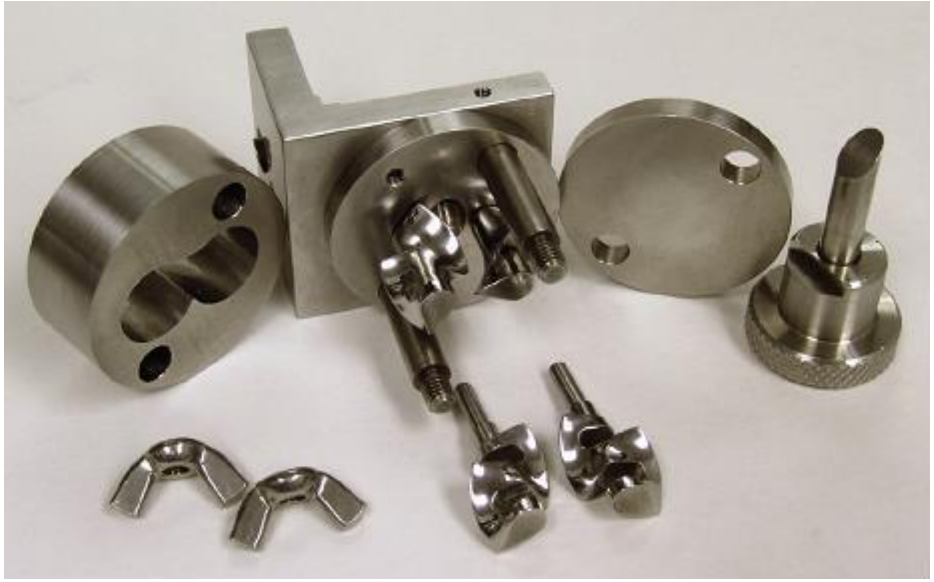
A Novel Miniature Mixing Device for Polymeric Blends, Nanocomposites, and Food Compounds



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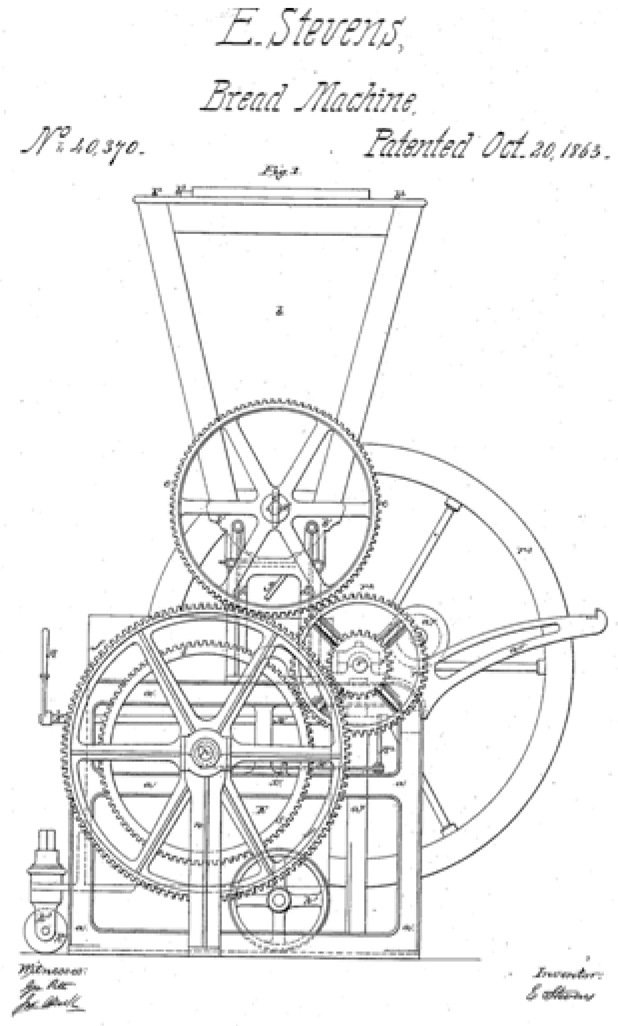
Mixing Technology

- Automated internal batch mixer devices with a container or enclosed mixing vessel/chamber have been in use since the nineteenth century for the preparation and compounding of foodstuffs, clays, cements, plastics, and other fluid-like media



Mixing Technology

- Although the art of automated mixing technology may have its roots from over a century ago, the science of automated mixing has slowly evolved over just the past few decades
- Regardless of the size or scale of the processing operation whether for the production of bread dough or the compounding of complex polymeric materials, the ability of individual material components to intimately interact in a controlled manner is a fundamental principle of all material mixing and compounding operations

Small-Scale Mixing

- Small laboratory-scale mixers were developed for mixing protocol evaluation purposes from a quality control and research & development capacity in order to study, develop, and qualify new materials and compounds, improve industrial-scale mixing efficiencies and predictability, and to reduce the cost and time associated with industrial-scale pilot mixing trials
- Although many commercially available laboratory-scale mixer devices currently exist, few can handle batch capacities less than 5 mL in total volume

Objective

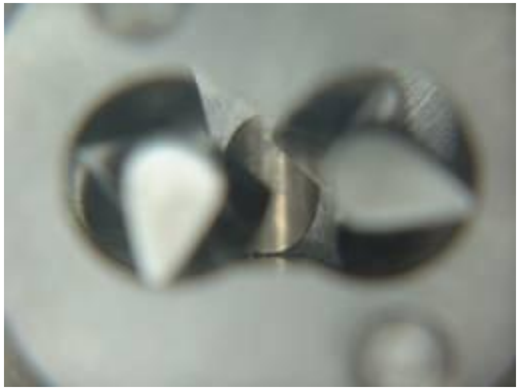
- Study the effectiveness of mixing and compounding of natural and synthetic polymer blends at batch volumes less than 5mL:
  - Develop and evaluate a novel miniature mixing device
  - Compare the mixing and rheological behaviors of compounds mixed in larger scale mixing devices

XIM Mixer

- The XIM is a detachable dual rotor/chamber mixer attachment for use on the new SER2 model line of SER Universal Testing Platform from Xpansion Instruments
- The XIM can be accommodated within the oven chamber of the host rheometer system and/or can be accommodated with independent band heater control

XIM Mixer Prototype

Chamber filling port



Bottom view of mixing chamber showing chamber filling port

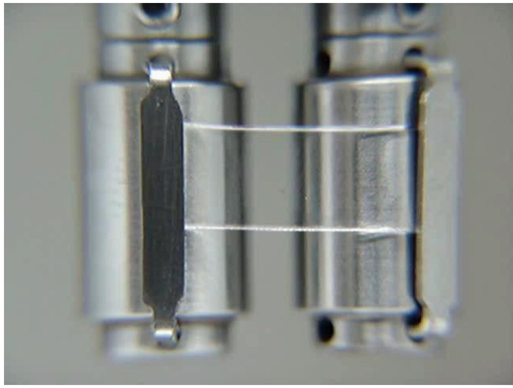
SER Universal Testing Platform

- The SER Universal Testing Platform is a miniature detachable fixture that can convert a conventional CSR or CRR rotational rheometer system into a single universal test station.

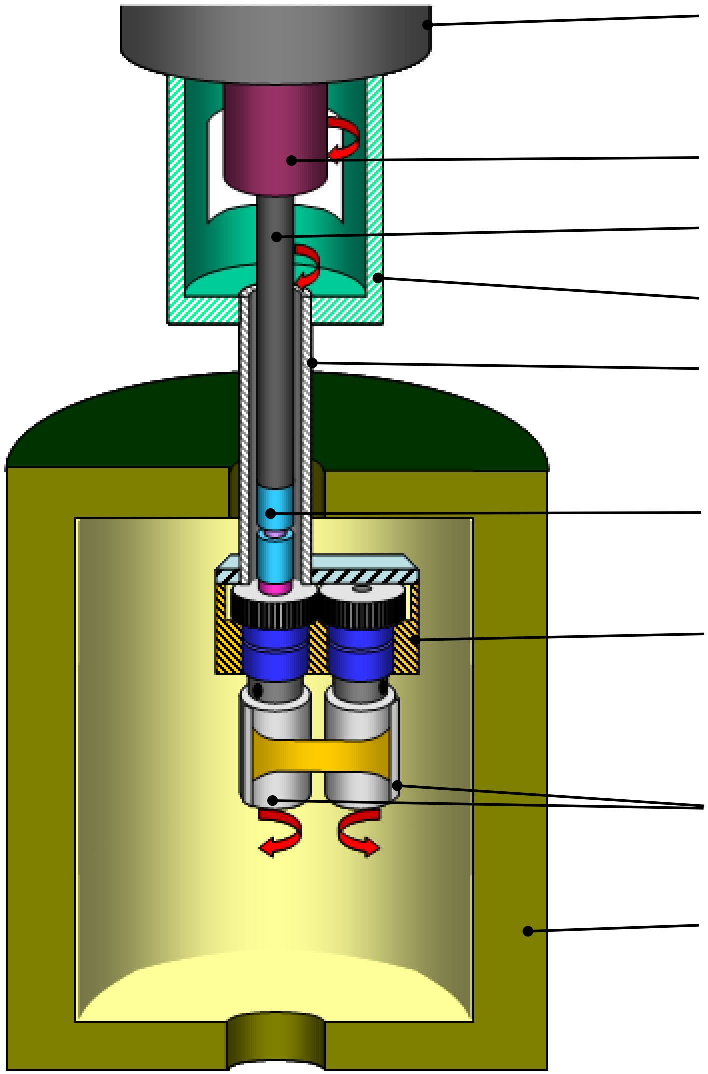
SER Universal Testing Platform

- Covering a broad spectrum of physical material characterization:
  - Extensional rheology, Solids Tensile Testing, Tear Testing, Peel/Adhesion Testing, Dynamic Friction Testing, and Cut Growth/Fracture Testing
- SER Technology translates the precision rotational motion and torque sensing capabilities of a commercial rotational rheometer into precision linear motions and loads.
- By utilizing counter rotating windup drums, linear deformations can be precisely controlled in a fixed plane of orientation which can be viewed at all times during the material deformation process.

New SER2 model line

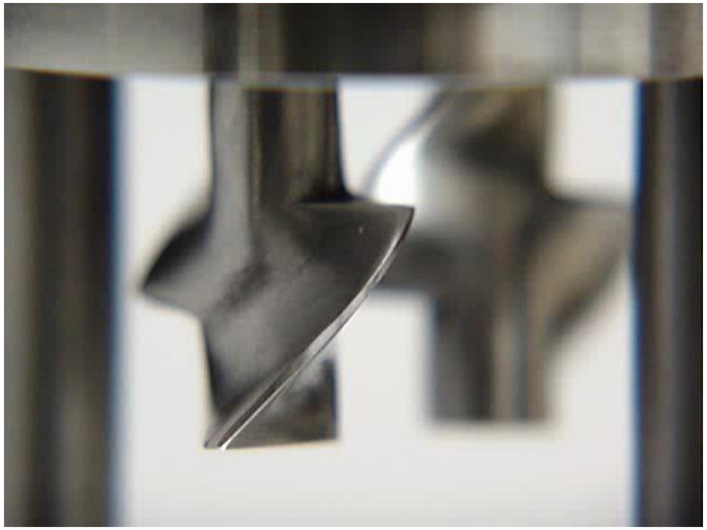


SER2 Configuration



- The rotational motion of the rheometer spindle drives the counter rotation of the dual windup drums.
- Hence, a sample attached to the drum surfaces experiences a controlled ***linear*** deformation all within the confines of the oven enclosure.

