Tacky Polymer-Modified H1 Greases and Their Low Temperature Fluidity

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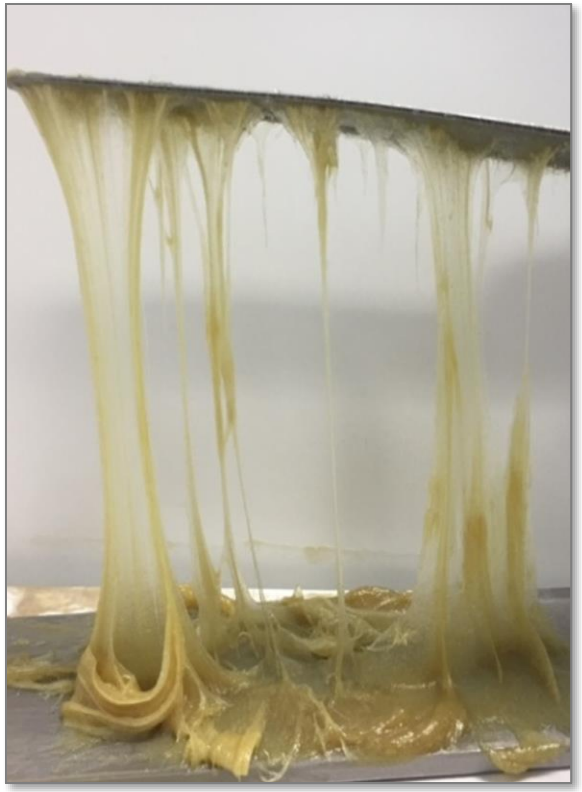
Outline

- Motivation
- Methods
- Materials
- Results on Tack and Low Temp
- Conclusions



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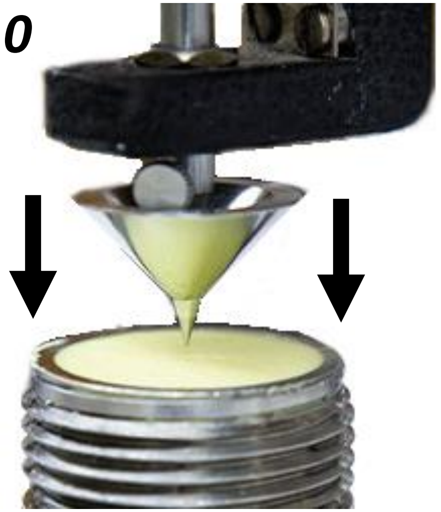
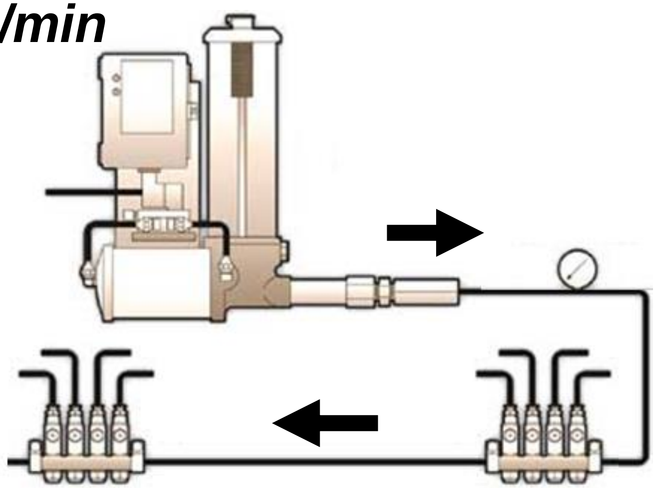
What Is Low Temperature Fluidity?

Torque	Consistency	Confined Flow
N·m 	***mm/10*** 	***g/min*** 
ASTM D4693 (NLGI GC-LB) IP 186 (DIN 51825) ASTM D1478 (**NLGI HPM**)	ISO 13737 (ISO 12925-3)	US Steel LT-37 (**NLGI HPM-LT**) Kest., DIN 51805-2 (**NLGI HPM-LT**) Lincoln Ventmeter (n/a)

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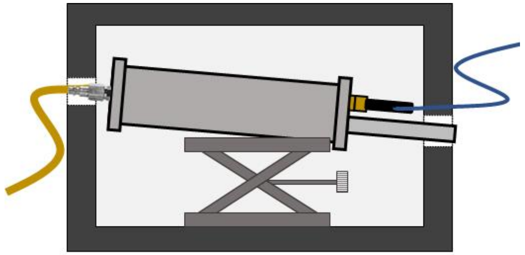
Methods - Low Temp Testing

