Load Carrying Behavior of Greases Using Highly Refined Oil with Viscosity Modifier

Dr. Erik Willett
Functional Products Inc.

NLGI India Chapter
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Agenda

- Research Goals
- Materials/Methods
- Experimental Plan
- Results
  - EP
  - AW
- Future Work

Functional Products Inc.

- Based in Ohio, USA since 1985
- NLGI India since 2008 (Aurangabad)

- Focused on customer driven solutions
- ISO 9001 with Design

- Expansion completed 2020

- Applying polymer science
  to the art of greasemaking

2020

2021

x2

Past R&D, Future Developments

- Tackifiers
- Viscosity Modifiers
- Grease Additives
- Industrial Additives
- Food Machinery HX-1
- Biobased / Biodegradable

- *Proudly distributed by platinum sponsor Environ*

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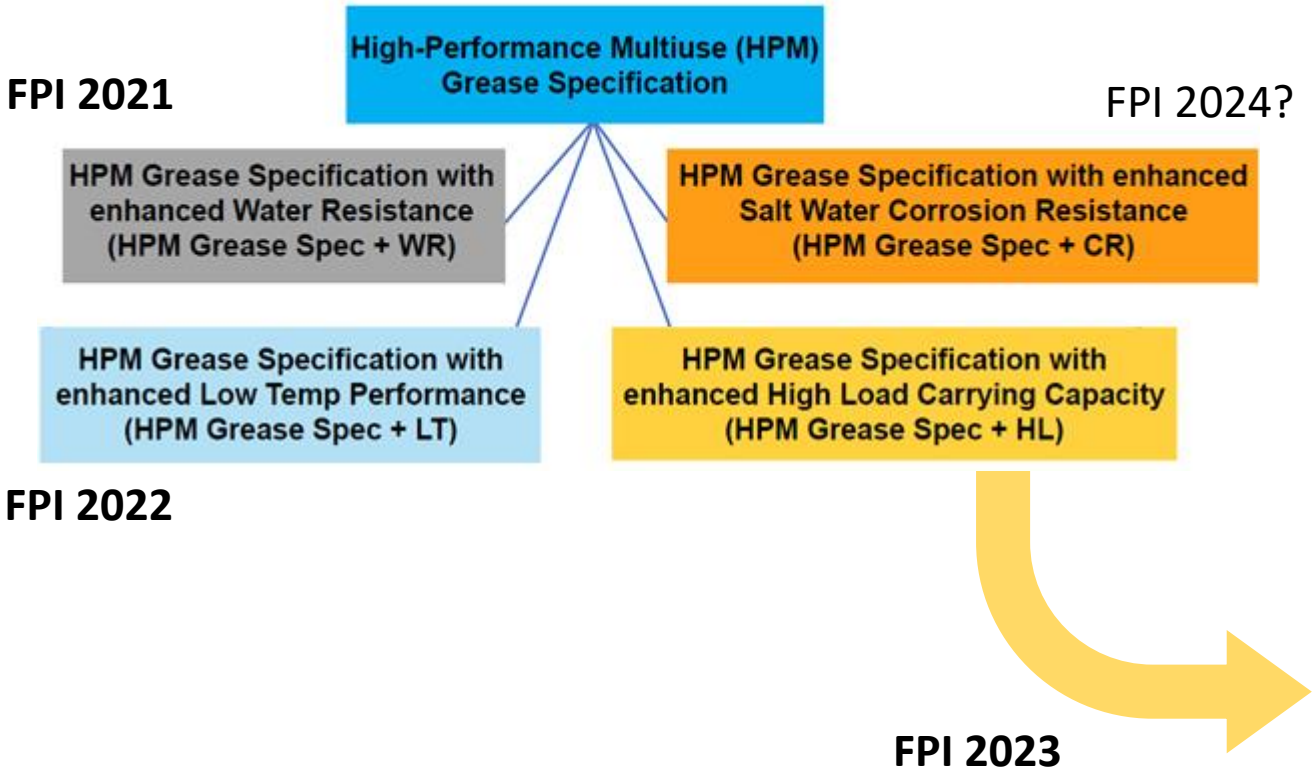
NLGI HPM and Polymers in Grease



