THE Molecular Homogenizer

A New Development In Single Screw Mixing

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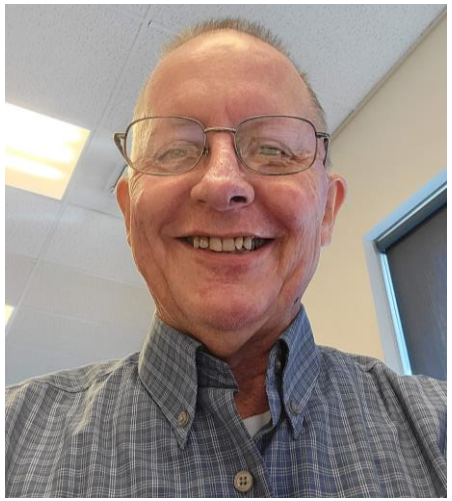
1974 -1987 Killion Extruders:

- Sale Manager 1974-1976
- Lab Manager 1974 to 1984
- Vice President of Development: 1984 to 1987

1988 to Present: Founder/President of Randcastle Extrusion Systems—a manufacturer of small extrusion equipment. Six patents on extrusion including compounding, pressure stability and coextrusion with an additional patent pending for compounding (the topic of papers being delivered for this conference).

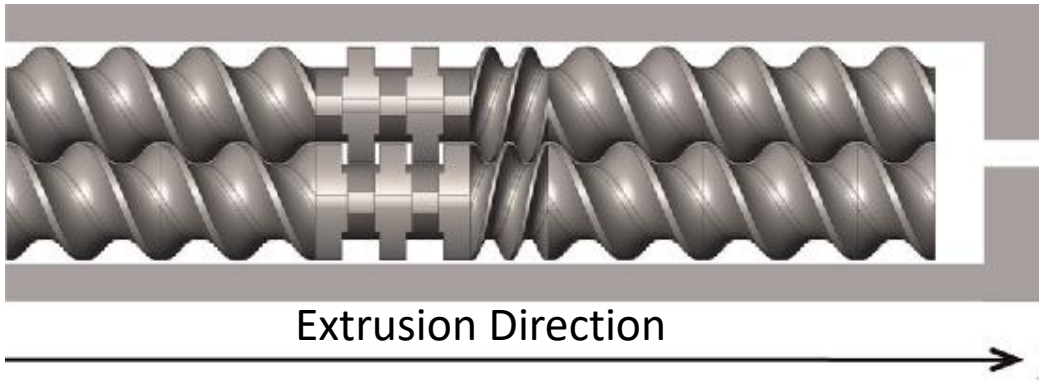
Speaker Photo:



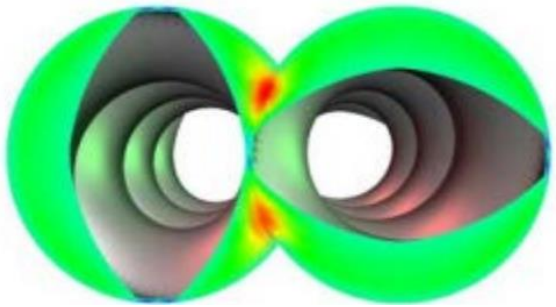
PREFACE

Corotating Twin Screw Extruder (CRTS): The corotating twin screw extruder is the workhorse of polymer compounding. It is well known for its dispersive and distributive ability and yet the CRTS has its flaws. It will be helpful, for the purpose of this paper and the Molecular Homogenizer, to point out some underlying problems with the CRTS with respect to its distributive ability.

Partly empty flights pump melted material into the kneading blocks. Sometimes, mixing is enhanced by reverse flights that force material to remain within the kneading blocks:



The mixing takes place primarily through elongation of the small mass at the intersection of the kneading blocks. From a distributive point of view, the CRTS is disorderly. (Graphic: Dispersion of Nanoparticles Using Twin Screw Extrusion Technologies; Challenges and Opportunities Dilhan Kalyon/Seher Ozkan/Halil Gevgilili/ Jim Kowalczyk* Stevens Institute of Technology * Material Processing & Research Inc. http://www.pica.army.mil/jocg/files/2006/15%20- %20Dispersion%20of%20Nanoparticles%20Using%20Twin%20Screw%20Extrusion%20Technologies,%20Challenges %20and%20Opportunities.pdf)



Disorder and chaos, cannot continually produce order. The CRTS suffers from disorderliness because:

- **Split Mass:** Only a small fraction of the mass is stretched at kneading block intersection. Most of the material, shown in green above, bypasses the intersection. The mass is not treated uniformly. Some of the mass is mixed. Some is not.
- **Uneven Distributive Mixing:** There are many intersections in a CRTS. Nevertheless, some material will not be processed through the mixing intersection. Some material will pass through intersections more than once. The mixing must be uneven.
- **Uneven Dispersive Mixing:** The forces at the mixing intersection of the kneading blocks are not uniform. Different stresses are shown by color in the graphic above. When such dispersive forces are uneven, uniformity is negatively affected.