LTT Ball Bearing, ASTM D1478

Torque Test

- Run at the NLGI HPM test lab @ -30°C



"Spindle Stall" Test, FPI

Consistency Test

- Steel rod in grease tube; 20 to -40°C
- Ad hoc viscometer, check for stalling

US Steel Mobility, LT-37

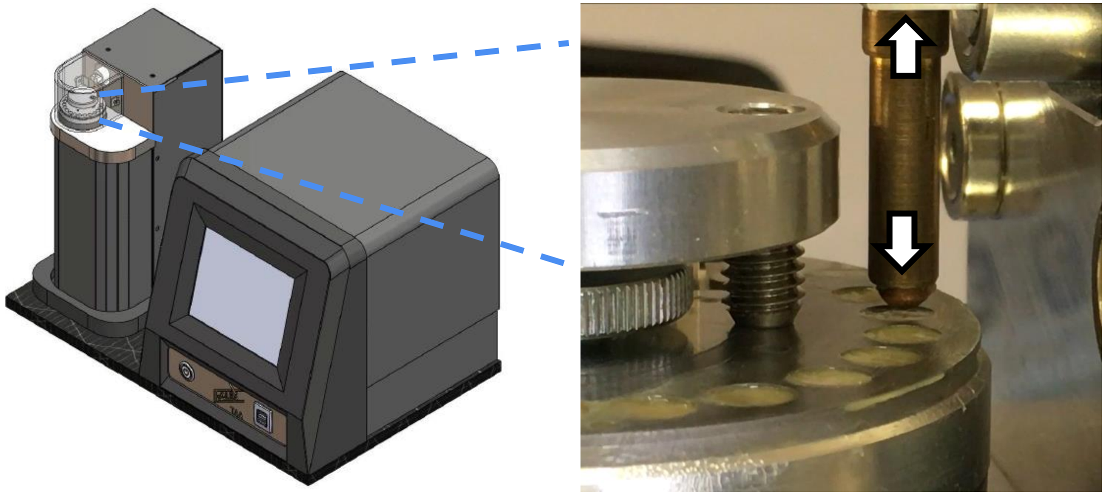
Confined Flow Test

- g/min flow, 0 to -20°C @ 150 psi
- Modified to run sideways to fit freezer

Methods - Tack Testing

Falex TAA

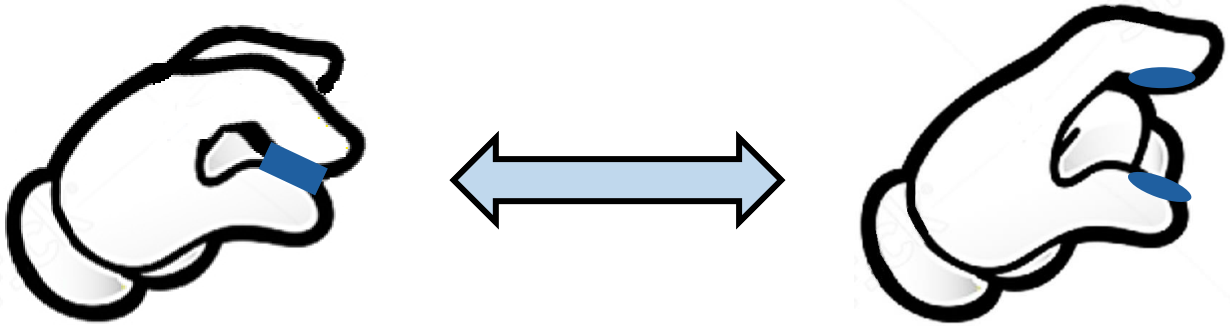
Tackiness Adhesion Analyzer



Probe w/ micro force gauge

15 sample wells on rotating carousel

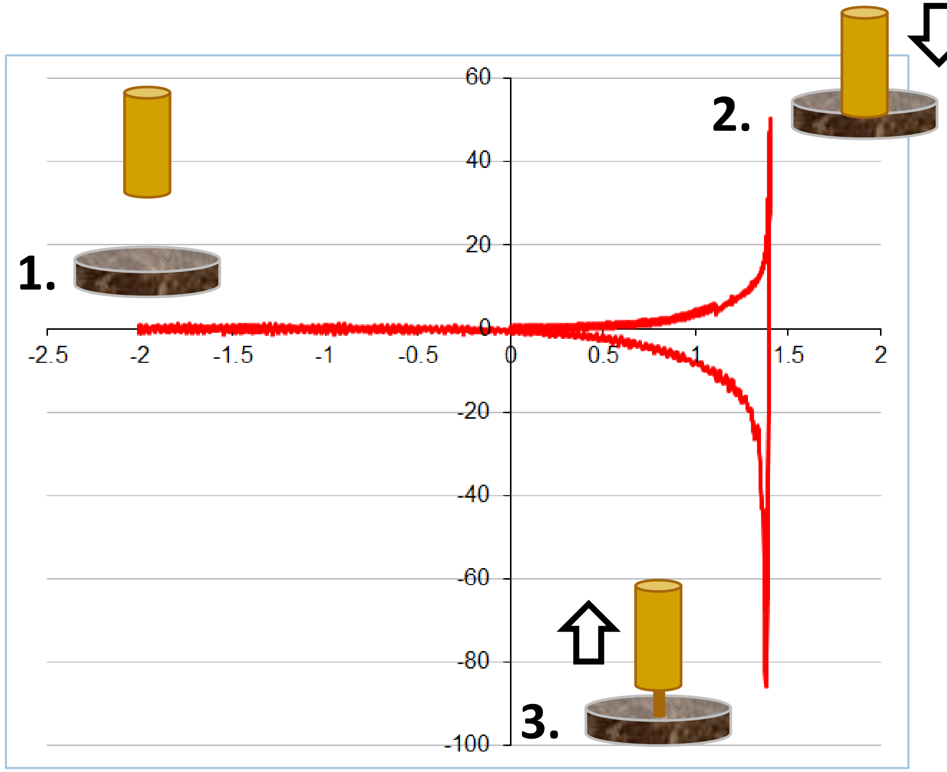
Visual Finger Tack Test



Read more in: 2019 ELGI AGM, Falex / Functional / Eldon's joint paper

Methods - TAA Output

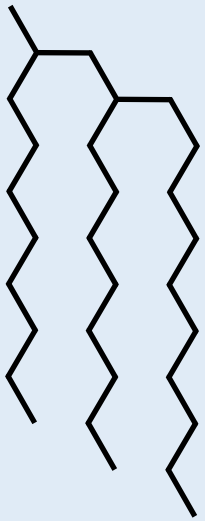
- Thread length, in mm
- Pull-off force, in mN
  - Peak force during retraction of probe
- Separation energy, in µJ
  - Force x Distance during retraction
- Compression energy, in µJ
  - Force x Distance during probe entering grease



Materials – ISO 150 Base Oil Blends

ISO 150 Base Blend

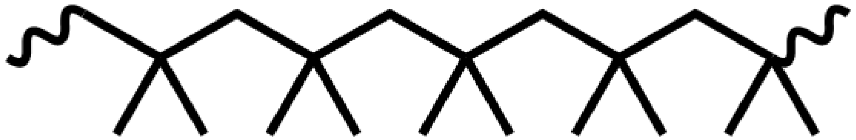
PAO 6 (80%)