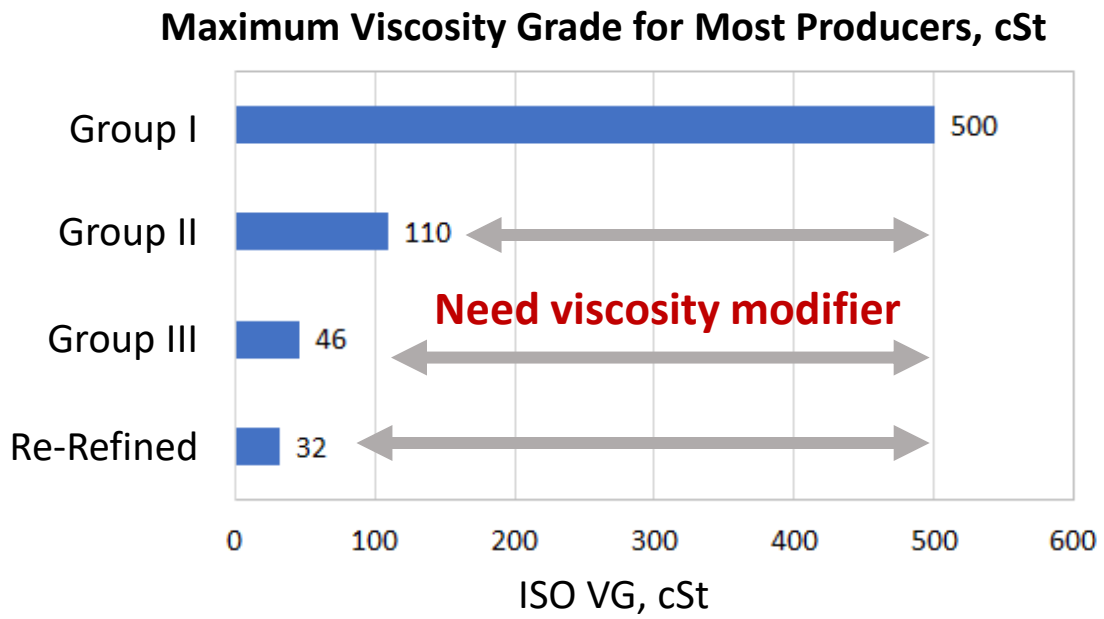
Where would it be challenging to test polymers vs. HPM-HL spec?

	Property	Test Conditions	Test method	Units	Min	Max
HPM +HL	Wear Preventive Characteristics of Lubricating Grease (Four-Ball Method) Wear Scar Diameter	75 °C, 1200 rpm, 60 minutes	ASTM D2266	mm		0.50
	Measurement of Extreme-Pressure Properties of Lubricating Grease(Four-Ball Method), Weld point	1770 rpm @ 27°C	ASTM D2596	kgf	400	
	Determining Extreme Pressure Properties of Lubricating Greases Using a High- Frequency, Linear-Oscillation (SRV)Test Machine	(Procedure B at 80°C)	ASTM D5706	N	800	
	Fretting Wear Protection by Lubricating Greases	Average of 2 runs,22 hours @ Room Temperature	ASTM D4170	mg		5.0
	Determining Fretting Wear Resistance of Lubricating Greases Under High Hertzian Contact Pressures Using a High- Frequency, Linear-Oscillation (SRV) Test Machine	50°C, 100N, 0.300mm, 4 hours	ASTM D7594	mm		0.500