XIM Configurations



2-Wing Rotors

Sigma-Blade Rotors

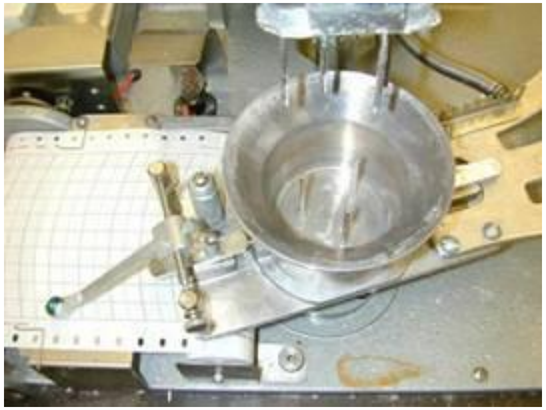
- The XIM has dual 2-wing rotor (3.75 mL mixer capacity) as well as dual sigma-blade rotor (4.0 mL mixer capacity) configurations

Mixing Comparison Experiments - Gluten

- Material - Gluten
  - Vital Wheat Gluten (Arrowhead Mills)
    - Same type of gluten used by

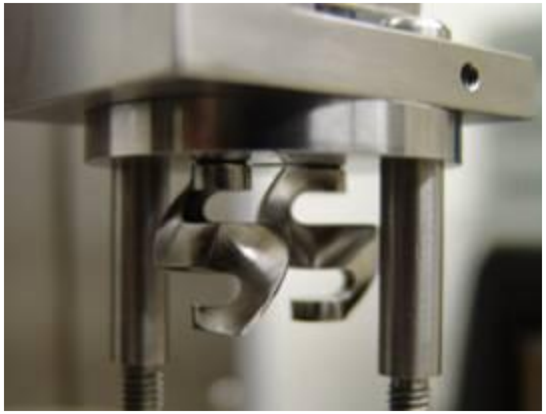
      Ng and McKinley at MIT

      Ng and McKinley, J. Rheol. **52**, 417-449 (2008)



Mixograph pin mixer (~40mL)

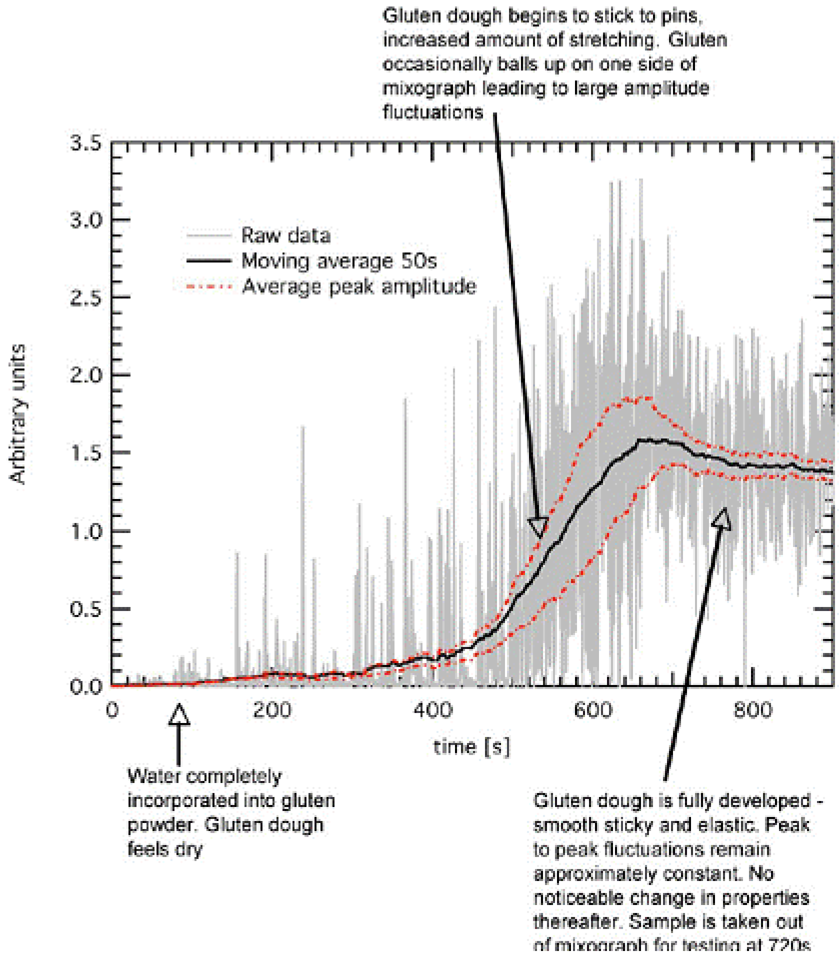
- Types of Lab Mixers used:
  - Ng and McKinley – Mixograph pin mixer (~ 40 mL capacity)
  - XIM accommodated on a model SER2-P hosted on an Anton Paar MCR 501 (Dual Sigma-Blade Rotors – 4.0 mL capacity)



XIM dual sigma-blades (4mL)

Torque Curve Comparisons - Gluten

Mixograph pin mixer: [Ng & Mckinley (2008)]
10g Gluten + 14g Water
88 rpm for 12 min



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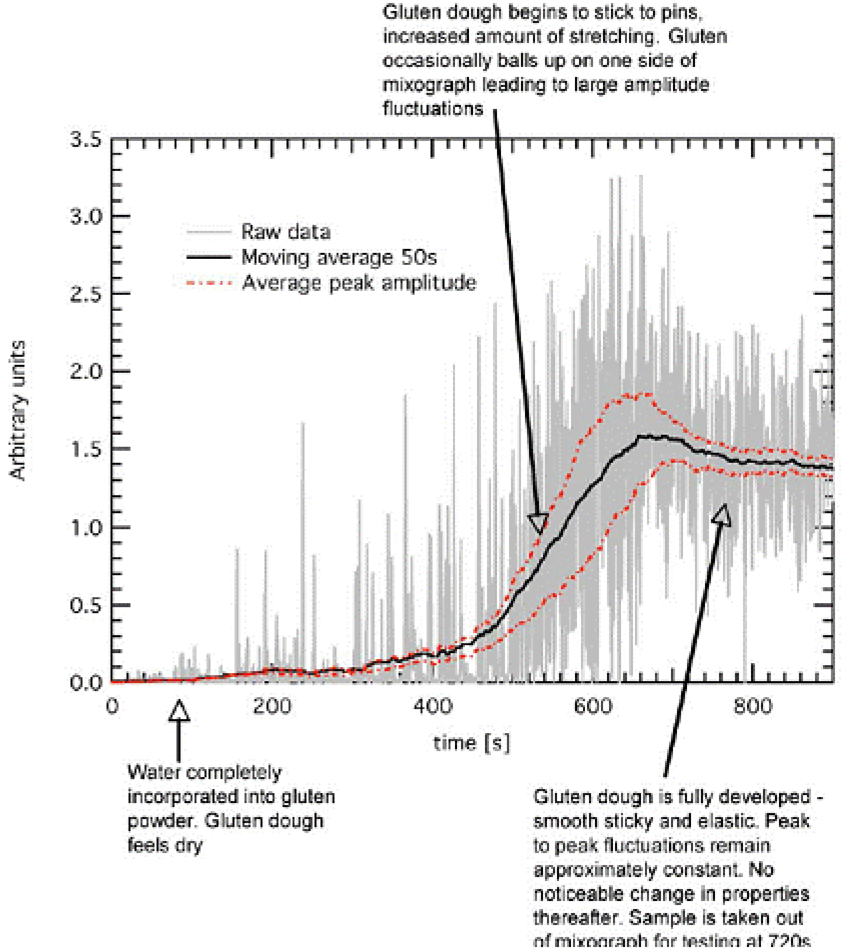
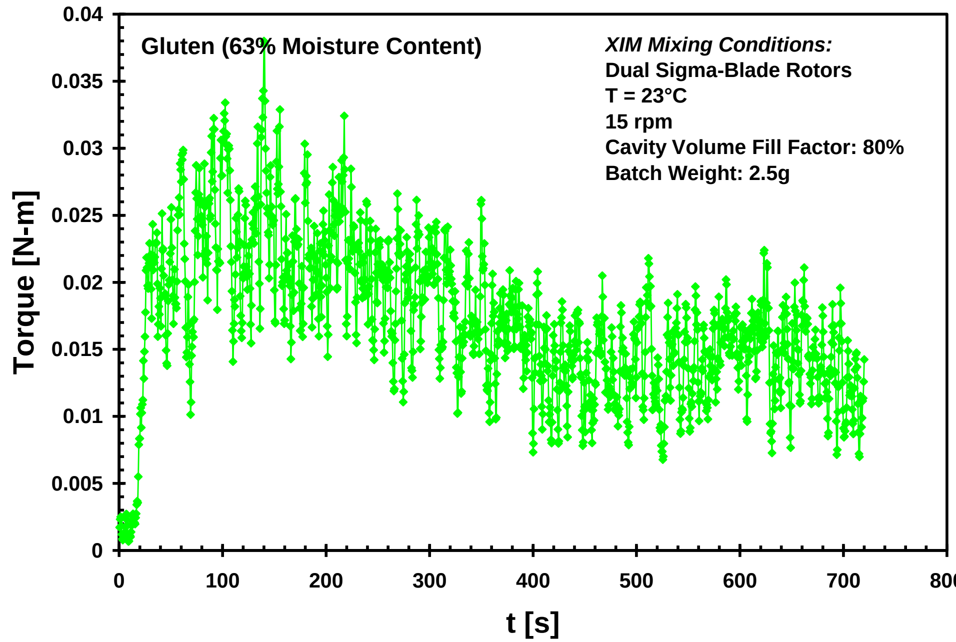
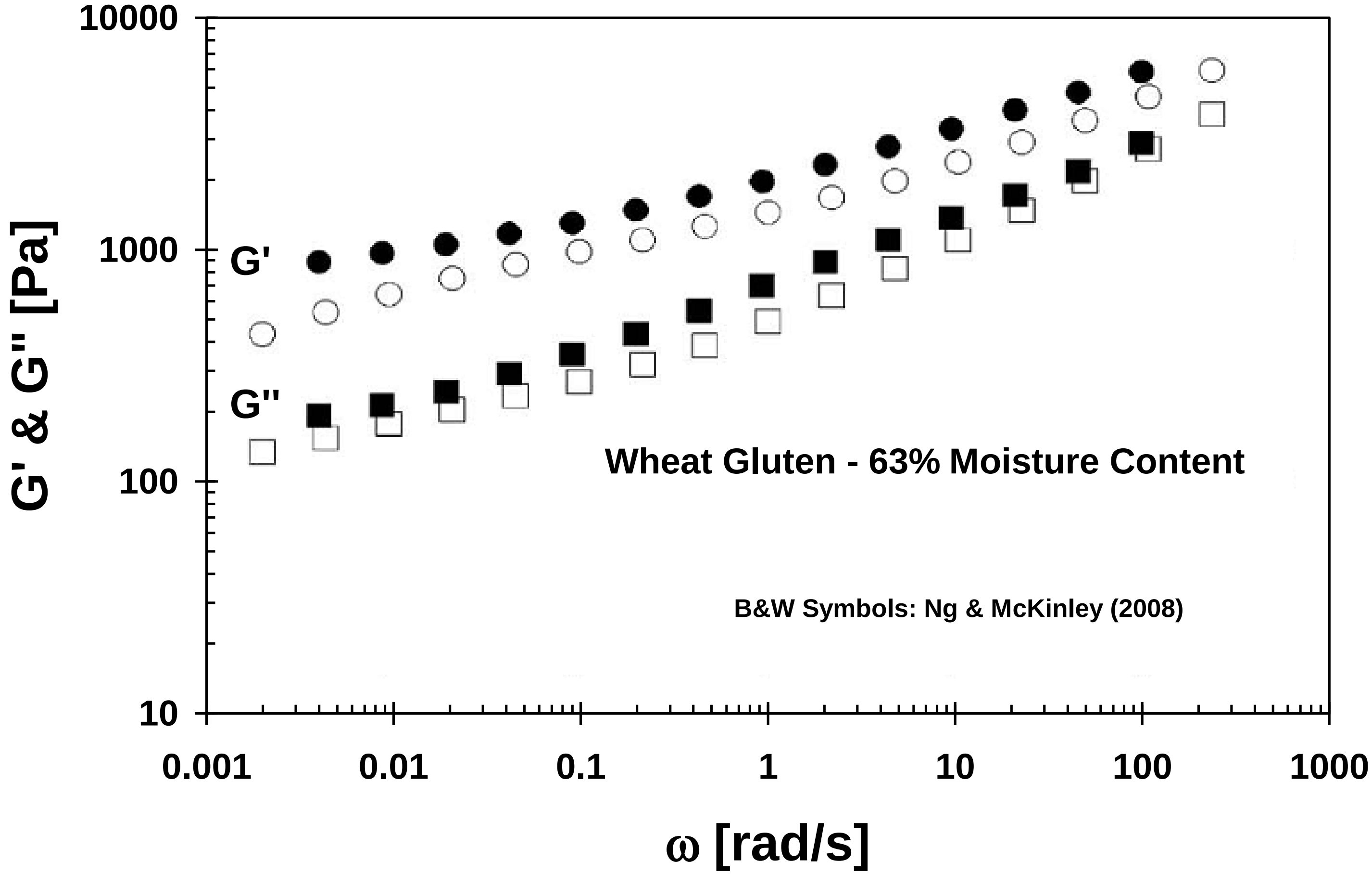