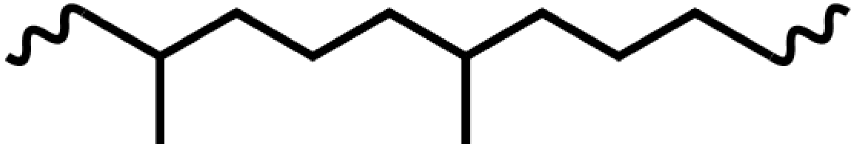
Diluent Oil (20-0%)

Polymer (0-20%)

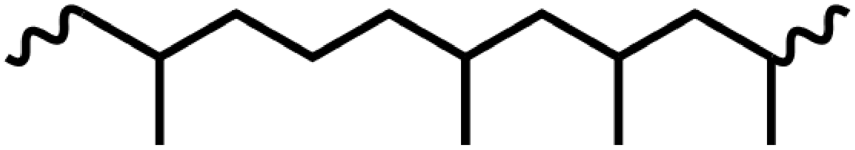
Polyisobutylene ("PIB")



Olefin Copolymer ("OCP")



Semi-Crystalline OCP ("CRY")



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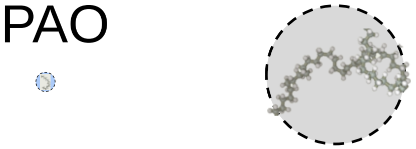
Materials – Polymer Selection

- 3 MW grades tested per polymer:

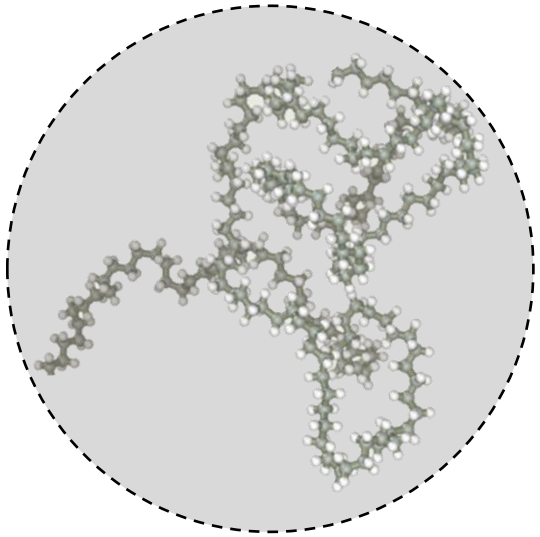
Oil/

PAO



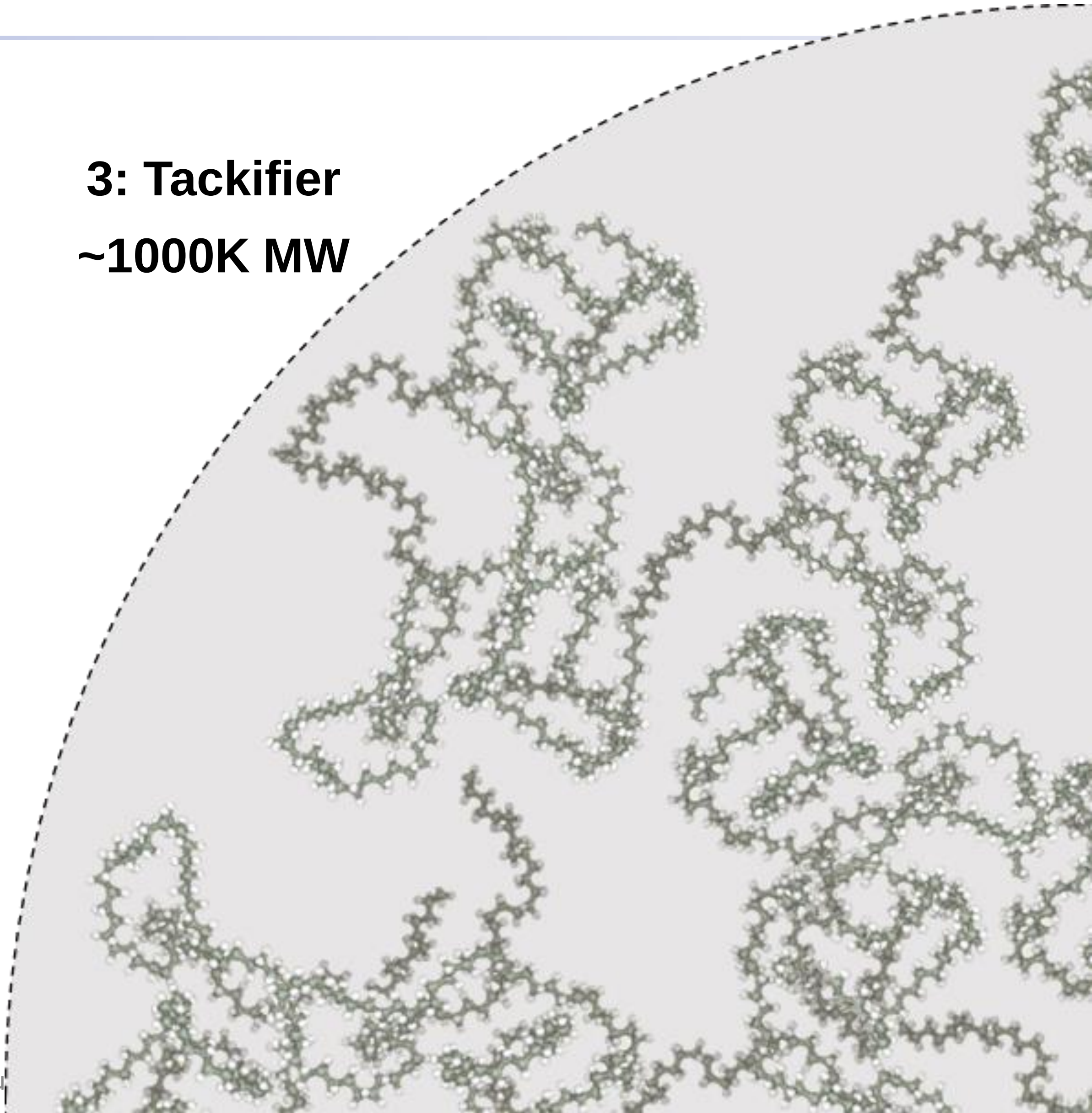
1: Basestock

~10K MW



2: Viscosity Modifier

~100K MW

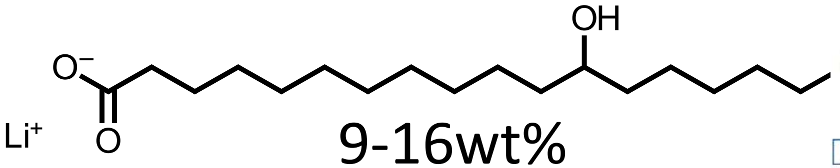


3: Tackifier

~1000K MW

Materials - Formulas

- Thickener: NSF HX-1 Lith12-HSA
- 9x experimental greases
- Control greases
  - 1x Syn. - ISO 150 mPAO
  - 2x Conv. - ISO 150 Gr.I, Naph.



9-16wt%

Nonfood Compounds
No. 163792

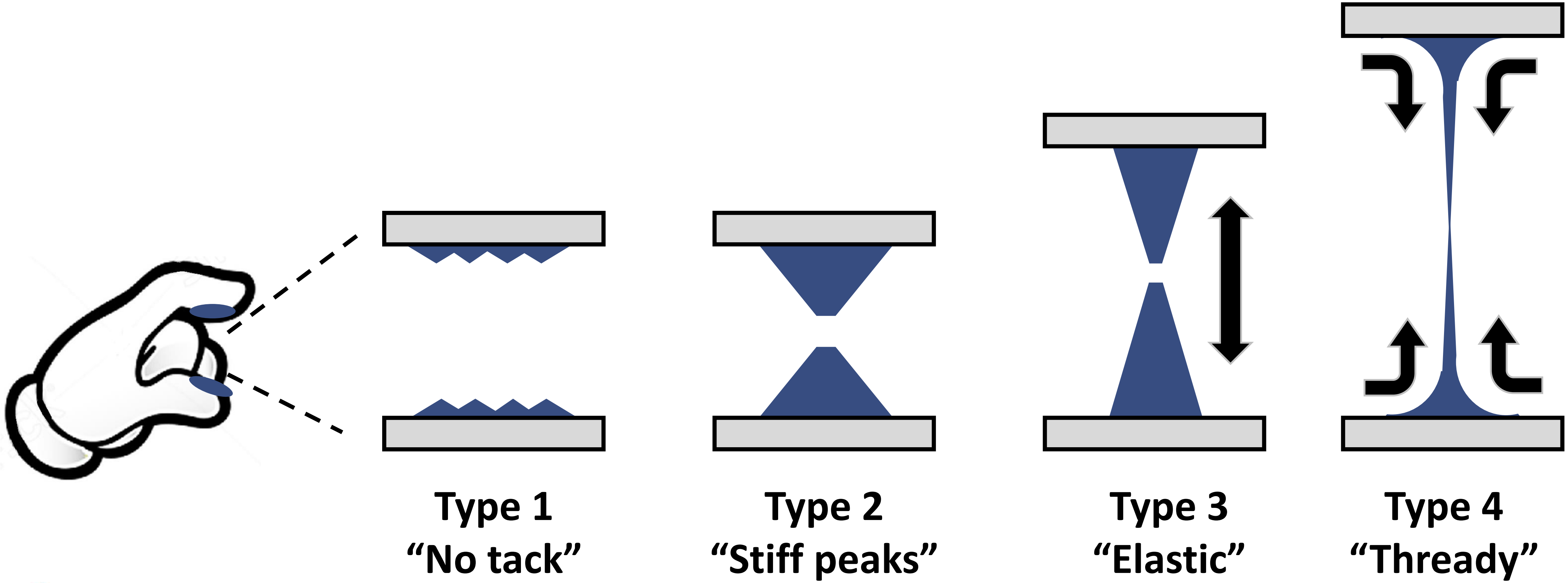
	Low MW / Basestock	Mid MW / VM	High MW / Tackifier
PIB	PIB1	PIB2	PIB3
OCP	OCP1	OCP2	OCP3
CRY	CRY1	CRY2	CRY3

Quantitative TAA vs. Qualitative Finger Tack

- TAA can probe grease and measure push/pull forces in infinitely different ways
  - Which test conditions are ‘correct’ or ‘useful’?
  - Tackiness is visual and tactile – TAA ought to reproduce what we see

Finger Tack Types

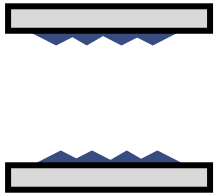
- Greases exhibit four subjective 'types' of finger tack behavior



Types by Grease Composition

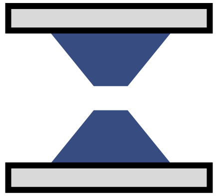
No Polymer / Low MW

- Petroleum controls



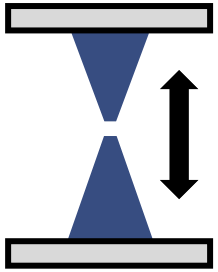
Type 1
"No tack"

- mPAO control
- PIB1, OCP1, CRY1



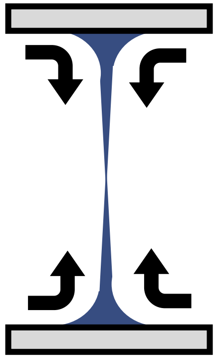
Type 2
"Stiff peaks"

Medium/High MW Polymers



Type 3
"Elastic"

- CRY2, CRY3
- PIB2



Type 4
"Thready"