Table 4 – HPM+HL specification

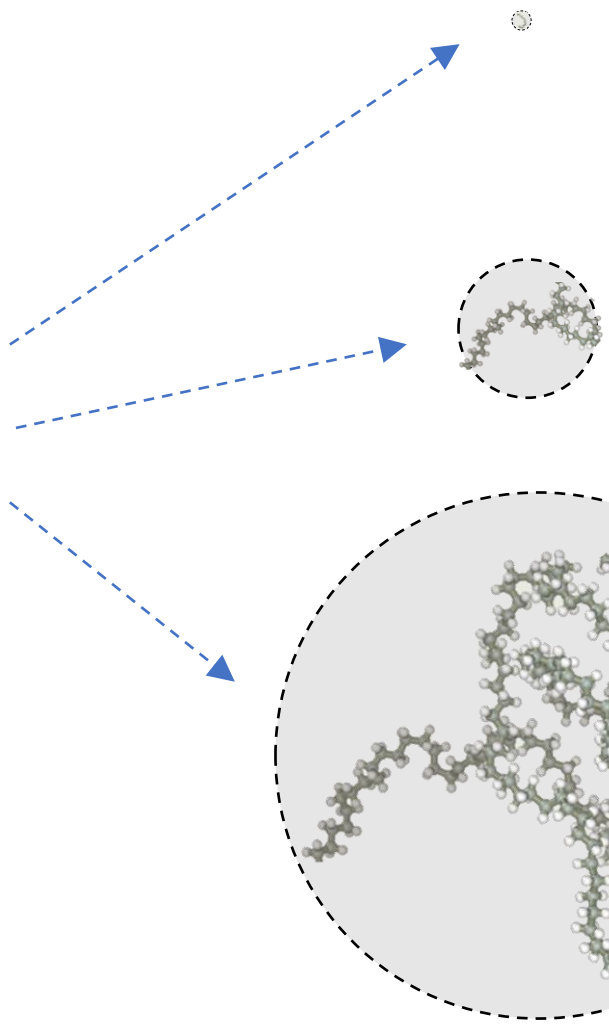
Research Goals

- What are the tribological limits of obtaining a high % of viscosity from VM?
- Is using viscosity modifiers still ‘high performance’?
- Can we allow the use of new low viscosity, highly refined oils in grease?



Viscosity Modifiers

Material	MW	# of Carbons
Petroleum Oil	200 - 700	10 - 50
Polymer Base Oils	1K – 10K	150 – 2K
Viscosity Modifiers	10K – 200K	2K – 30K



Starts at VI 90 – 120
Relatively low cost
Limited viscosity ranges

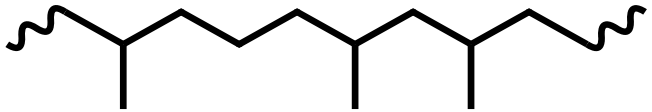
Builds VI 140-170
High Treat Rate
High $/cSt in Cost
Not Shear Sensitive

Builds VI 180+
Low Treat Rate
Lowest $/cSt
Shear Sensitive

Scaled by molecular radius, approx. (# carbon)$^{0.5}$

Materials – Viscosity Modifiers

- Six ethylene-propylene viscosity modifiers
  - Mw 10,000 to 150,000
  - Amorphous (40-60% C2)



- Used to build **ISO 220 base fluids** from ISO 25 re-refined Group II+

	OCP-0	OCP-1	OCP-2	OCP-22	OCP-35	OCP-45
Pure Form	Liquid	Liquid	Liquid	Bale	Bale	Bale
Shear Stability Index (SSI, D6278)	0	0.5	2	25	35	45
Typical Usages	Gear	Gear/HF	HF	Engine	Engine	Engine
wt% in 120N for ISO 220	~30%	~22%	~11%	~3%	~2.5%	~2%
Viscosity Index	172	172	184	194	194	190
FUNCTIONAL product name:	**V-736**	**V-732**	**V-739**	**V-100**	**V-135**	**V-101**

Methods – Grease Production

1	Pre-Formed lithium 12-HSA	8 – 9 wt%
1	Antioxidant blend	0.7 - 1 wt%
1	ISO 25 Group II+ re-refined oil	47 – 85 wt%
2	Viscosity modifier polymer	0 – 30 wt%
3	Ashless gear oil package	3 – 6 wt%
3	Liquid molybdenum complex	2 – 4 wt%