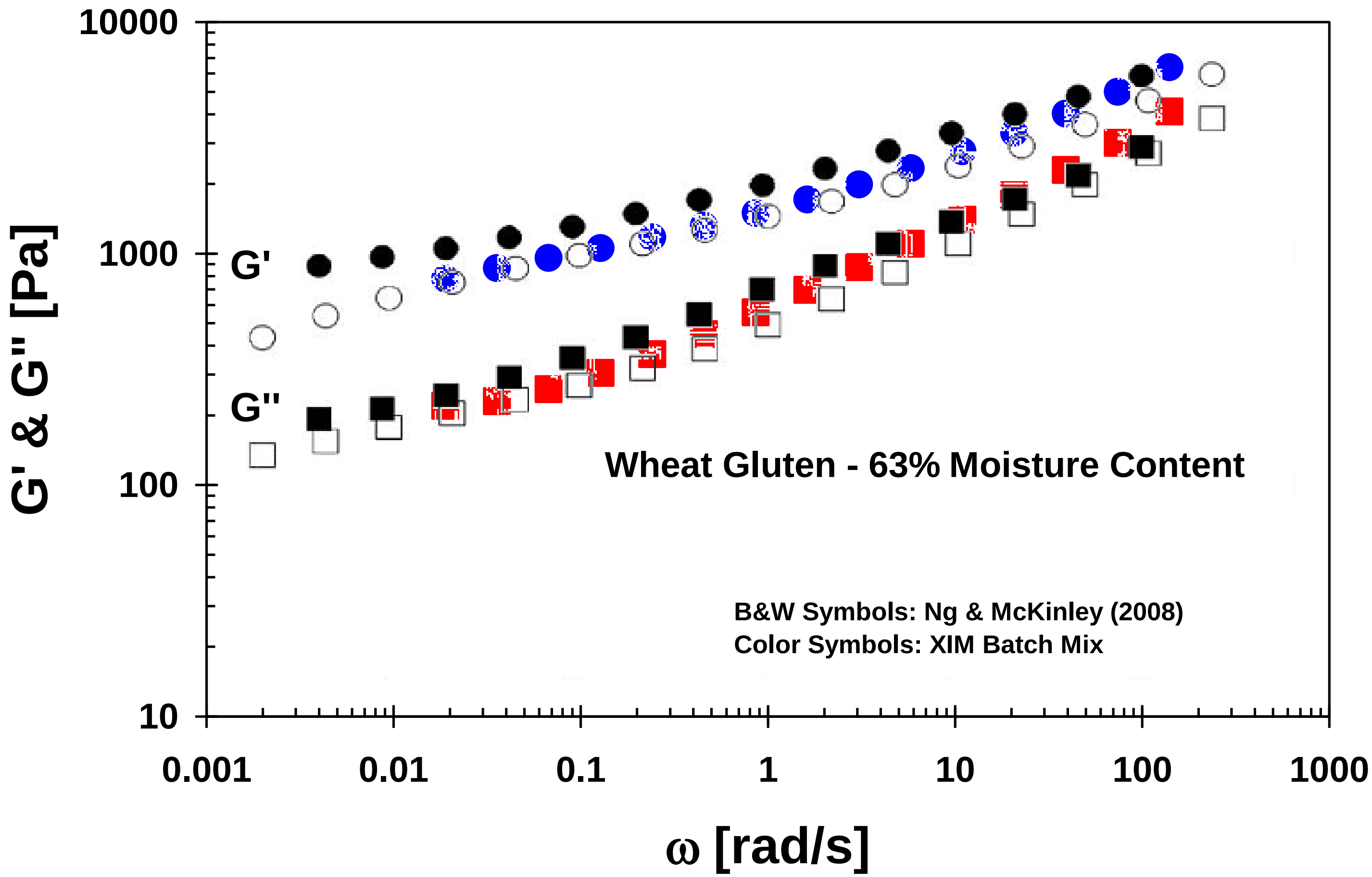
XIM dual sigma-blade mixer:
1.06g Gluten + 1.48g Water (80% Fill Factor)
15 rpm for 12 min



LVE Data Comparisons - Gluten

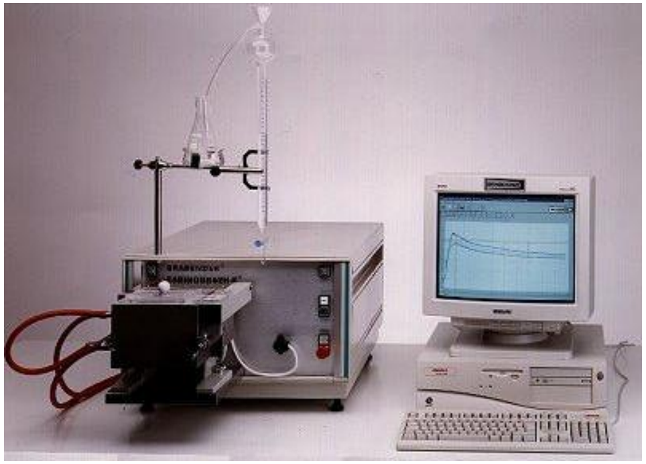


LVE Data Comparisons - Gluten

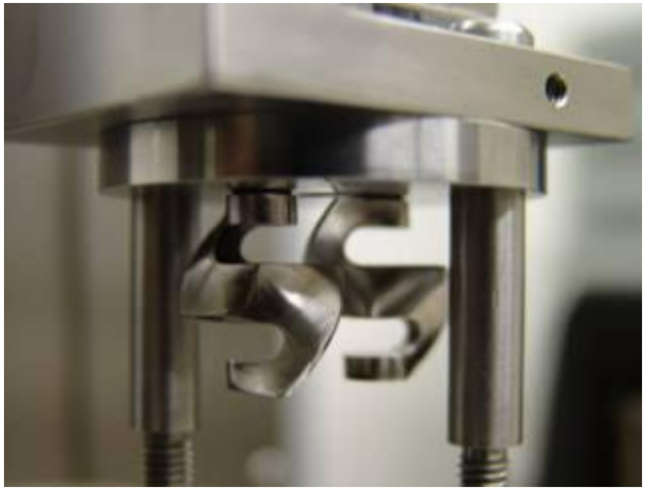


Mixing Comparison Experiments - Dough

- Material – Bread Dough
  - Gen. Purp. Wheat Flour (Robin Hood)
- Types of Lab Mixers used:
  - UBC – Brabender Farinograph Dual Sigma-Blade Mixer (1000 mL capacity)
  - XIM accommodated on a model SER2-P hosted on an Anton Paar MCR 501 (Dual Sigma-Blade Rotors – 4.0 mL capacity)



Brabender Farinograph (1000mL)

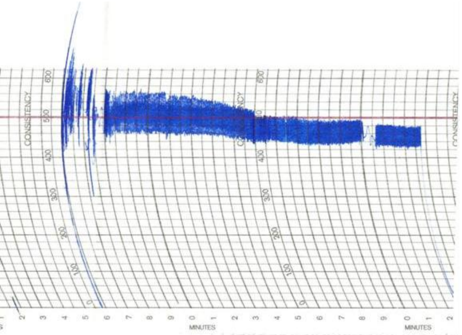


XIM dual sigma-blades (4mL)

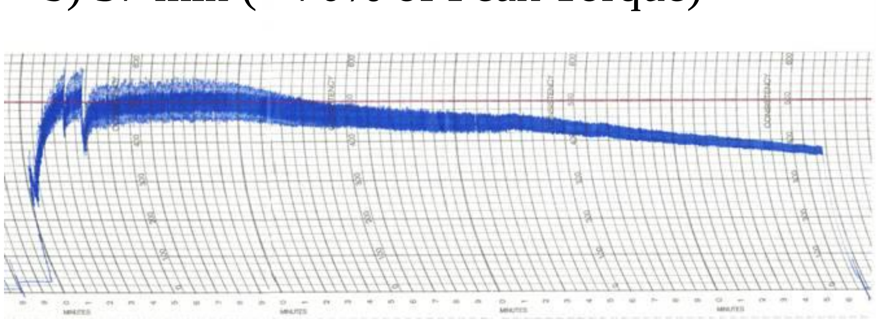
Torque Curve Comparisons - Dough

Farinograph Mix:
300g Flour (38% Moisture Content)
66 rpm for:

a) 12 min (~ 90% of Peak Torque)



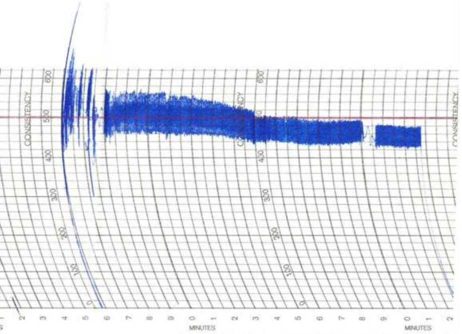
b) 37 min (~ 70% of Peak Torque)