- OCP2, OCP3
- PIB3

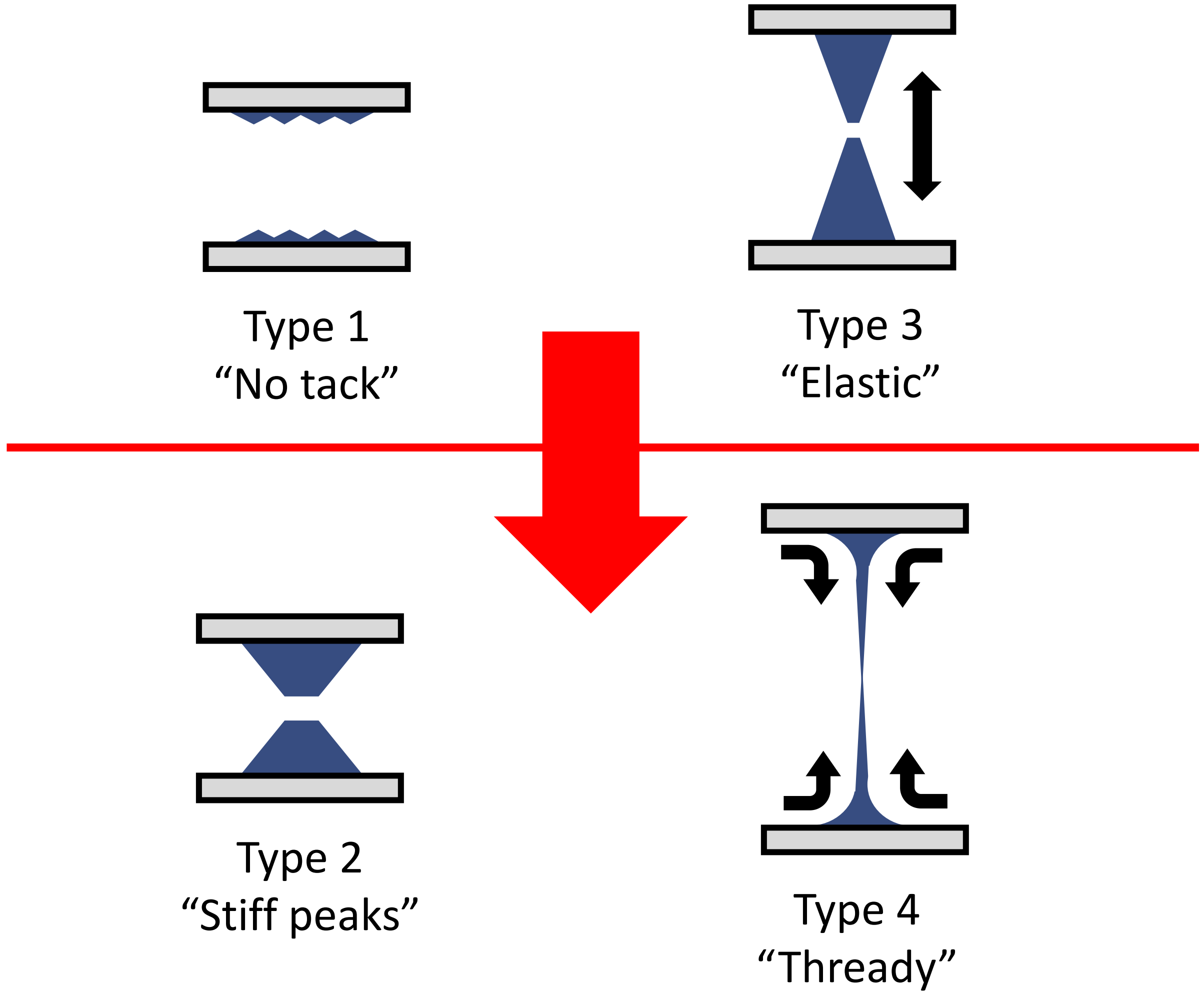
Types vs. TAA Probe Speed

Newtonian Behavior

- If speed changes then forces and energies remain similar
- Low compression energy (non-damping)

Non-Newtonian Behavior

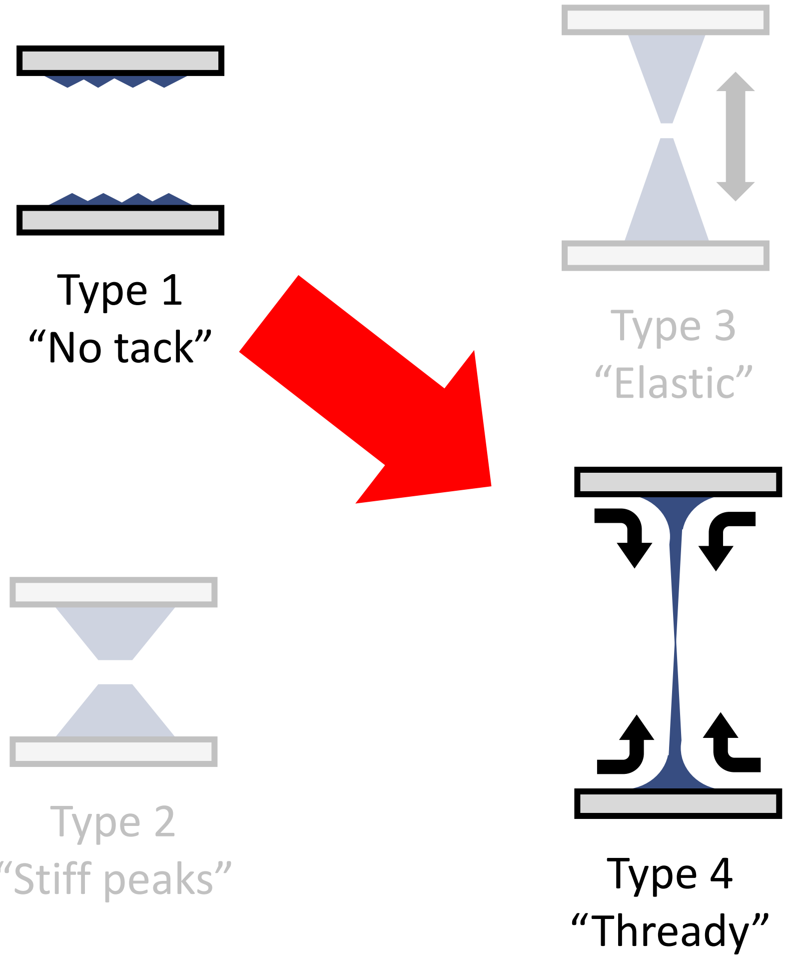
- If speed changes then forces and energies change (speed dependent)
- High compression energy (viscoelastic damping)