"Gr. I Control" grease:
ISO 220 Group I blend, no VM

FUNCTIONAL GA-614
Ashless gear oil package

Sequence of Events:

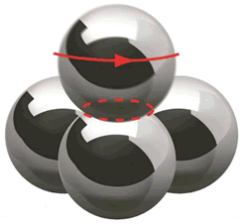
1. Charge Li-12HSA, AO, and oil to open kettle
   - Ramp to 180-190°C until pre-formed Li-12HSA is molten
   - Cool and mill
2. Adjust with viscosity modifier and/or oil to NLGI #2; mill.
3. Proceed to EP/AW testing. Adjust with gear package and/or Mo complex.
   - Replace % oil with % additives in the final formula. Adjust to #2 again if needed.

Methods – HPM-HL Test Plan

Prepare Base Greases (#2 Li, ISO 220) with Experimental Base Oil Blends

+/- 0.5wt% Gear Package

4-Ball EP

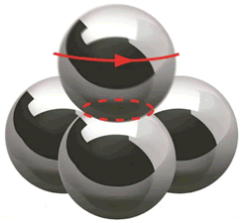


D2596

400 kgf Weld Load

+/- 0.5wt% Liquid Mo Complex

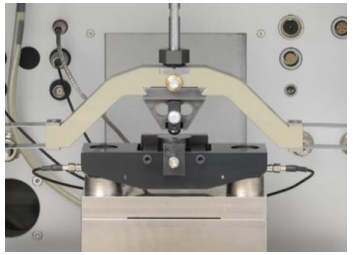
4-Ball Wear



D2266 @ 40kg

<0.50 mm Wear

Re-check EP Weld



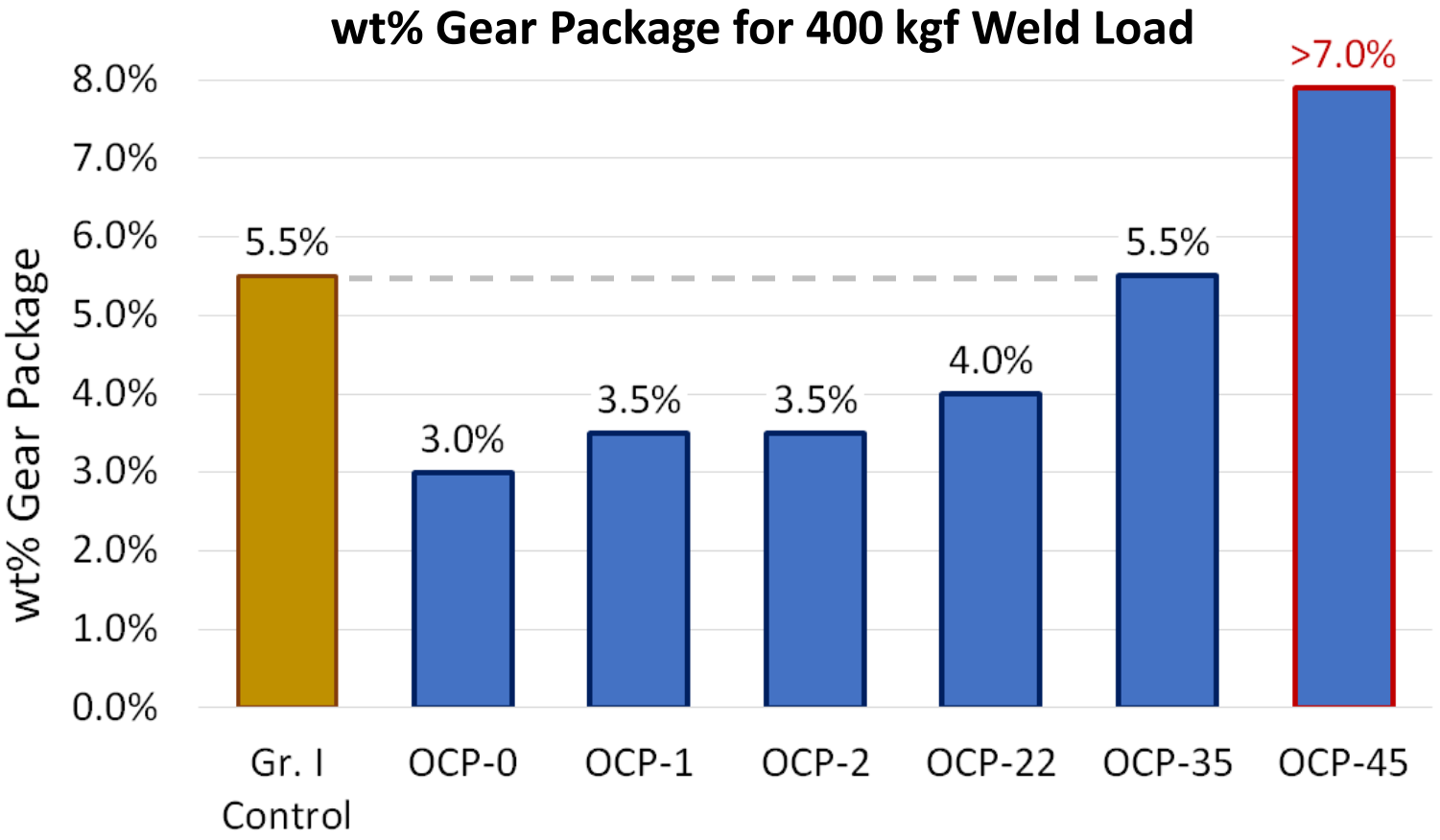
SRV

Fretting

NLGI US Meeting

EP Findings

- Use of VM reduced wt% gear package by 35-45% (5.5wt% vs. 3-3.5%)
- Lowest treat = best inherent EP performance of base fluid?

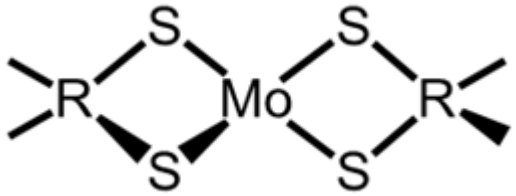


Package Details:

- Ashless S/P/N
- Active sulfur chemistry
- Suitable for GL-5 gear at 3-4%
- Designed in ISO 220 Gr.I
- FZG 12+

