Synthesis and Characteristics of the Optimum Urea Base Grease for Rolling Bearing

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(FAG Bearing Korea)

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Synthesis and Characteristics of the Optimum Urea Base Grease for Rolling Bearing

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Introduction

- High performance characteristics such as long life, low torque, small temperature rise, low noise, low wear and high corrosion resistance are required for rolling bearings.
- Rolling bearings are classified one of the most important machine elements.
-The various function of bearings are greatly influenced by grease, and higher performance of rolling bearings is required to improve bearing lubrication.

Objectives

- Characterization of the synthesized three urea/ether oil greases with different content of the thickener.
- Investigation of the important factor dominating the performance of the grease
- Investigation of typical grease properties and FE-9 grease life test.
- Selection of optimized greases by SSRED Program (Six Sigma Robust Engineering Design)
- The evaluation of optimized greases by means of bearing endurance tester.

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Experimental

- Test of physical properties of grease
- Test of FE-9(DIN 51821) grease life
- Micrographs & SEM
- FT- IR, TG/DSC & TAN(㎎KOH/g)
- Test of oxidation induction time
- Synthesis of the optimum greases
- Evaluation of bearing endurance test

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Composition of Grease

Thickener	+	Base oil	+	Additives
10 ~ 25 %		70 ~ 95%		2 ~ 5%
- Heat resistance - Water washout - Mechanical stability		- Lubrication - Heat resistance - Low temperature performance - Oxidation stability		- EP - Oxidation stability - Rust preventive

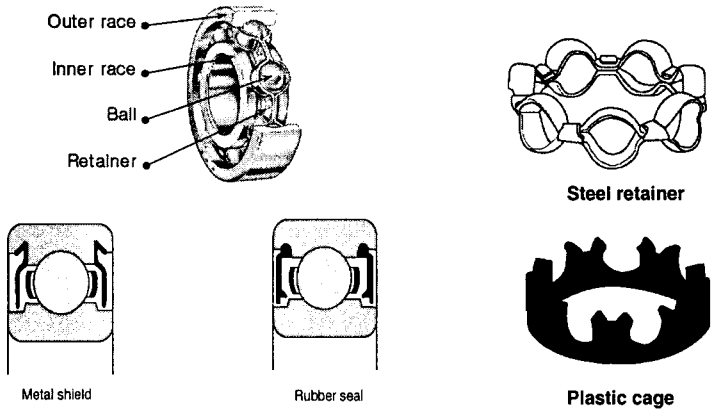

Fig. Structure of rolling bearing set.

Table Composition of Synthesized Greases

Grease	Thickener	Base oil	Content of thickener(wt%)
A	Urea	Ether oil	15
B	Urea	Ether oil	17
C	Urea	Ether oil	20

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Table Properties of Greases

Test method \ Grease			A	B	C
Thickener		-	Urea	Urea	Urea
Base oil		-	Ether	Ether	Ether
Worked penetration		KS M 2032	295	287	285
Dropping point ℃		KS M 2033	245	255	260
Oil separation (100℃×24hr, wt%)		KS M 2050	1.5	1.3	1.2
Oxidation stability (99℃×100hr, kg/㎠)		KS M 2049	0.17	0.15	0.1
Bearing rust preventive (52℃, 48hr)		ASTM D1743	# 1	# 1	# 1
Viscosity of base oil (cSt)	40℃	KS M 2014	96.5	97	96.1
	100℃		11	10	10

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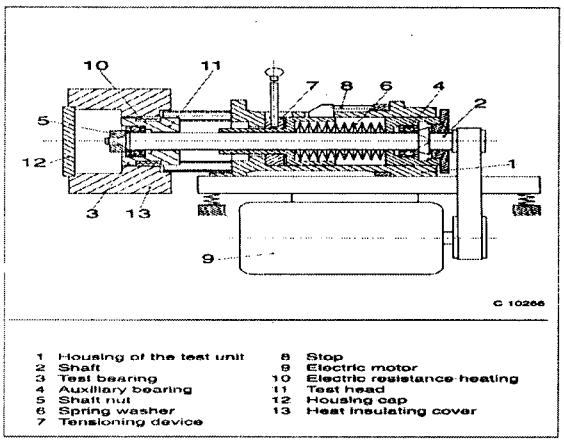

Fig. Schematic diagram of FE-9grease life tester.

Table Conditions of FE-9Grease Life Test

Test bearing	FAG 529689 B/R (Angular contact ball B/R)
Amount of grease	1±0.1g (35% volume of free space)
Temperature	160℃
Rotation speed	6,000 rpm
Applied load	150 kg_f

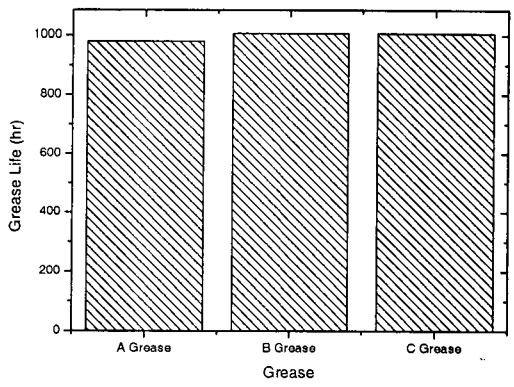

Fig. Results of FE-9grease life test.