Types vs. TAA Pull-Off and Thread Length

- Highest pull-Off force
  - **High adhesion**

- Lowest thread lengths
  - **Low cohesion**



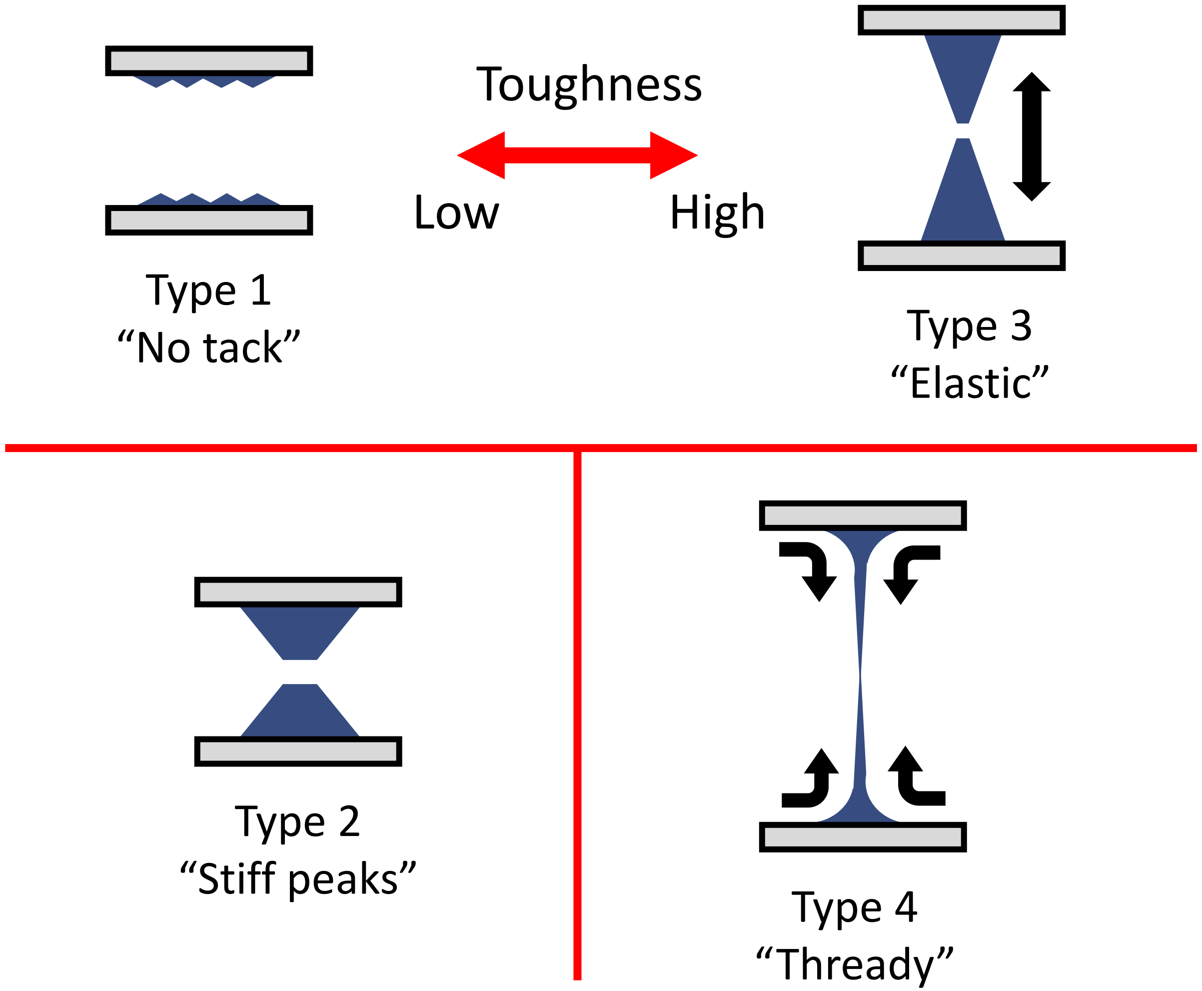
- Lowest pull-off force
  - **Low adhesion**

- Highest thread lengths
  - **High cohesion**

Further Differentiation

- **Type 1** from **Type 3** measure similar forces and energies

- Related energetically but mechanically and visually different



- **Type 2** intermediate between **1&3** and **4**

- Highest thread length per unit sep. energy

- **Type 4** has uniquely low separation energy

- Most non-Newtonian

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Low Temperature Testing

- LT-37 appears to be the most stringent to pass

	LT-37 US Steel @-20°C(g/min)	**"Spindle Stall"** @ -40°C(RPM)	**ASTM D1478** Start Torq. @-30°C(mNm)	1Hr Torq. @-30°C(mNm)
HPM-LT limit	> 10	N/A	< 1000	< 100
PIB1	58.2	1.0	96.3	30.6
PIB2	14.3	3.0	124	37.7
PIB3	7.1	1.5	121	30.6
OCP1	57.4	5.5	121	29.3
OCP2	175	10.5	36.4	16.6
OCP3	8.9	0.5	244	13.4
CRY1	0.0	0.0	N/A	N/A
CRY2	11.6	0.0	N/A	N/A
CRY3	6.4	0.0	N/A	N/A

