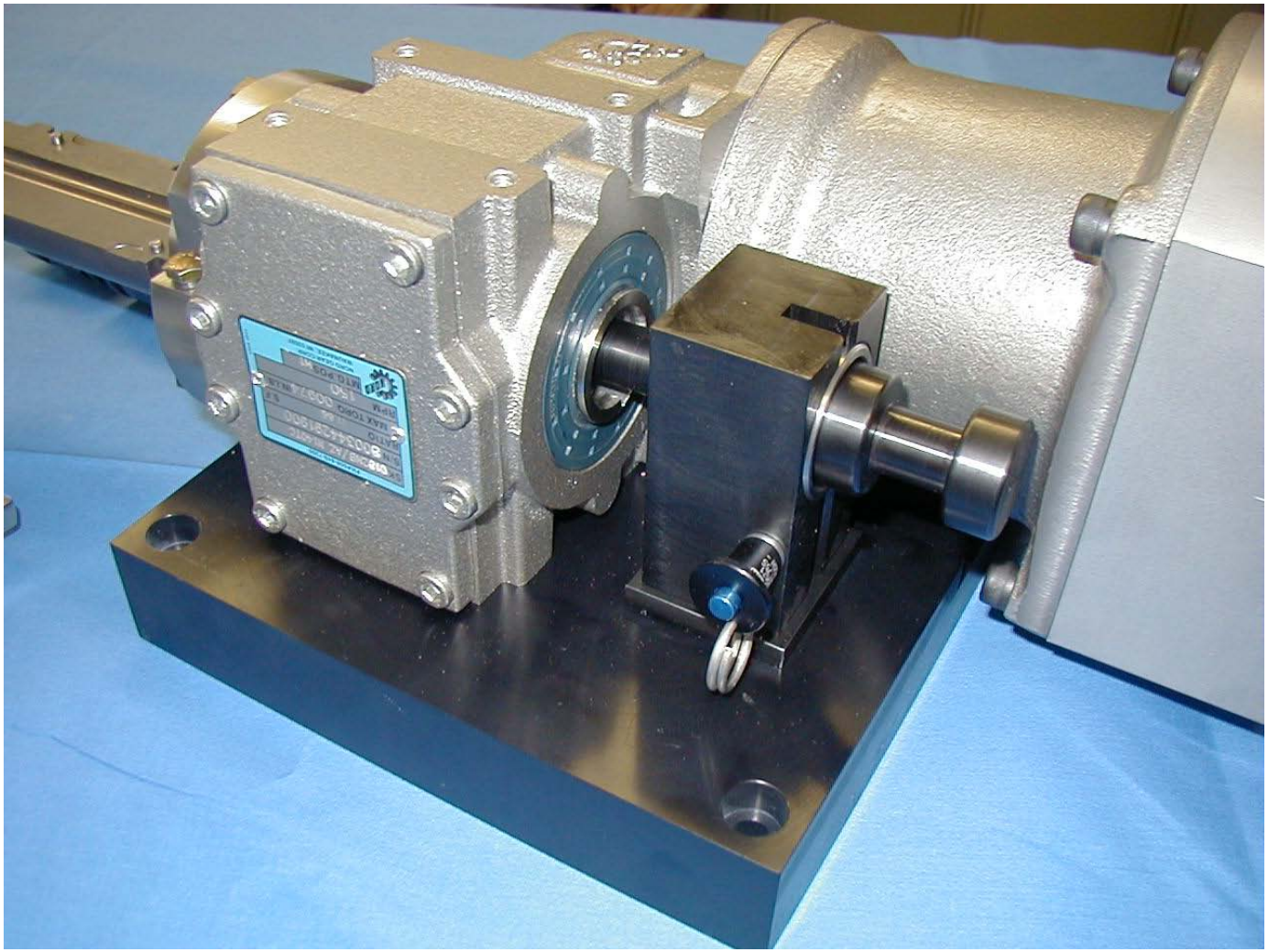


Figure 33

Temperature control

- Temperature control through circulation of thermal oil at the desired temperature
- The heating/cooling channels in the extruder are about 6 mm diameter cross drilled cores
- Separate temperature control zones for the die and the extruder