Static Mixers: Static mixers are very orderly, Nevertheless, it is useful to point out some of their drawbacks. A variety of static mixers exist. Static mixers are positioned after the extruder and use the extruder's pressure to divide the flow into many streams. The streams are then recombined in different locations for mixing. The main claims of static mixers are for temperature uniformity and localized color mixing.

However, some undesirable aspects are:

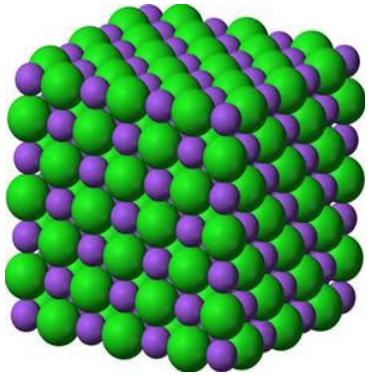
- Insufficient Layer Creation: While the number of layers they create may seem large, it is not enough to mix to the small molecular level.
- No Axial Mixing: While all mixing is three dimensional, to mix well in three dimensions requires the creation of many layers in all three dimensions. Static mixers have no machine direction mixing.
- No Pressure Uniformity Improvement: Static mixers do not improve the pressure stability that the extruder produces.
- Pressure Consumption: Since static mixers consume the extruder's pressure to function, the output of the extruder can be less.

Various static mixers are discussed by Rauwendaal (Polymer Extrusion, 4th Edition, 376 to 386).

Pumping: Many mixers have attempted greater shear intensity by pushing melted material through a narrow opening. This is the method employed, for example, in the UC mixer, Egan mixer, and many barrier screws. In order to understand how Randcastle's Molecular Homogenizer works, it is necessary to understand a fundamental concept: When one smooth surface passes another, with polymer in between, pumping occurs. This is not a high-pressure pump. But, it is a very useful low-pressure pump. It is important to remember that low pressures are used for elongational mixing in the CRTS.

INTRODUCTION

Atomic Scale Mixture: The scale of mixing is ultimately defined by the atom. As such, a crystal composed of two different size atoms represents the ideal mixture. It is an orderly three-dimensional arrangement of atoms.



As such, it is the ideal mixture to emulate. Presumably, science fiction's Star Trek replicator could produce such a crystal by assembling the mixture one atom at a time in a perfectly orderly fashion. At this time, the Star Trek replication is unavailable.

Small Molecule Mixing: At a larger scale, gases such H2O are still very small. We judge the quality of a gaseous mixture in a polymer in many ways. For example, when processing hygroscopic materials, if we see bubbled extrudate, we judge the drying insufficient. Yet, we do not concern ourselves about a single water vapor molecule within the matrix being a gas. We do not think one molecule important during extrusion or molding; more, we do not concern ourselves with how many individual water vapor molecules exist in polymer as long as they do not agglomerate into water or affect viscosity. A polymer mixer that keeps water vapor molecules from agglomerating into visible bubbles measures the quality of the mixer.

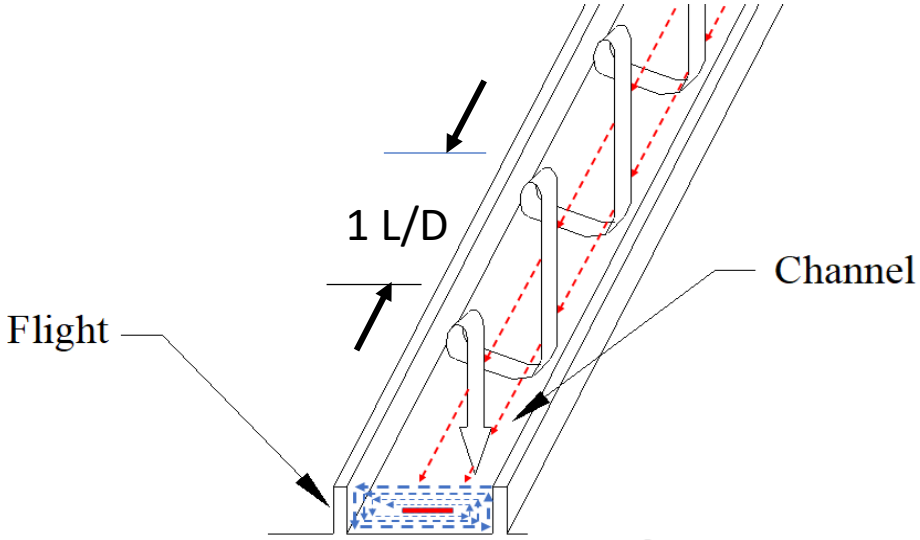
Another measurement of a mixer's quality can be assessed during reactive extrusion. A polymer may be hygroscopic and yet bubble free if the water vapor is kept from agglomerating into visible bubbles by mixing. It is also possible that the mixer may have additional capacity. In the case of an undried hygroscopic material where a reactive agent is also added, an unbubbled extrusion would demonstrate the mixer's quality.