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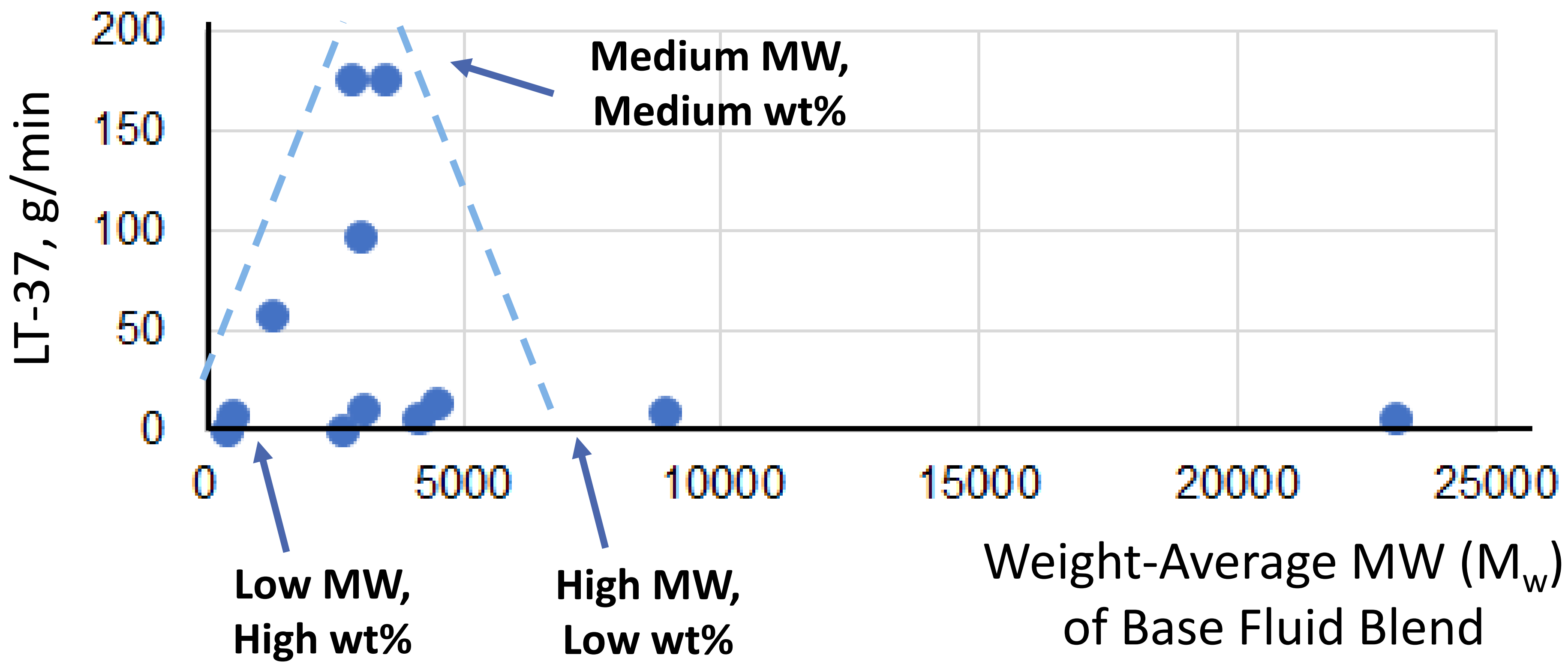
A Common Motif in Low Temp. Testing

- ‘Triangle plot’ feature in some data
- Not too much polymer, not too much MW – moderation is key



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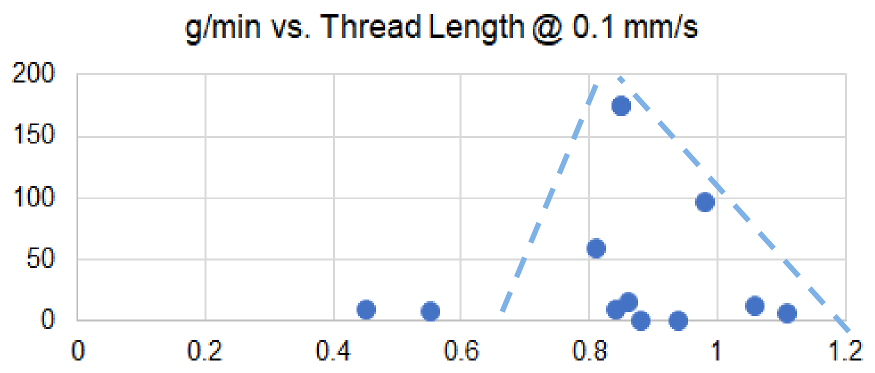
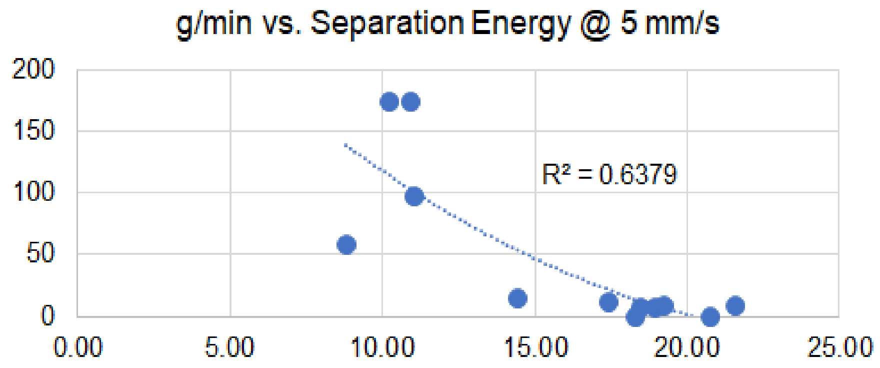
Tack vs. LT-37