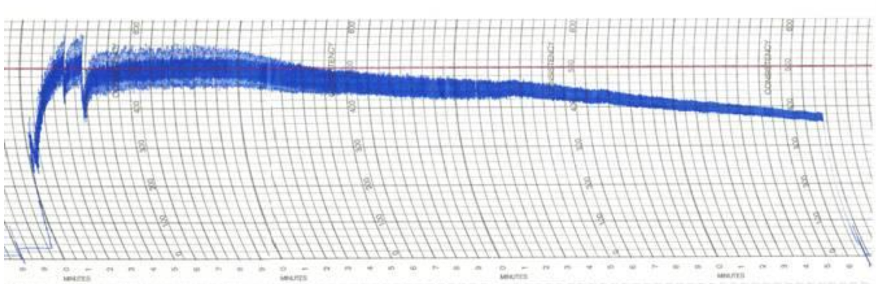
Torque Curve Comparisons - Dough

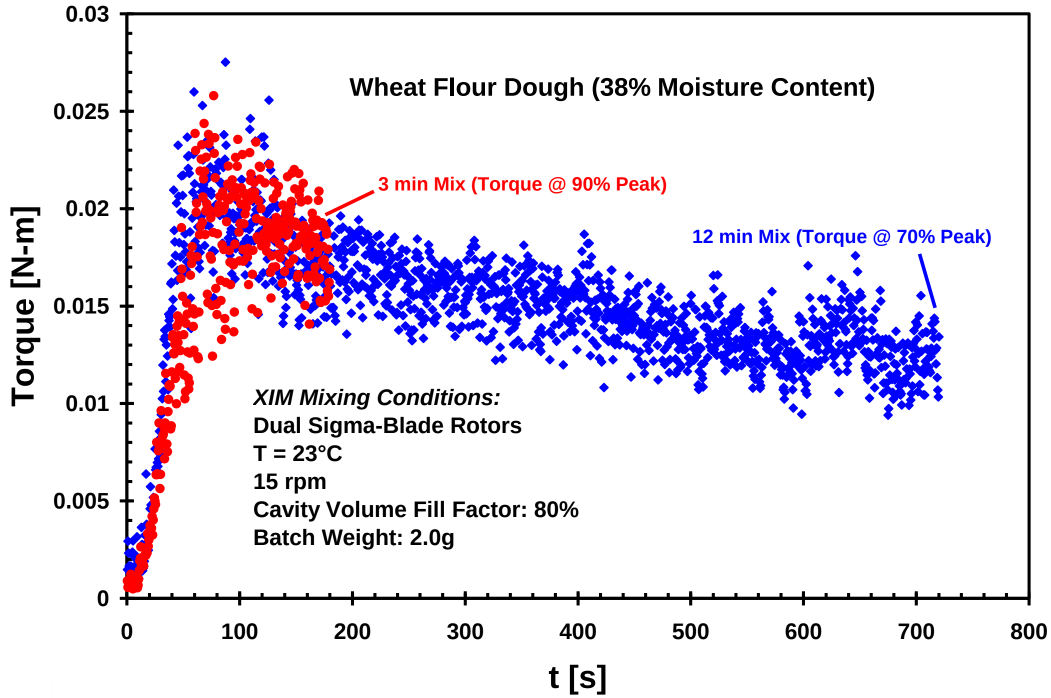
Farinograph Mix:
300g Flour (38% Moisture Content)
66 rpm for:

a) 12 min (~ 90% of Peak Torque)



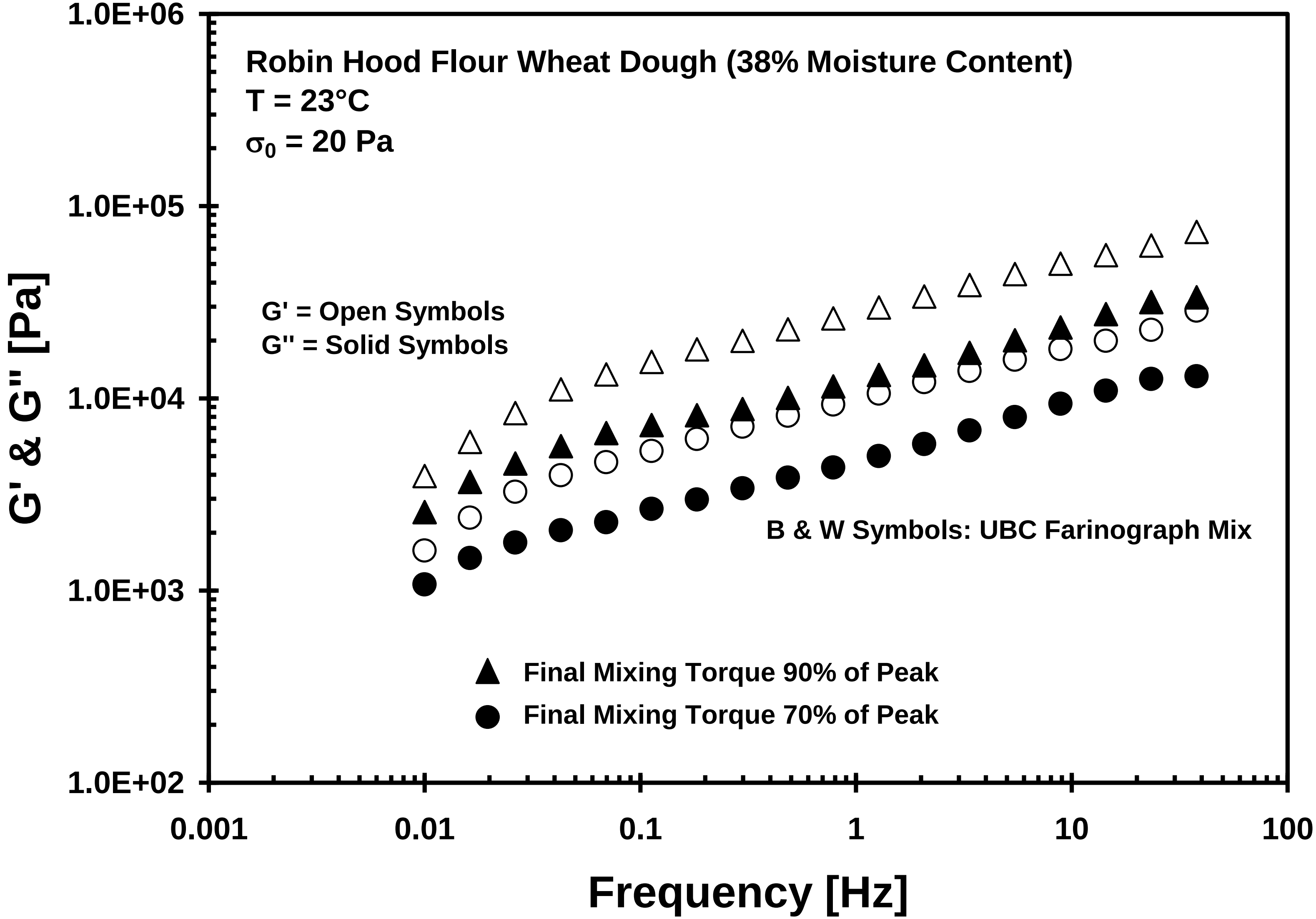
b) 37 min (~ 70% of Peak Torque)



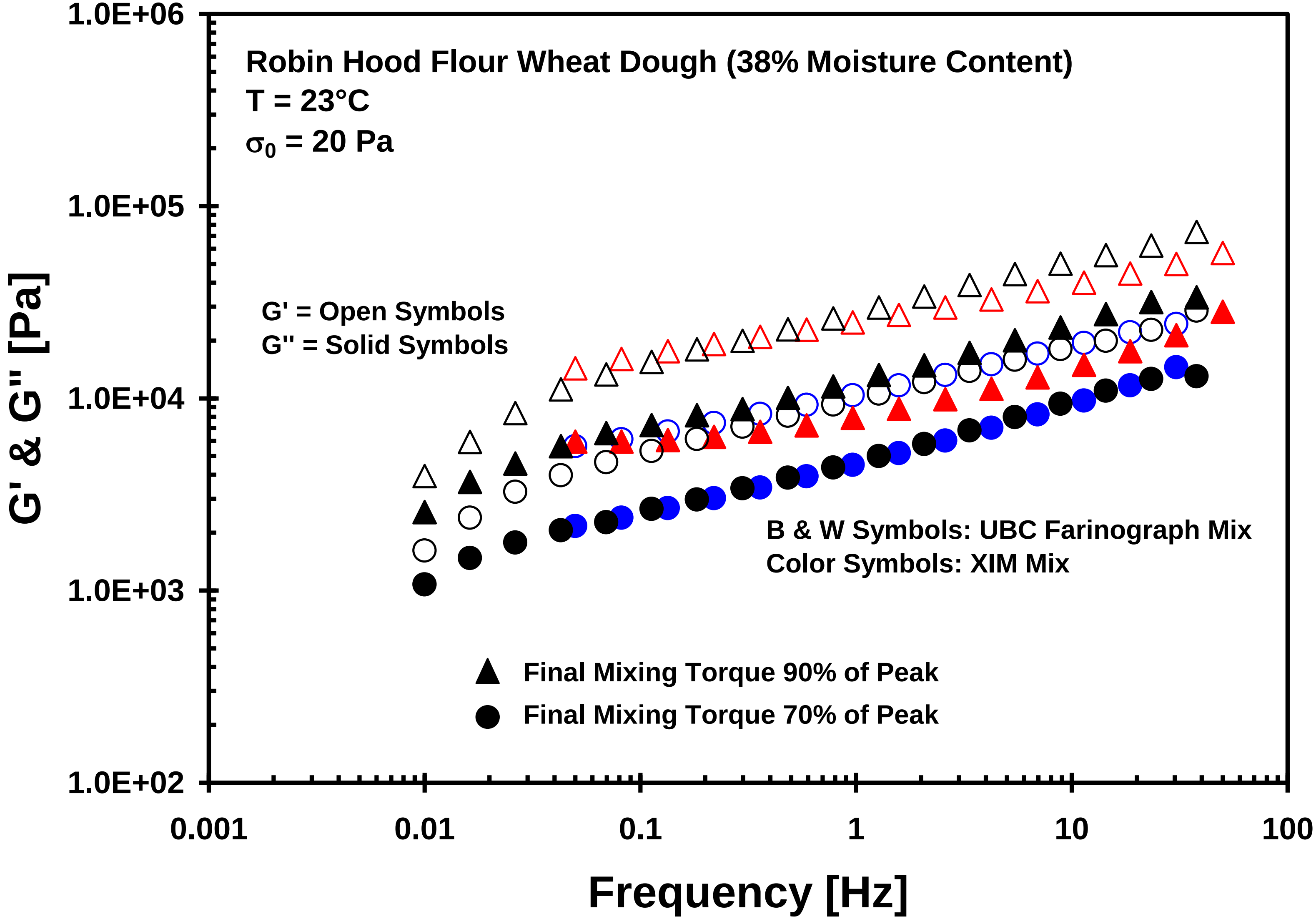


XIM dual sigma-blade mixer:
1.24g Flour + 0.76g Water (Fill Factor: 80%)
15 rpm for:
a) 3 min (~ 90% of Peak Torque)
b) 12 min (~ 70% of Peak Torque)