Many dry, pelletized hygroscopic polymers are known to absorb moisture in a less than 6 hours when exposed to water vapor in the atmosphere. A mixer that first pelletized dried pellets and delayed the moisture absorption substantially demonstrates the mixer's quality.

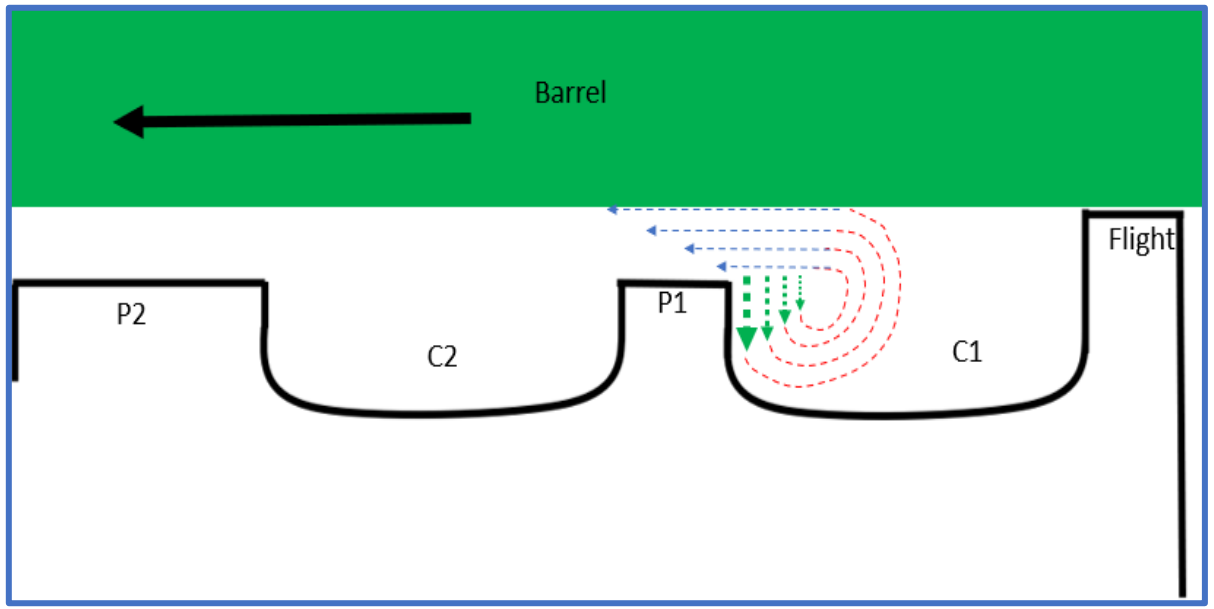
Requirements For Polymer Homogenizer Of Small Molecules:

- **Scale:** Water vapor and other gases are very small molecules; polymers are macromolecules. So, an improved polymer mixer must be capable of processing both large and small molecules.
  - **Screws:** Screws are well known to process large polymer molecules.
  - **Small Molecule Mechanism:** A mixing mechanism capable of mixing the smallest molecules in all three dimensions within the macro molecule matrix is necessary. Because of the size difference between macro molecules and very small molecules, a mixing mechanism will demonstrate mixing performance over many orders of magnitude.
- **Low Energy:** A polymer homogenizer must not significantly damage the polymer, by temperature, time, or shear, in the process of mixing.
- **Orderliness:** A polymer homogenizer must be orderly. It must treat the entire mass uniformly. It cannot be chaotic or uneven.

Conventional Metering Section Mixing: The flow in a conventional metering section is shown below as an unwound spiral. The barrel, not shown, can be thought of as a flat plate driving polymer forward against the inside of the channel wall. This forms a helical flow as material moves down the side of the channel, across the channel bottom, up the channel wall and along the barrel (represented by the helix and in blue in the end view) as material is conveyed forward. At the same time, pressure flow in the middle of the helix moves at a slight faster speed represented in red. The difference in velocity between the spirals and central flow is the mixing mechanism. Since spiral creation per L/D is very limited and the velocity differential between the spirals and inner flow is nearly inconsequential, it is a very poor mixer. Note that the friction along the channel surfaces and across the barrel is substantial with consequent temperature rise.