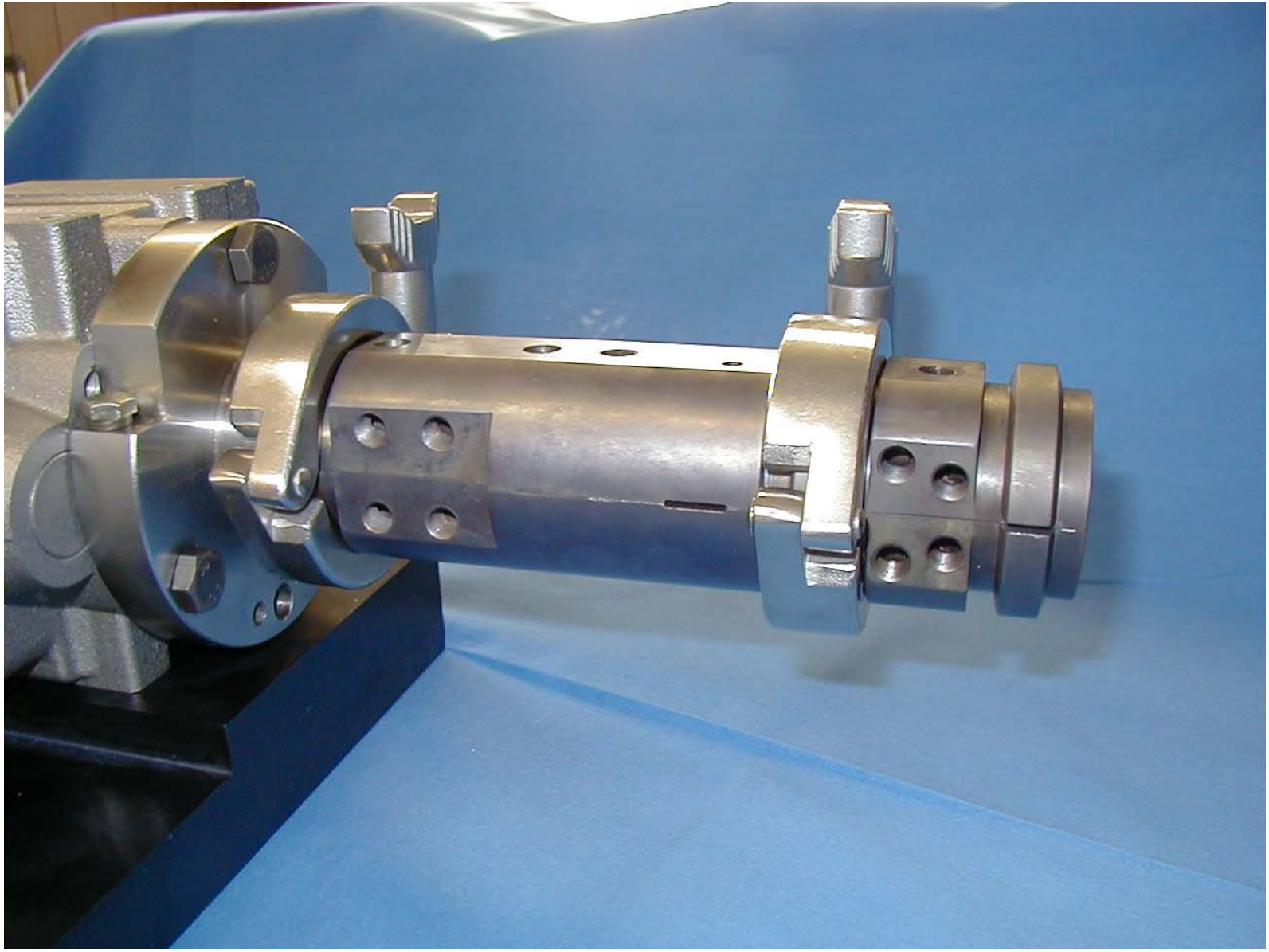


Figure 35

Opening/closing-alignment: Split barrel

- The barrels and the integrated die are held together with three quick disconnects. One is at the stuffing box housing and two more at the die extruder interface where the maximum pressure will be developed.
- The screws just lift up when the gear is disengaged. Pull straight back.
- No tools are required to open and remove the screws. There is a knob with a half moon, when turned 90 degrees to facilitate pulling the sleeve backwards, the shafts lift straight up.
- Up to 5000 psi pressure is acceptable.
- Alignment is not an issue. Dowel pin assembly.

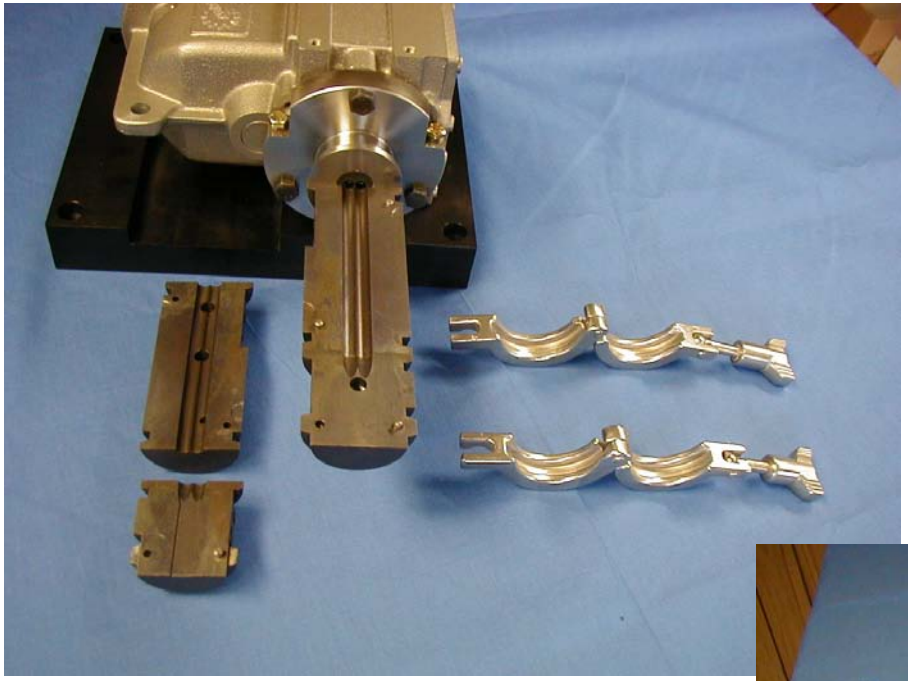


Figure 37

Instrumentation

- **There is one Dynisco (explosion proof) pressure transducer and thermocouple combination at the converging section.**
- **Two thermocouples are placed in the barrel and one at the die. The heating channels for the die and the extruder are different.**

Time-table

- Manufacture of the extruder including the solid screws is completed
- Sensors, electronics and field point based data acquisition and control of the twin screw extrusion is also completed
- Installation of the twin screw extruder at Stevens Institute of Technology for testing and implementation is also completed