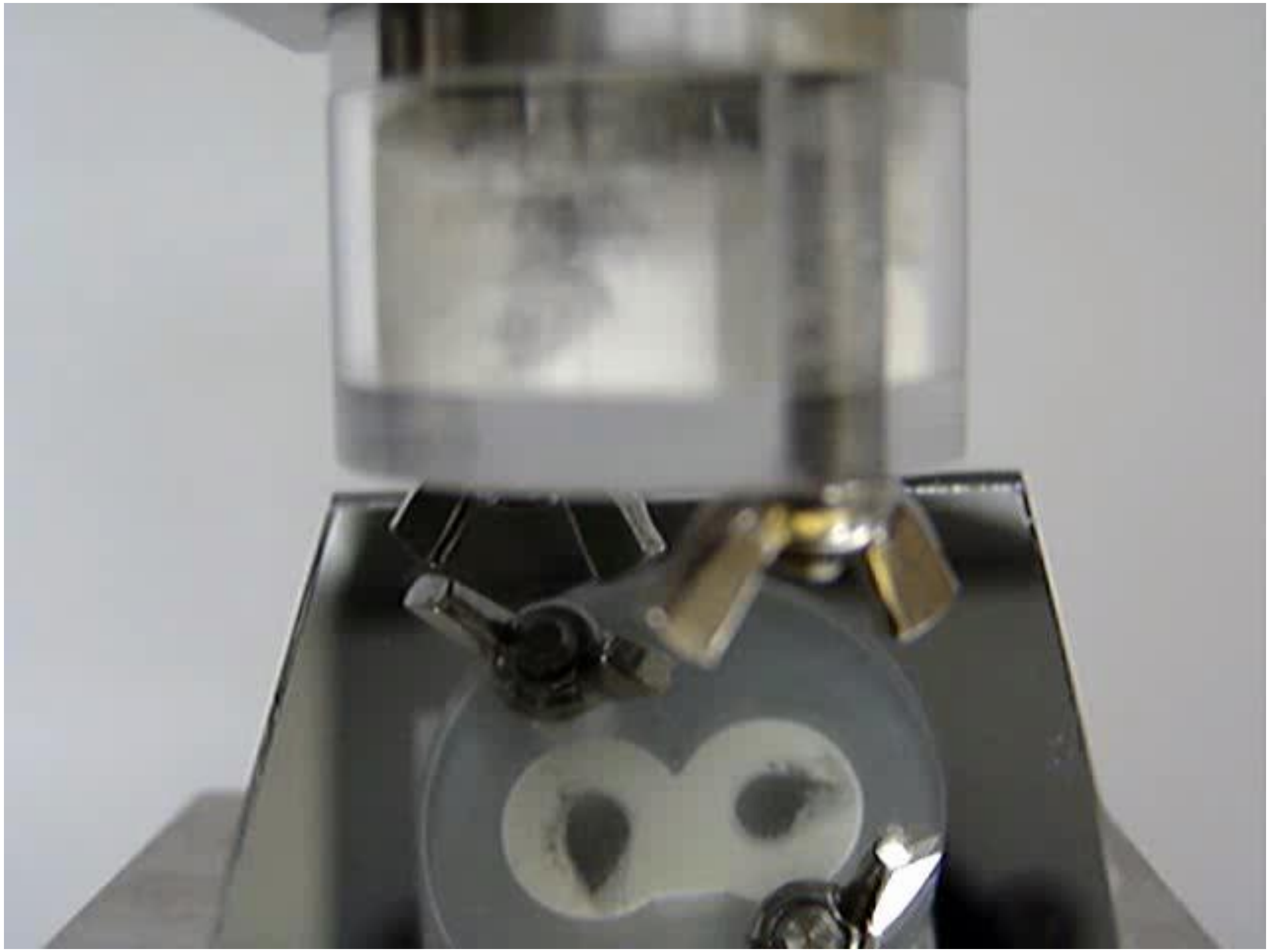
LVE Data Comparisons - Dough



LVE Data Comparisons - Dough



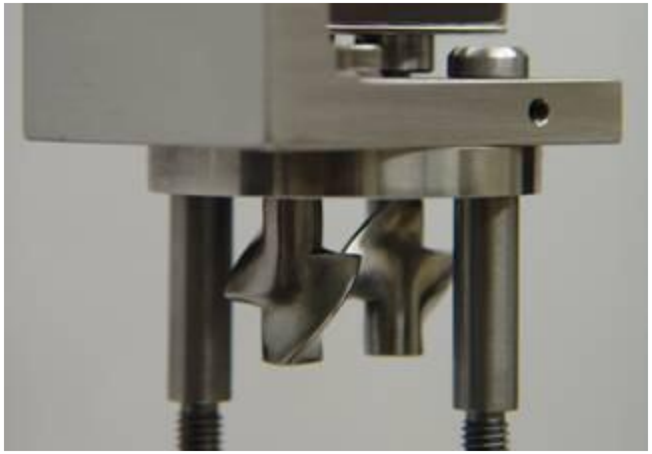
Observing the Mixing Process



- With a polycarbonate mixing chamber on the XIM, the mixing process is visible throughout the entire operation

Mixing Comparison Experiments – Polymer Blends

- Material – Linear/Branched PE (50/50)
  - Exact 3128 (m-LLDPE; Exxon Mobil) / EF606 (LDPE; Westlake)
  - LL3001.32 (LLDPE; Exxon Mobil) / 662i (LDPE; Dow)
- Types of Lab Mixers used:
  - Single screw laboratory extruder
  - Haake Rheograph 3000 (300 mL capacity)
  - XIM accommodated on a model SER2-P hosted on an Anton Paar MCR 501 (Dual 2-Wing Rotors – 3.75 mL capacity) and accommodated with a band heater for independent temperature control of mixing chamber



XIM dual 2-wing rotors (3.75mL)

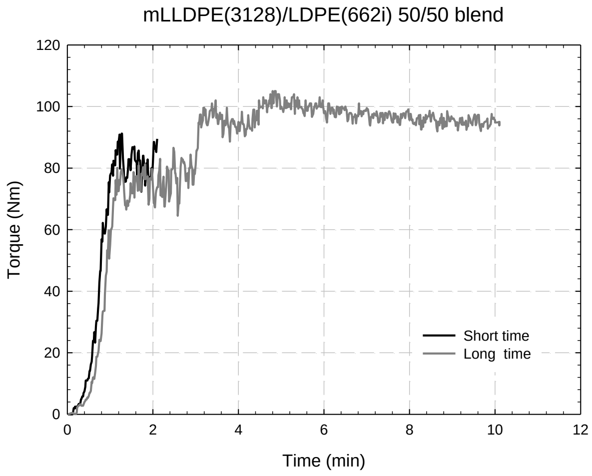
Band heater jacketing XIM chamber

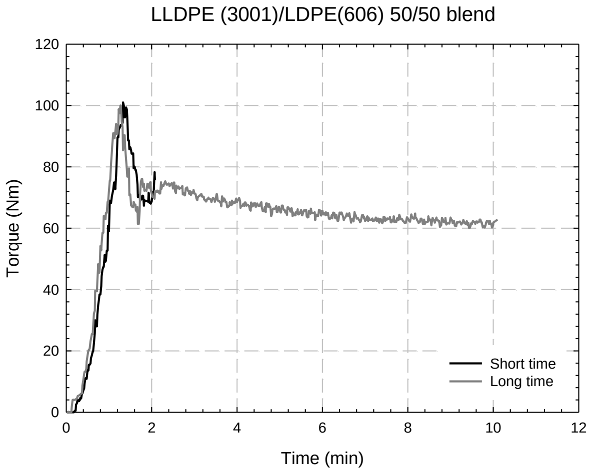
PE Resin Properties

Sample ID	Resin Type	Melt Index (g/10min) (190^{o}C)	Density (g/cc) (25^{o}C)	η_o (Pa.s) 150^{o}C
ZN-LLDPE- I	LL3001	1.0	0.917	17,488
m-LLDPE II	Exact 3128	1.3	0.900	11,383
LDPE-I	EF606A	2.2	0.919	44,234
LDPE-II	Dow 662I	0.47	0.919	127,900