D2266 AW Findings

- ~0.6-0.7 mm → reduced to ~0.45 mm w/ Mo complex

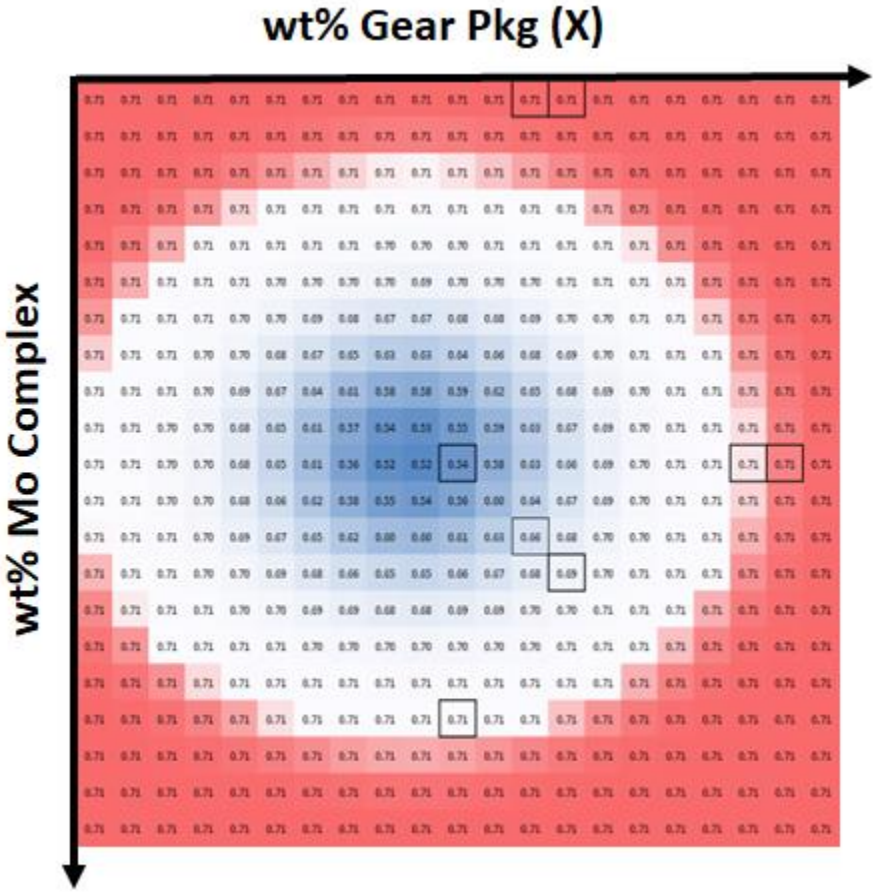


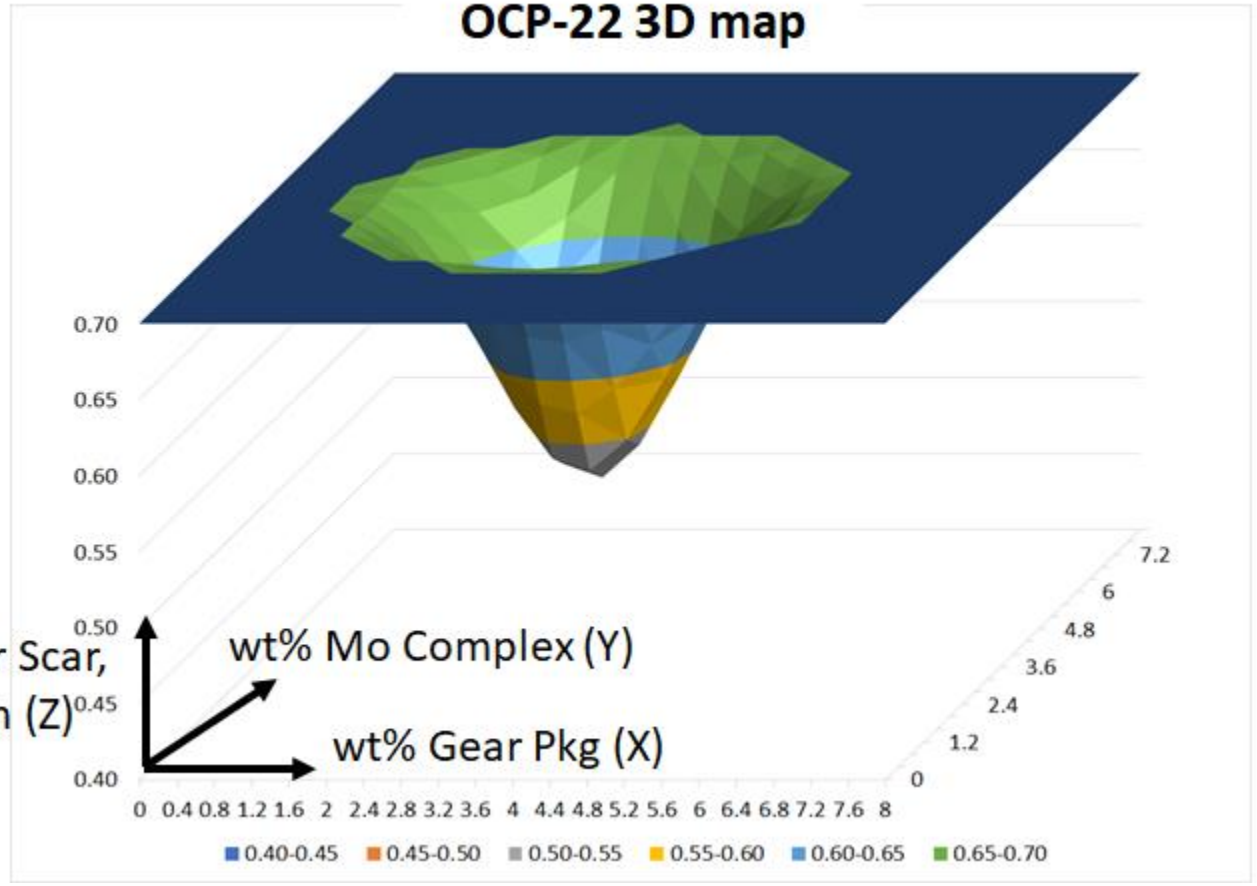
- Borates or graphite also help

	Gr. I Control	OCP-0	OCP-1	OCP-2	OCP-22	OCP-35	OCP-45
SSI of Viscosity Modifier (D6278)	n/a	0	0.5	2	22	35	45
Optimal wt% Ashless Gear Pkg	5.5%	3.0%	2.5%	4.0%	3.5%	3.5%	n/a
Optimal wt% Liquid Mo Complex	3.0%	3.0%	2.5%	4.0%	4.0%	4.0%	n/a
Wear Scar @ 40kg, mm (D2266)	0.46	0.47	0.46	0.44	0.54	0.55	n/a
EP Weld Load (D2596)	400	400	400	400	400	400	n/a
FUNCTIONAL product name:	**n/a**	**V-736**	**V-732**	**V-739**	**V-100**	**V-135**	**V-101**

AW Optimization 'DOE'

- wt% Gear Pkg and wt% Mo Complex vs. wear modeled as inverted 2D Gaussian
  - Model used to guess best wt% then results used to retrain the model





Summary of 4-Ball Testing for HPM-HL

- For #2 simple Li grease with ISO 220 oil, where 90% of visc from VM:

	Gr. I						
	Control	**OCP-0**	**OCP-1**	**OCP-2**	**OCP-22**	**OCP-35**	**OCP-45**
SSI of Viscosity Modifier (D6278)	n/a	0	0.5	2	22	35	45
Can meet 400 kgf weld (D2596)?	Y	Y	Y	Y	Y	Y	N
Can meet < 0.5 mm wear (D2266)?	Y	Y	Y	Y	N	N	N