- Finger Tack Type
  - #2 (stiff peaks), #4 (thready) pass LT-37



- Thread Length by TAA:
  - Low speed – moderate is best, triangle plot

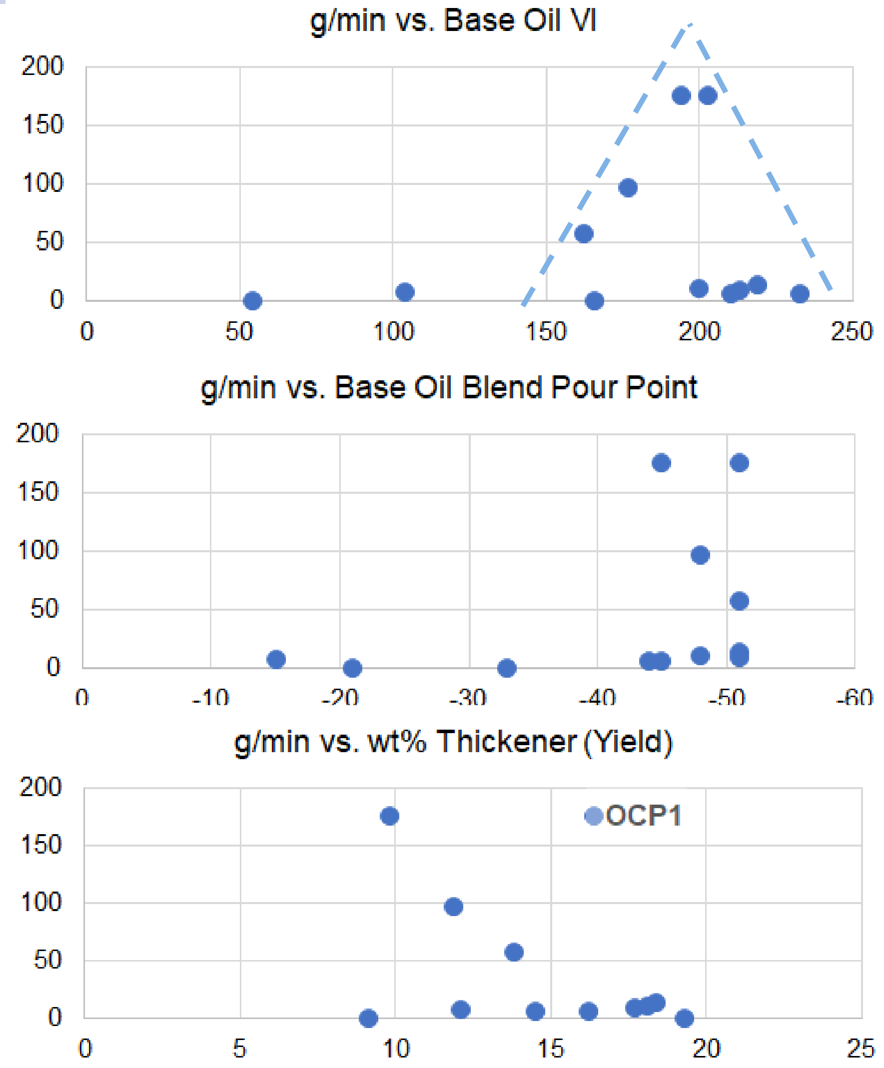


- Separation Energy by TAA:
  - High speed – lower is better; nice curve

"Low Speed" = 0.1mm/s; "High Speed" = 5.0mm/s

Grease Composition vs. LT-37

- Base Fluid
  - Viscosity Index: >150; >200 is too much
  - Pour Point: <-40°C needed
- Thickener
  - Usage: <15wt% Li-12HSA; less % is better



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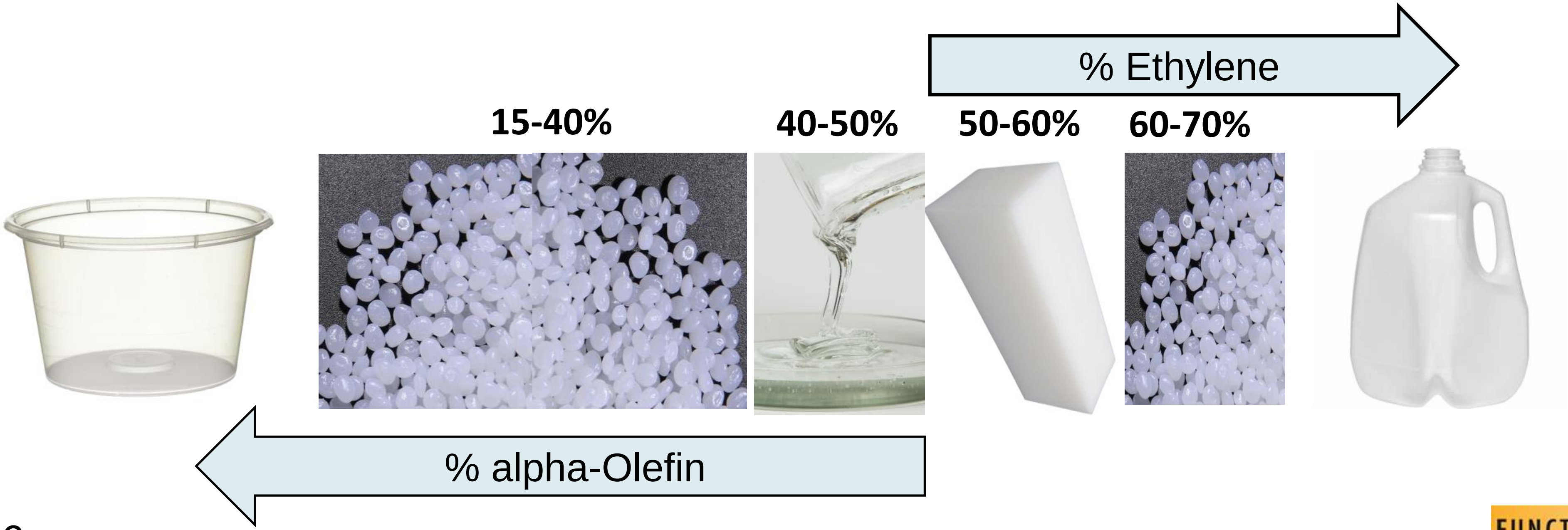
Polymer Type vs. Low Temp.

- Semi-crystalline (CRY) was unfavorable in all low temp testing
  - Use sparingly for tack, water resist, oil bleed (0.5-1wt%) as grease polymer
    - Using as VM here was too much (~3wt%), bad selection



Conclusions

- Four types of tack behavior observed
- ‘Tack types’ correlated with forces and energies measured in TAA
- Heavily polymer-modified PIB and OCP greases perform well at low temp.
- Tack correlates with low temp performance – best were moderately tacky

Thank you for your attention

Please ask me or see the future article
in Eurogrease for further details or clarification!

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