Torque Curve Comparisons – PE Blends

Rheograph Mix:
T = 160°C
50 rpm





Torque Curve Comparisons – PE Blends

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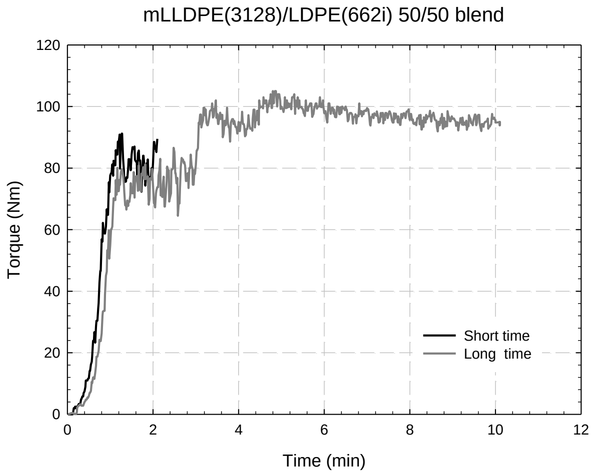
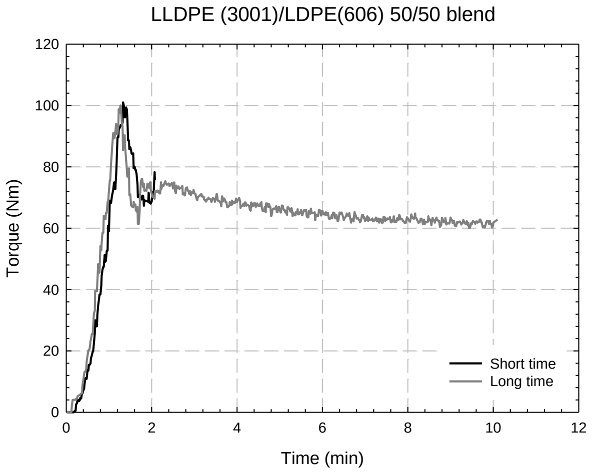
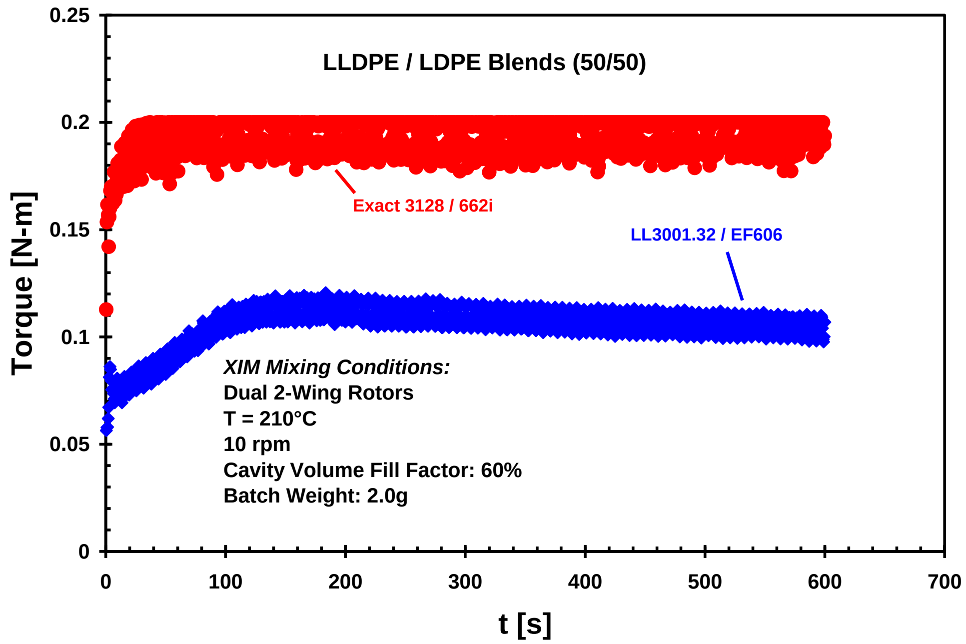
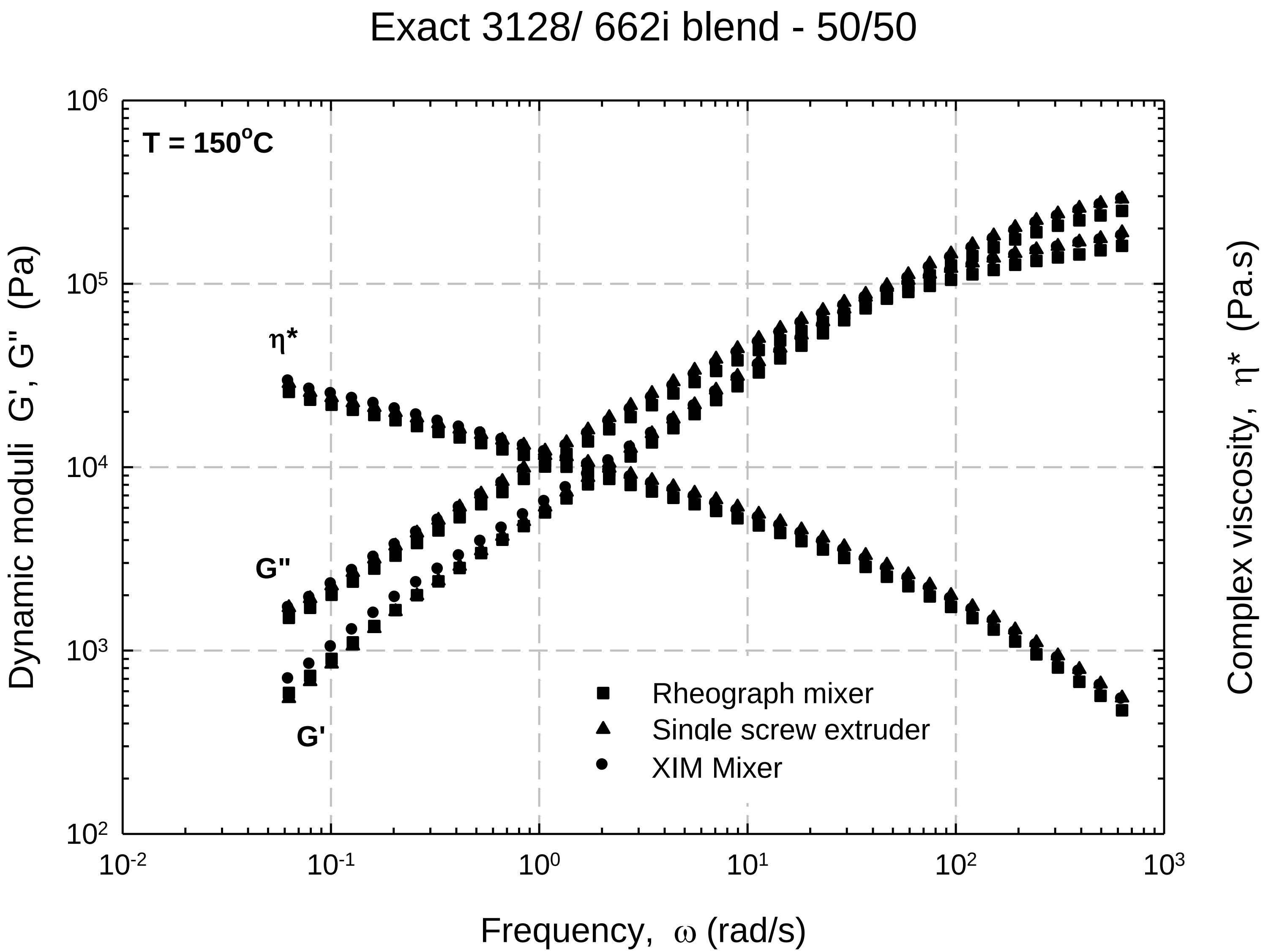