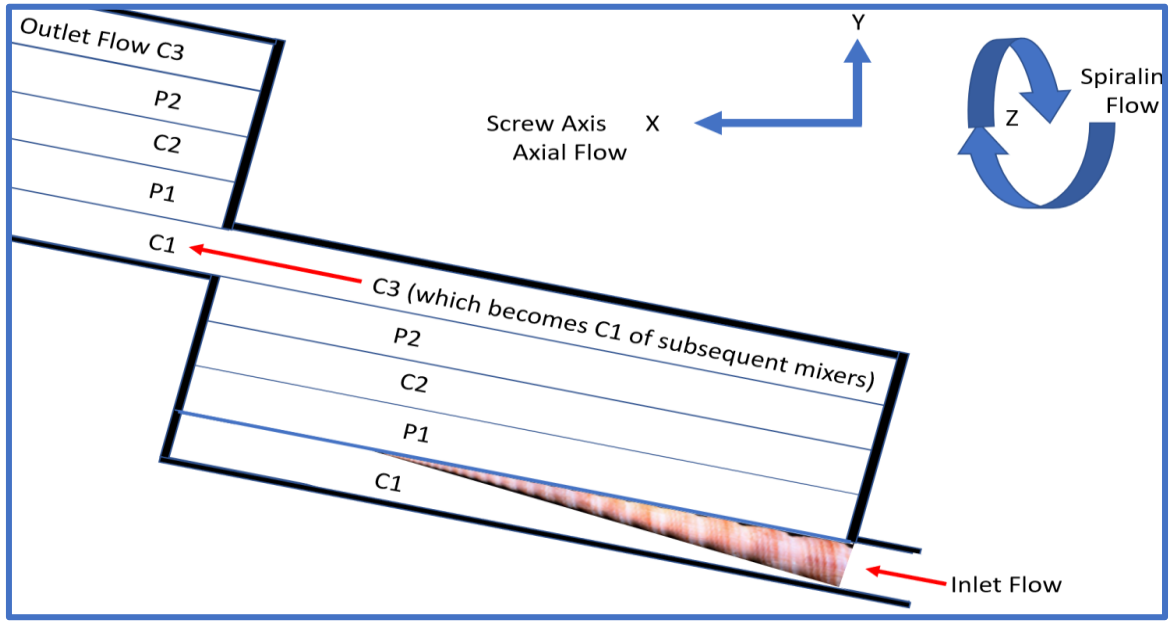
Starved Metering Section: However, a starve fed screw near a pump (two smooth surfaces presented with polymer) can display a completely different behavior, as below. The downstream side of the channel, in cross section, can become circular and tethered only to the side of the flight. The barrel will drive the molten "ball" in a cross-channel direction, in tension and elongating the flow, while the "ball" moves downstream against the flighted surface.



Note that the green arrows indicate the thickness of the layers and that the layers thin thereafter, drawn by the barrel from the tethered surface, as the layers spiral downstream.

Note that the flow over P1 is in planar shear. There is no pressure to push the material through the P1 clearance. Additionally, because there is no pressure pushing material through the narrow opening, the temperature rise in the gap is minimized. This is unlike the flow in a UC, Egan mixer, or barrier screw, where pressure flow detracts from the drag flow of planar shear and causes temperature rise.

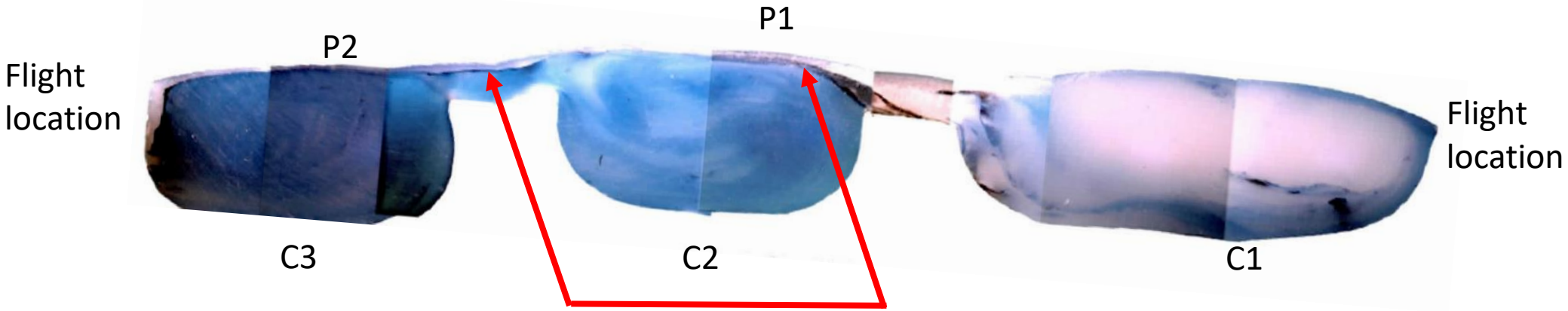
Unlike the constrained spiral in a typical metering section, the number of spiral increases dramatically in the Molecular Homogenizer. This is more readily seen in the following schematic where the mixer is unwound into a flattened view. As material is pumped into the C1 channel, the barrel drags material forward in the Y direction. The inlet flow becomes conical. Without restriction beyond the tethered section of the channel, spirals form over a short distance. The flow in the C1 channel represented below is about 2 L/D long. At the larger end of the cone, fewer spirals will form than at the smallest end.