Can meet SRV EP (D5706)?	tbd	tbd	tbd	tbd	n/a	n/a	n/a
Can meet fretting (D4170)?	tbd	tbd	tbd	tbd	n/a	n/a	n/a
Optimal wt% Ashless Gear Pkg	5.5%	3.0%	2.5%	4.0%	3.5%	3.5%	n/a
Optimal wt% Liquid Mo Complex	3.0%	3.0%	2.5%	4.0%	4.0%	n/a	n/a
FUNCTIONAL product name:	**n/a**	**V-736**	**V-732**	**V-739**	**V-100**	**V-135**	**V-101**

Future Work

- Remaining NLGI HPM-HL requirements:

- **SRV and fretting wear (D5706, D7594; D4170)**
  - Samples ready
  - *Will be discussed at NLGI US*

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Table 4 – HPM+HL specification

Summary

- New HPM-HL spec, re-refined oils, Group III – good time to study fundamentals where viscosity modifiers most important

- Shear stability recommendations
  - 22 and 35 SSI VM for GC-LB wear (0.5-0.6mm) + 400 kgf weld
  - 2 SSI VM for NLGI HPM-HL (<0.5 mm) + 400 kgf weld

- Gear package and liquid Mo complex must be carefully optimized for meeting target wear performance

Q & A

Thank you for your attention

Any questions?

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www.functionalproducts.com for product info