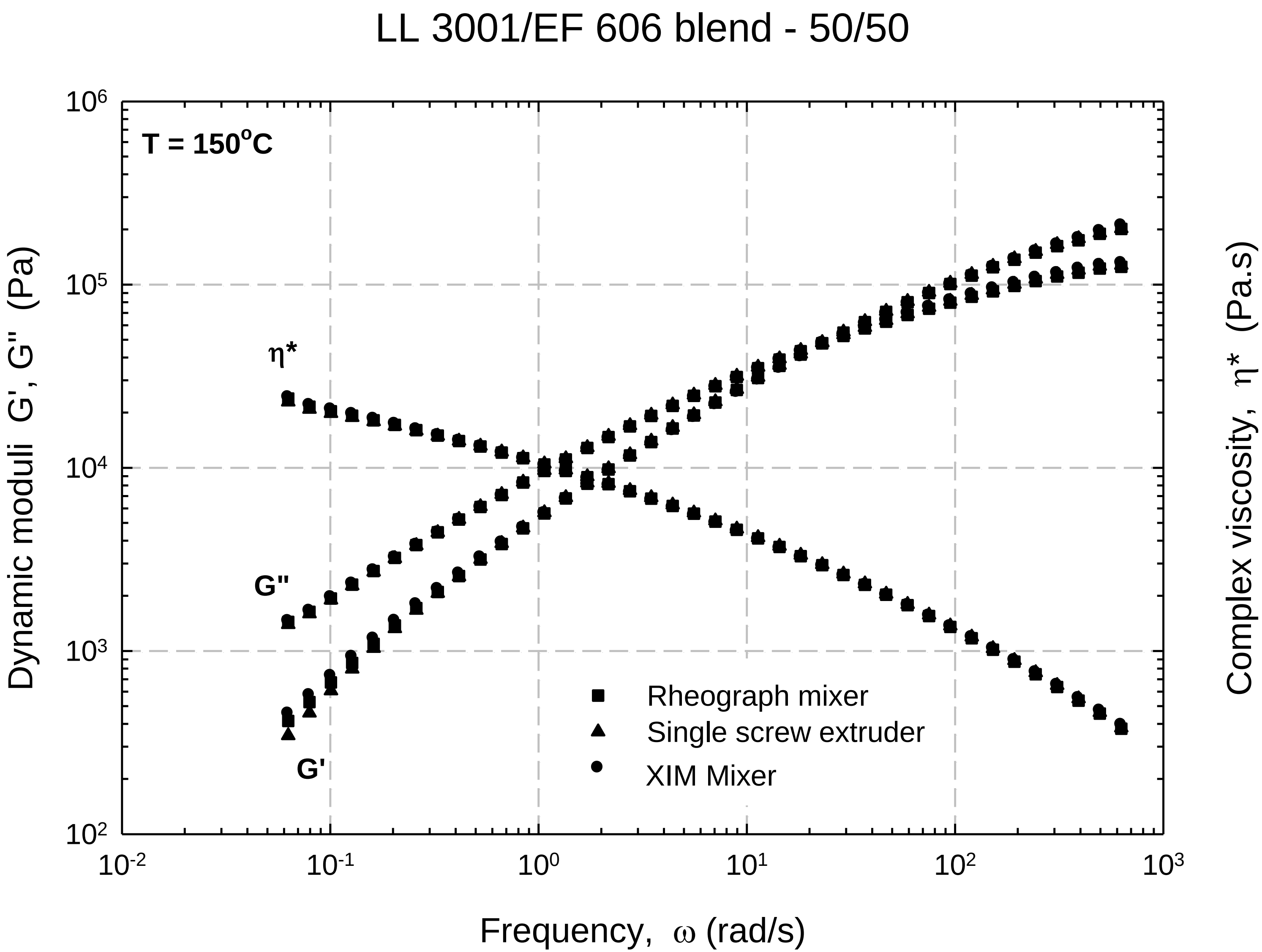
XIM Mix:
T = 210°C
10 rpm



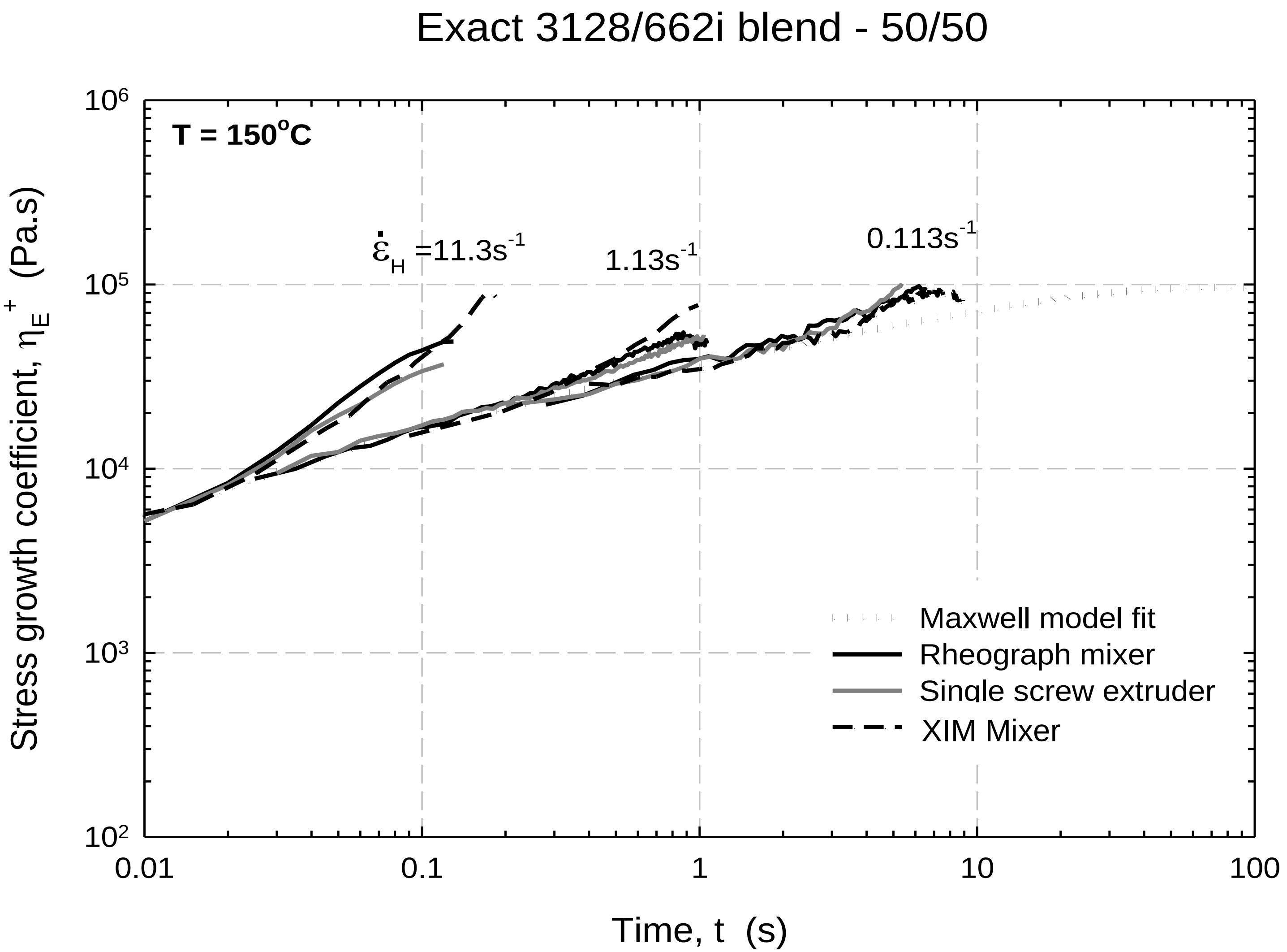
LVE Data Comparisons – 3128/662i



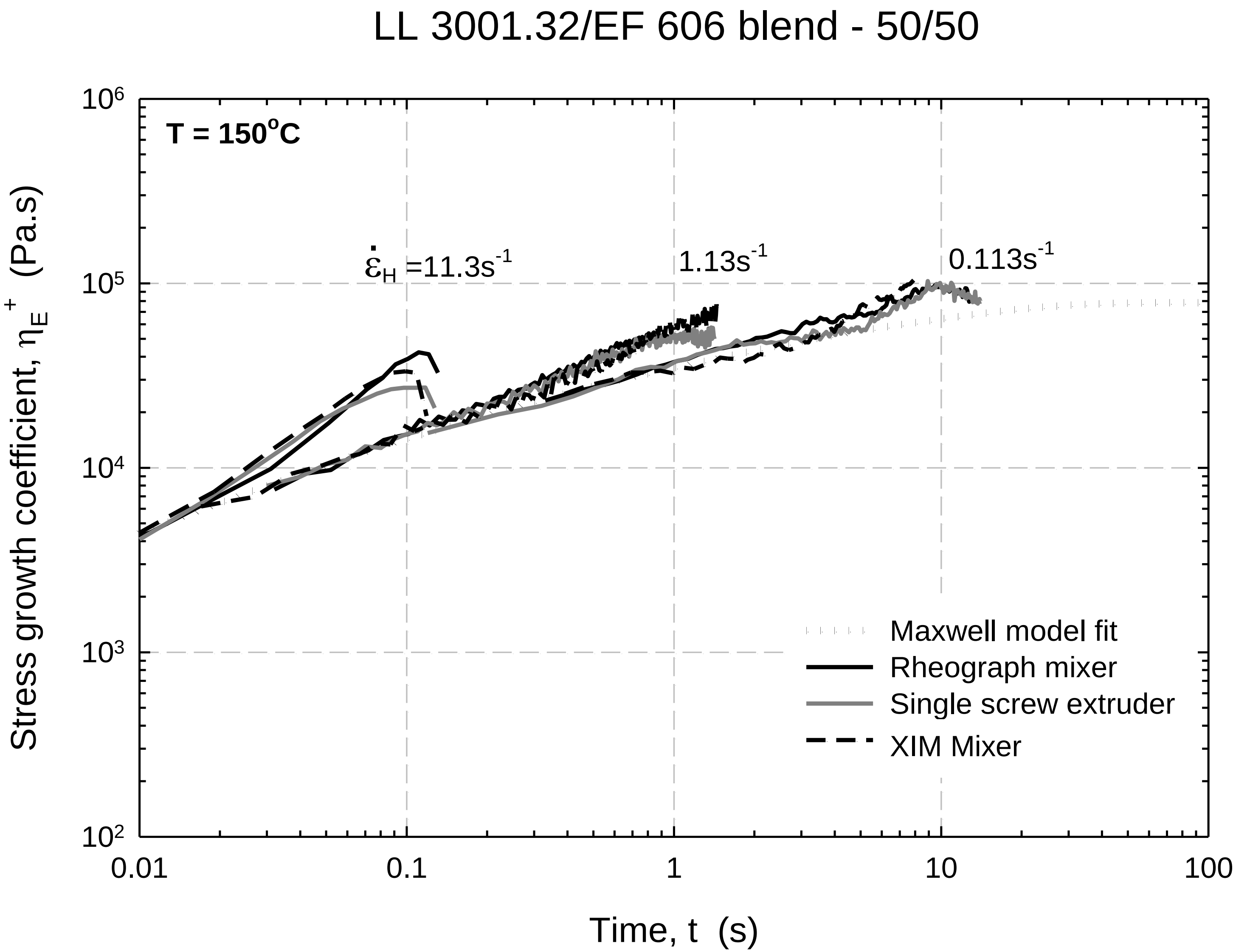
LVE Data Comparisons – 3001/606



Extensional Data Comparisons – 3128/662i

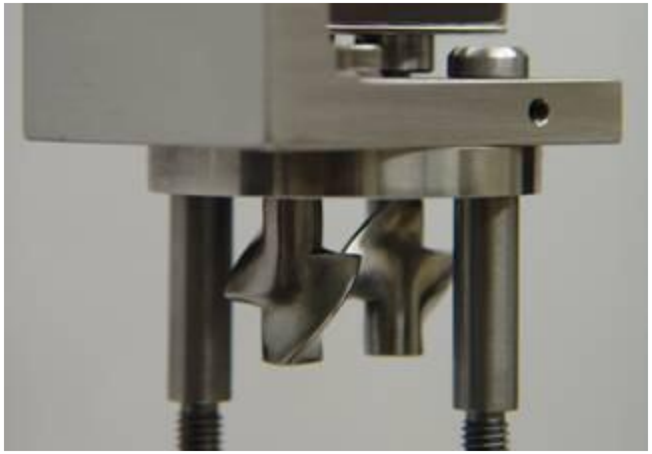


Extensional Data Comparisons – 3001/606



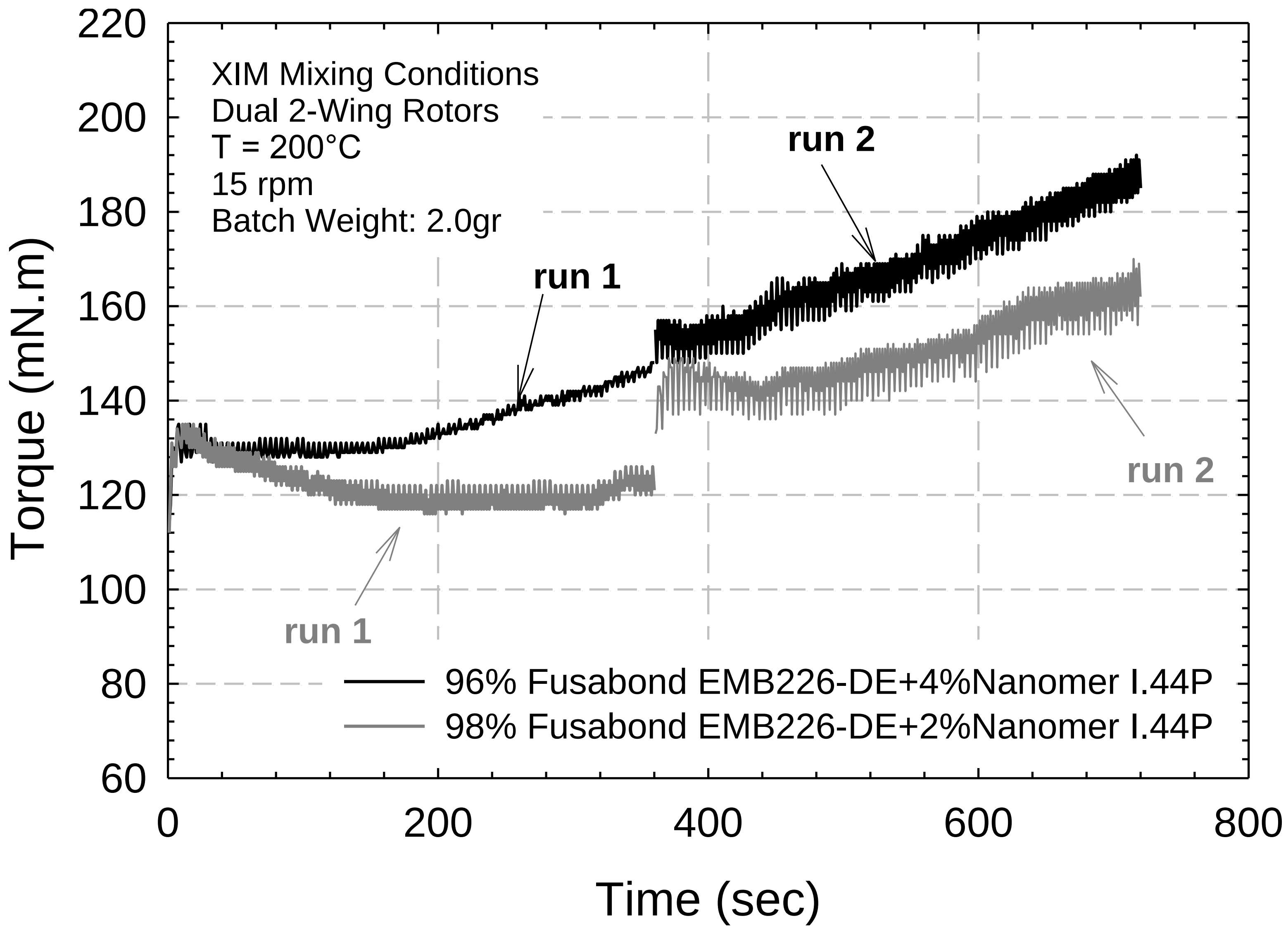
Mixing Comparison Experiments – Nanocomposites

- Material –PE/Nanoclay
  - Fusabond EMB-226DE (Maleic anhydride grafted-PE; Dupont)
  - Nanomer I.44 P (Montmorillonite clay; Nanocor Inc.)
- Types of Lab Mixers used:
  - Twin screw laboratory extruder (Micro 27, Leistritz [L/D = 36, D = 27mm], 200rpm, 10kg/hr, 200°C)
  - XIM accommodated on a model SER2-P hosted on an Anton Paar MCR 501 (Dual 2-Wing Rotors – 3.75 mL capacity) and accommodated with a band heater for independent temperature control of mixing chamber