The energy required to drive the melted spiraling tethered ball is very low thus keeping the melt cool. In the experimental 1 inch screw used for these experiments, the motor amperage of a 5HP drive was measured and required 5.6 amps to drive an empty screw. During several experiments, the amperage required (for the melting and mixing) was only 5.8 - 6.1 amps. The maximum drive amperage at shutdown, often achieved during experiments with a conventional screw, is 13 amps. The very low amperage is a measure of how much energy the motor requires to drive the mixing process and for the Molecular Homogenizer is it very little.

Note that the P1 pump draws material from the outside of the inlet flow while the inside of the spiraling flow moves downstream. Note too, that the surface of the C1 flow continuously exposes new material. This is very useful when considering very small molecules, such as gases. Gases can be beneficially extracted for venting.

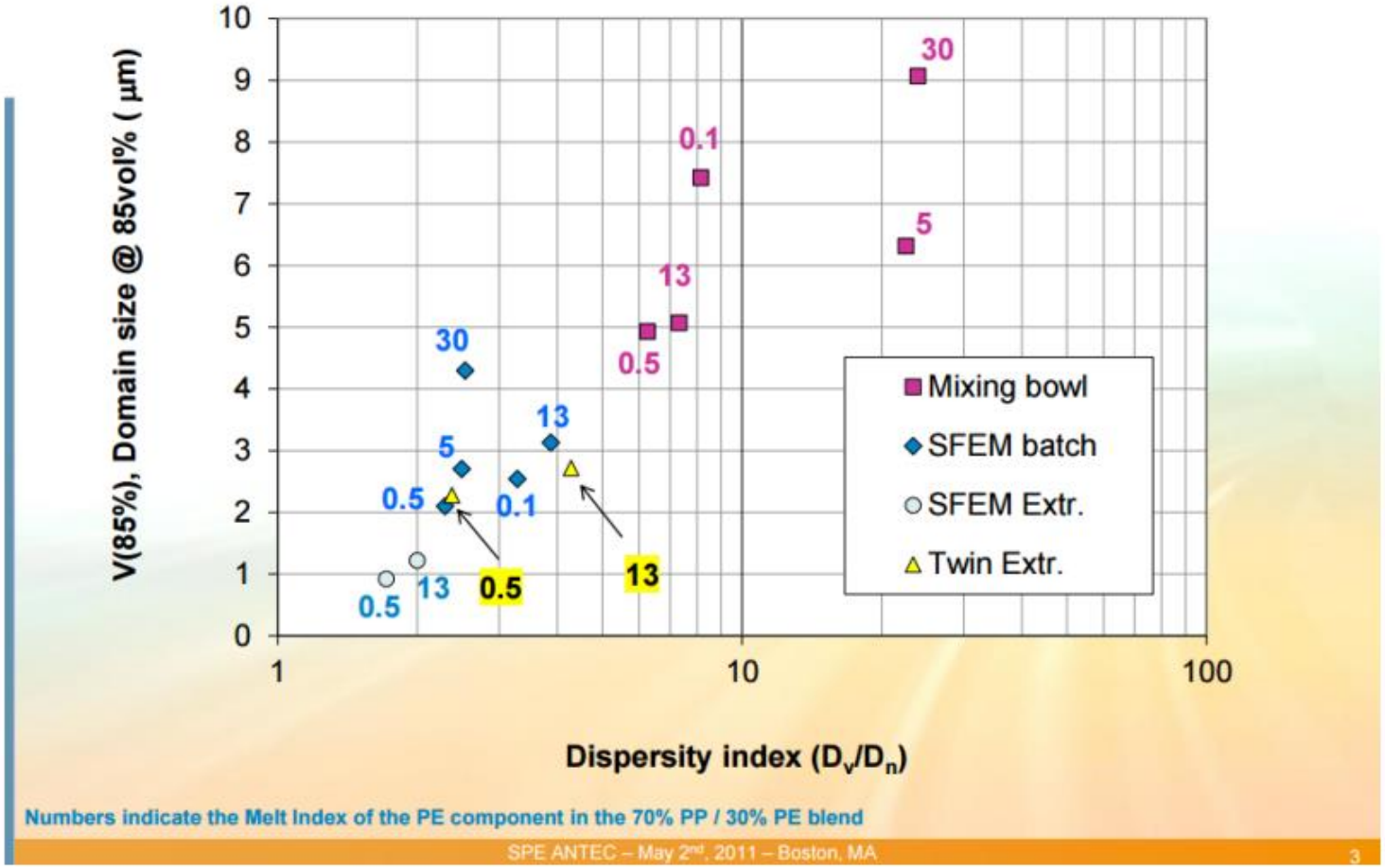
The next downstream part of the mixer is the C2 channel. Previous work demonstrated that the flow from a pump was stretched dramatically. Here a cooling experiment in a flood fed screw with blue colored pigment that was extracted to show the stretching (Antec: INVESTIGATION INTO A HIGH OUTPUT POLYPROPYLENE SCREW AND ITS MIXING MECHANISM, Keith Luker, Randcastle Extrusion Systems, Inc., Cedar Grovce, NJ, Thomas M. Cunningham, Extrusion Technical Services, Brodheadsville, PA)