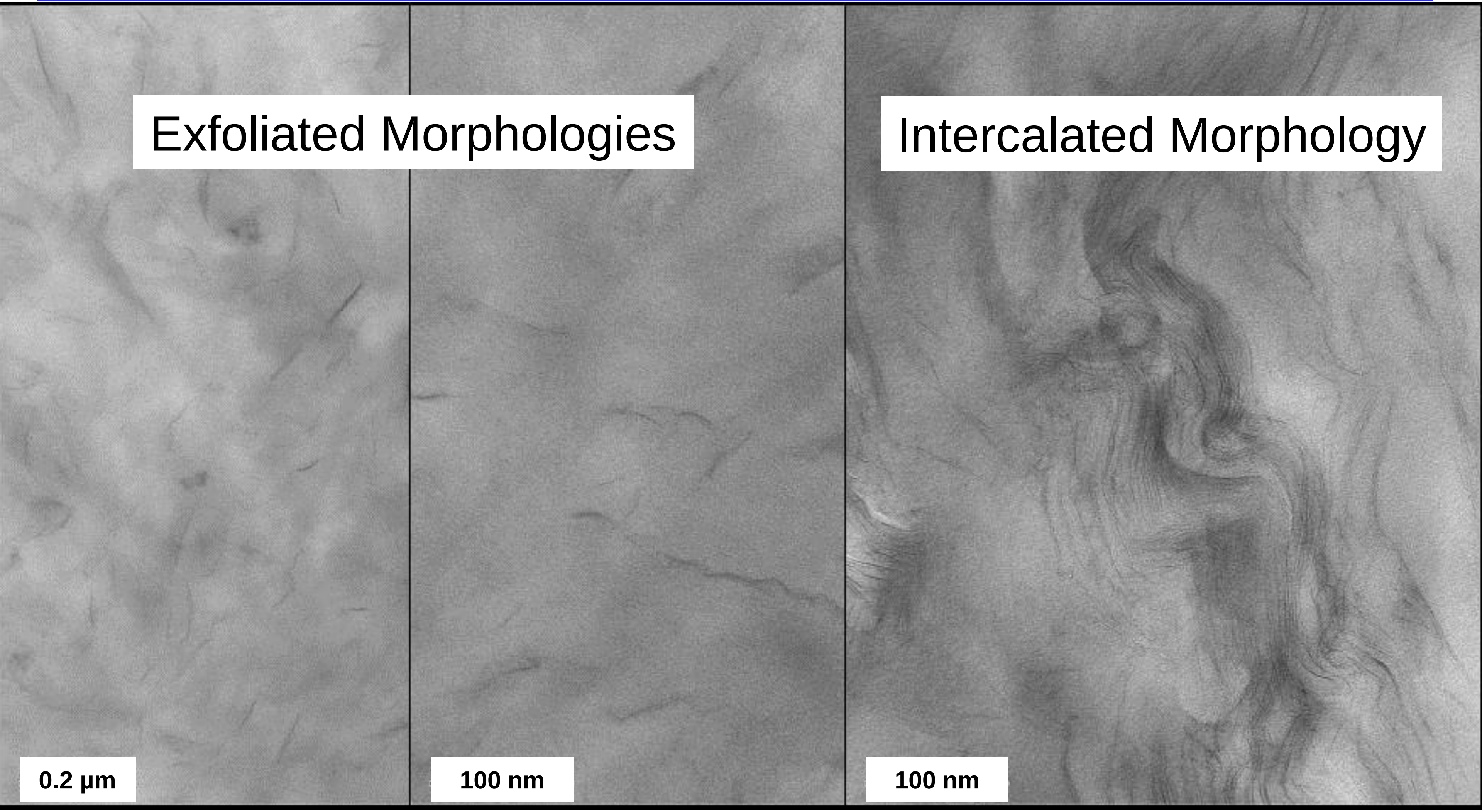

XIM dual 2-wing rotors (3.75mL)


Band heater jacketing XIM chamber

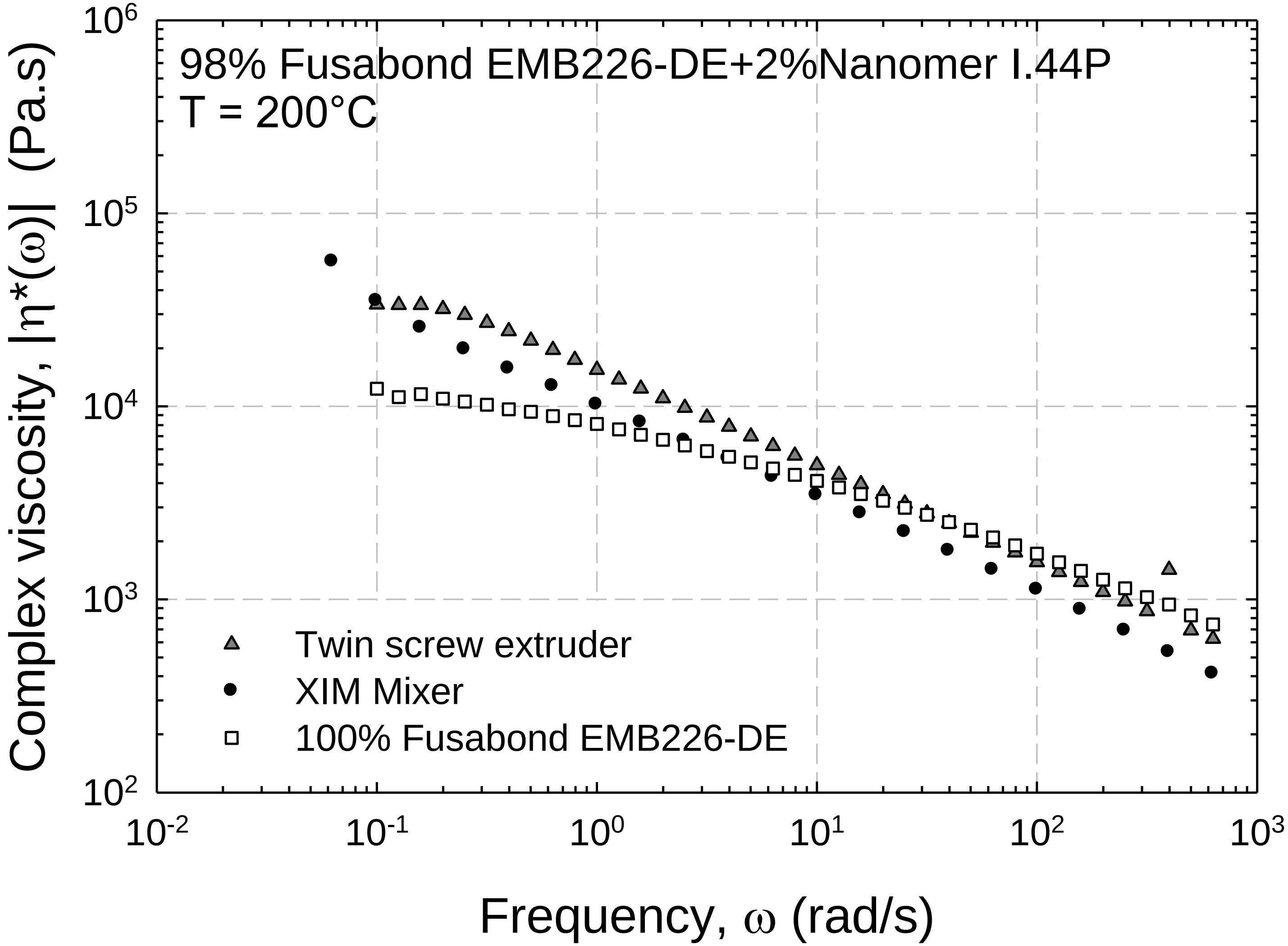
XIM Torque Curves – Nanocomposites



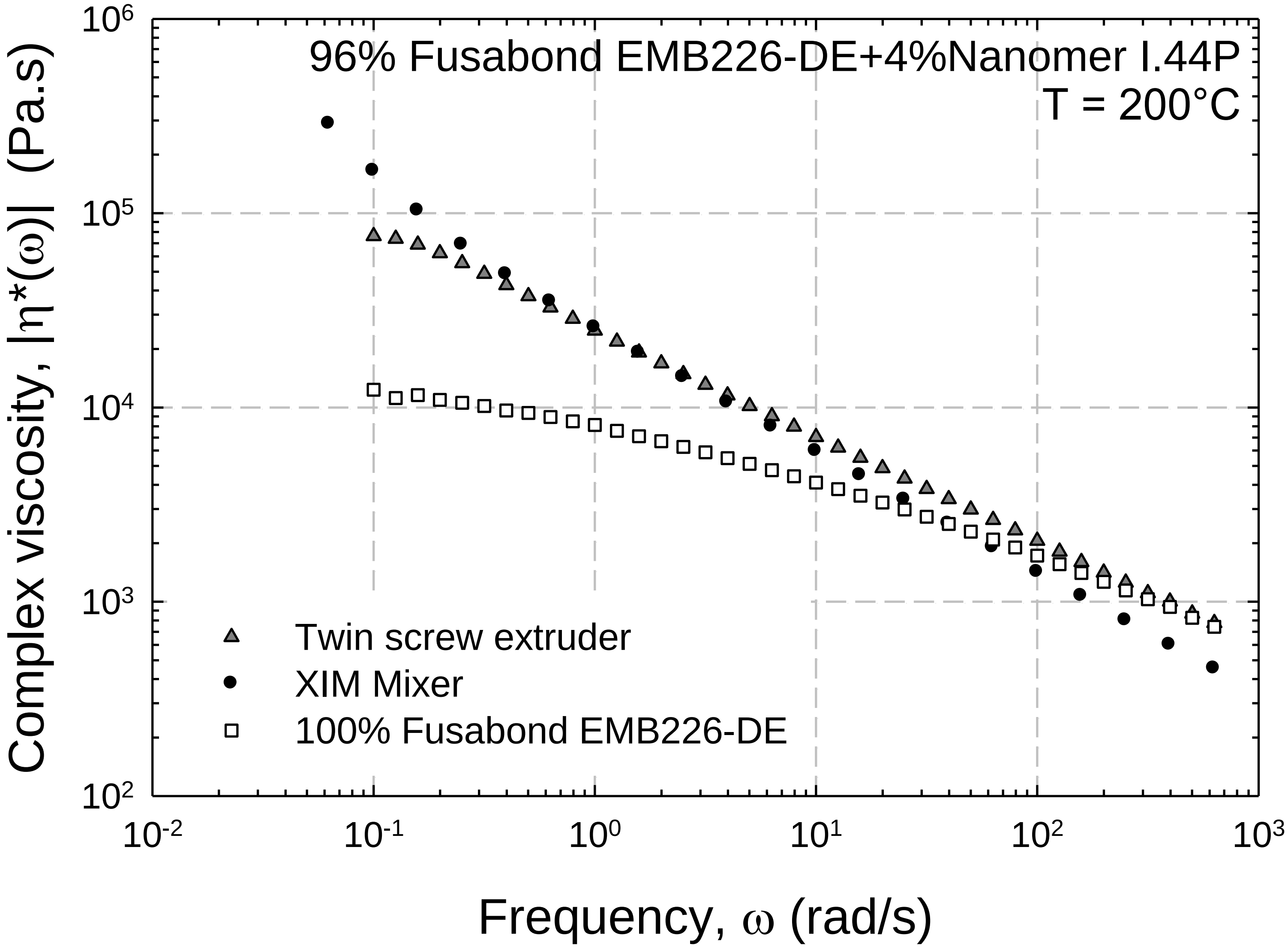
TEM Images – 96% Fusabond + 4% Nanoclay



LVE Comparisons – 98% Fusabond + 2% Nanoclay



LVE Comparisons – 96% Fusabond + 4% Nanoclay



Summary

- A novel miniature mixing device, the XIM, was developed for use in blending and compounding materials in batch sizes less than 5 mL in volume
- Rheological results on materials mixed with the XIM superpose with data for materials mixed on much larger scale mixing devices
- These results demonstrate that materials can be mixed and compounded at very small batch sizes with the XIM and have similar physical property characteristics with materials prepared with conventional compounding operations
- These results will appear in publication PE&S (2009)