The depth of the channels is 0.18 inches and the P1 and P2 clearance is 0.04 inches. The flight clearance is 0.0025 inches so the stretched film leaving P1 is about 0.001 or about 1.4 orders of magnitude smaller than the gap. Although the C2 channel is filled in this case, the stretching still takes place because the barrel is dragging the film from the P1 gap and drawing it towards the barrel.

A similar experiment with immicible polymer blends was described by Dow Chemical. In their oral presentation at Antec, they included work developed after the paper was presented in the following graph. The paper (FACILE TPO DISPERSION USING EXTENSIONAL MIXING Stéphane Costeux, Mark Barger, Keith Luker*, Anand Badami, Kim Walton The Dow Chemical Company, Midland, MI (U.S.A.) *Randcastle Extrusion Systems, Inc., Cedar Grove, NJ (U.S.A.) showed that the diameter of the two tested materials **was half that of the Dow twin screw so that the volume was 8 times less per domain or 8 times better mixed**. The dispersity index shows that the distribution was **twice as good as the twin**.

Dispersion map



Numbers indicate the Melt Index of the PE component in the 70% PP / 30% PE blend

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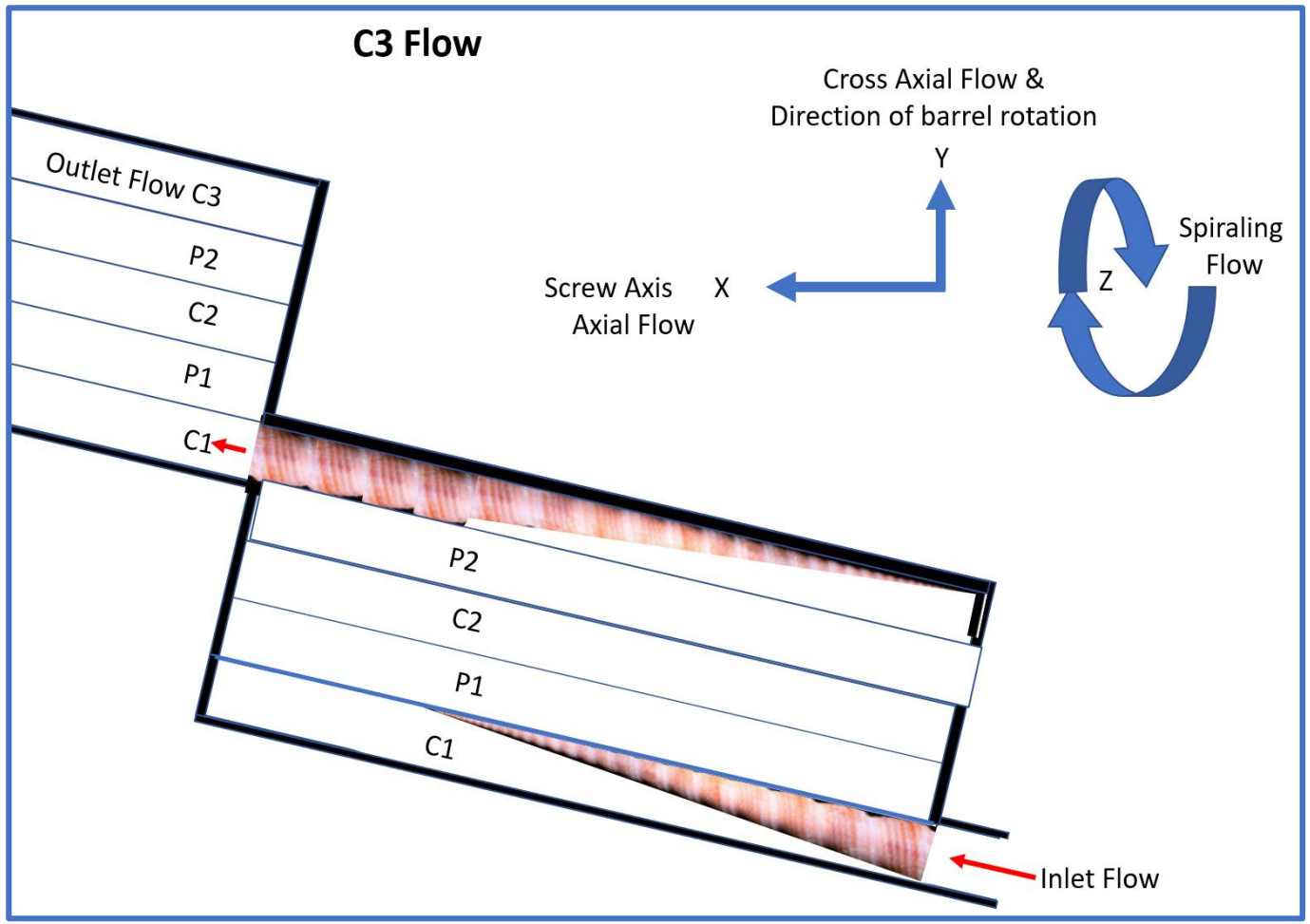
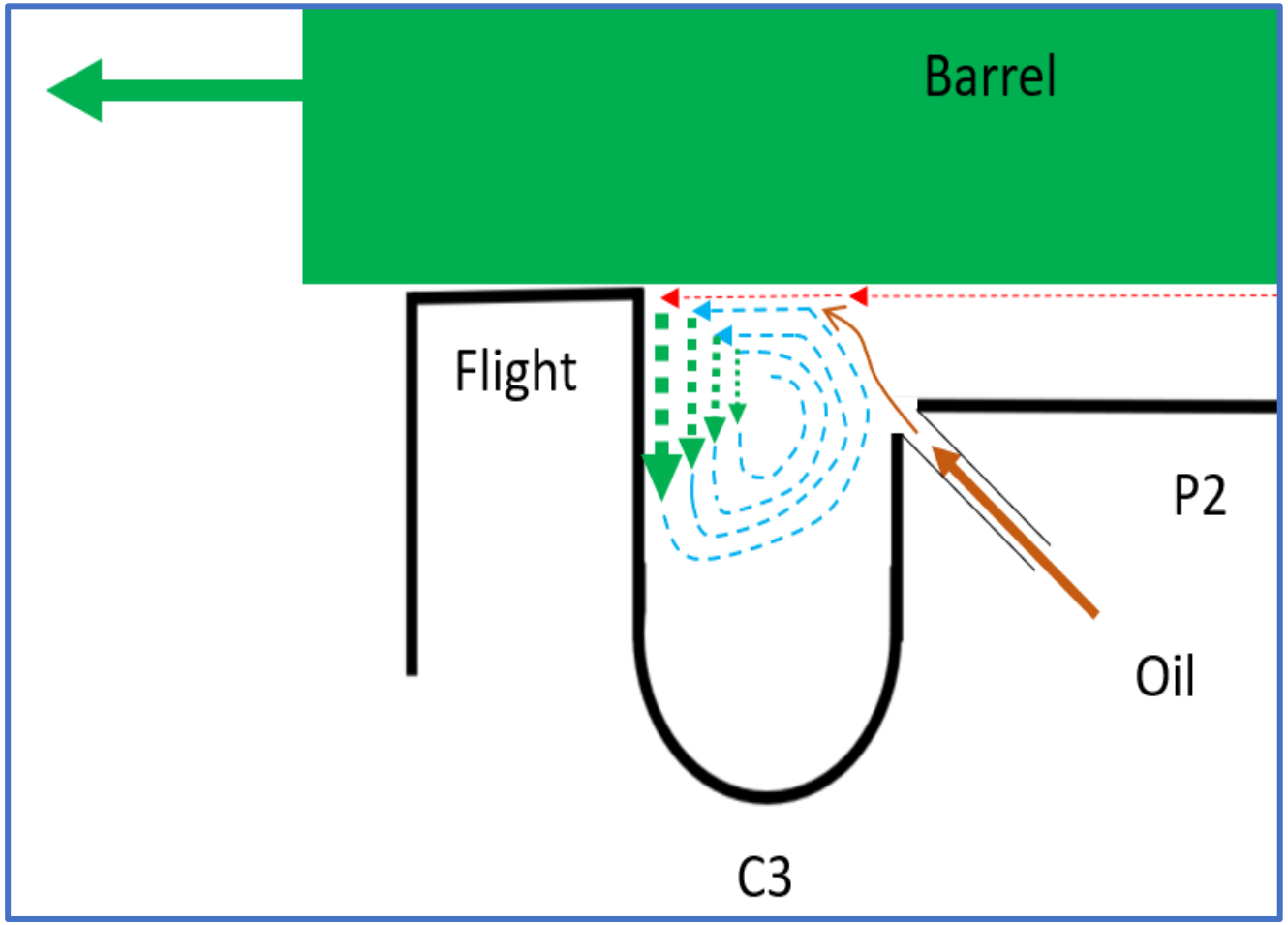
The last part of the mixer is the C3 channel. Here, the very thin film stretched over C2 becomes the inlet flow of C3. It is generally described the same way as the flow in C1 in that it creates many spirals. However, the upstream spirals are many and the downstream spirals few. When the number of spirals from both channels are added together, the entire flow exiting the C3 channel will be the same. Unlike the C1 flow, new surfaces are not created but wound.



Instead, the thin layers wrap around the core from the upstream flow. This also leads to the possibility of trapping fluids such as oil (through the screw) or gases flooded into the screw, though vents or under pressure. Oils are usually problematic for single screws because the barrel surface becomes lubricated which stops conveyance. By introducing oil through the screw, it might be possible to mix more oils or other slippery materials than had been previously possible.



Conclusions:

- **Energy and Handling:** Every undried hygroscopic material tested so far shows no sign of bubbles. The range of tested materials includes PET, PET with 25% regrind, polycarbonate, PMMA, SAN with color concentrate, PEEK, coffee chaff in LDPE, PVA copolymer and PVA copolymer with a reactive component. This represents a wide range of materials.
    - We cannot see what's happened to the water vapor but, it may be that the water vapor is kept from agglomerating into water by the mixer.
    - It may be that water vapor has agglomerated into water, but the bubbles are so small that we cannot see them. In that case, the materials might be considered micro-foams.
    - If the properties of the polymer are not diminished in some way, the energy and handling savings are substantial.
- **Delayed Water Pickup:** Dried PMMA typically displays water as bubbles or surface degradation in about 4 to 6 hours when exposed to atmosphere. However, dried PMMA processed through the Randcastle Molecular Homogenizer, and exposed to the atmosphere for three days, did not show any sign of bubbles or surface roughness after three days. This represents a very significant delay in water vapor pickup. We are currently working to extend this time substantially in the hopes that material can be kept dry, outside of a polymer reactor.
- **Additional Gas Capacity:** Two materials were tested that showed more capacity to keep gases from appearing. The first was a PVA copolymer with 3% metalorganic reactive material. Neither the hygroscopic PVA nor the conversion of some of the polymer chain into gas from the reaction showed bubbles. Neither did the two SAN color concentrate (black and white) display signs of water. So, the Molecular Homogenizer kept, not only the SAN but also the water vapor in the pigments from appearing.
- **Very Large Surface Exposure:** Continually exposed surfaces are themselves a measure of mixing. In addition:
    - **Venting:** Contrast the surfaces in the Molecular Homogenizer to the typical two stage single screw extruder screw used for venting. *Some testing has taken place and will be discussed in the compounding breakout.*
    - **Gaseous Introduction:** It seems possible, too, that gases may be efficiently trapped especially at the C3 tethered ball between extremely thin layers. A preliminary trial show significant increase in water vapor take up after exposure to nitrogen. *This will be discussed in the compounding breakout.*
    - **Other Smallish Molecules:** With our previous technology, we showed that CNT could be very well mixed to the 1 micron scale (Luker, 3 Dimensional Mixing An Orderly Mixer For Single Screw Compounding, www.Randcastle.com). Carbon nano tubes, graphene and other large molecules may be ideally suited for this technology. *While no testing has taken place, a conceptual mechanism for incorporation of such materials will be discussed in the compounding breakout.*
- **Three-Dimensional Mixing:** While all mixing is three dimensional, we will discuss the three axis of mixing during the compounding breakout paper. This will include the notion of error mixing.
- **Other:** There are many possibilities for the Molecular Homogenizer including alloys, immiscible polymer blends, enhancing twin screw mixtures, conductive materials, etc. Work is now underway with various customers on their individual projects.