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C Grease

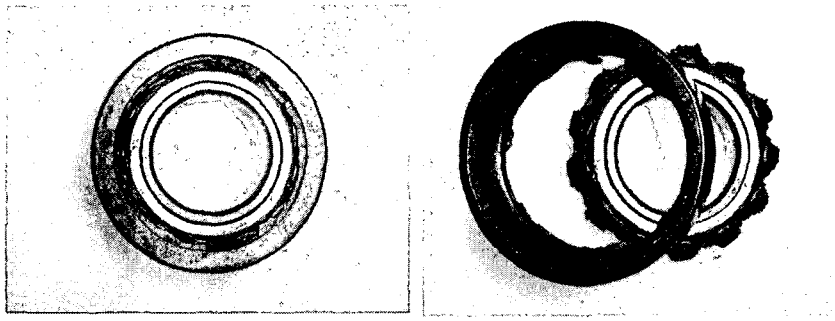

Fig. The grease B/R after FE-9 grease life test.

Micrographs

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C Grease

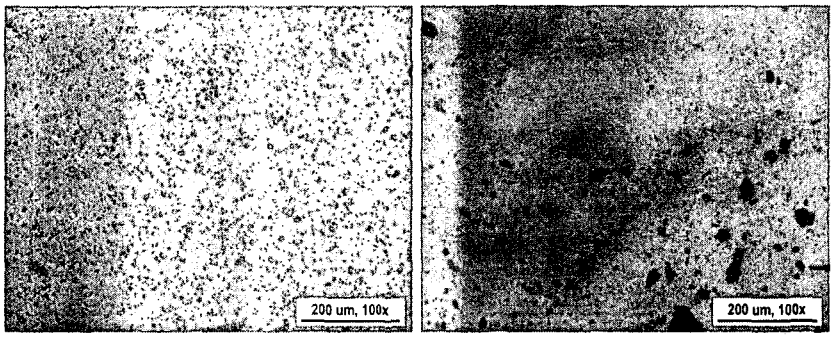

Fig. Micrographs of grease C before(left) and after(right) life test.

SEM

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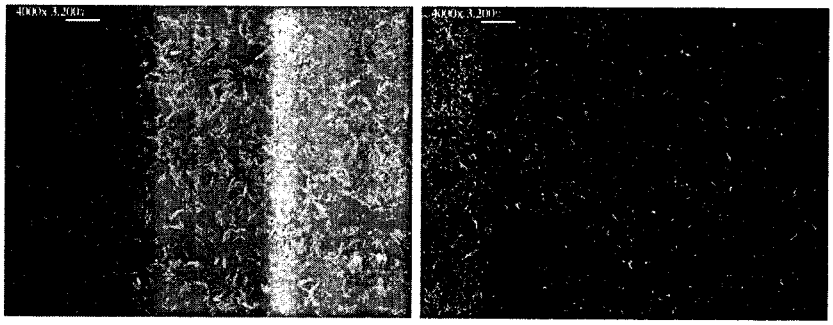

Fig. Electro micrographs of thickener in grease C before life test.

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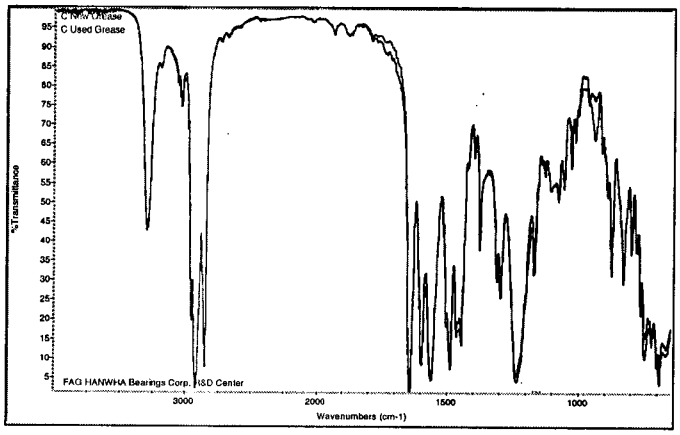

Fig. IR transmittance spectra of grease C before and after life test.

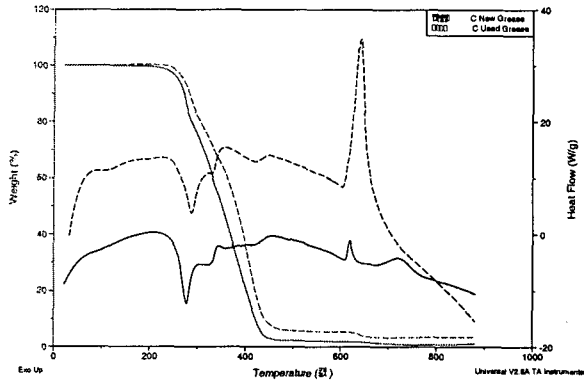

Fig. TG/DSC analysis curves of grease C before (solid line) and after(dotted line) life test.

Table OIT value of greases before and after life test

Grease	New Grease	Used Grease
A Grease	38	20
B Grease	37	27
C Grease	40	34

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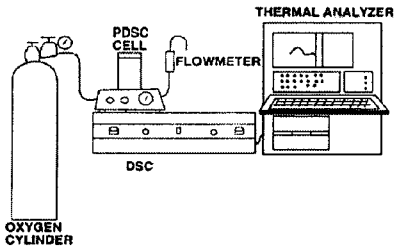
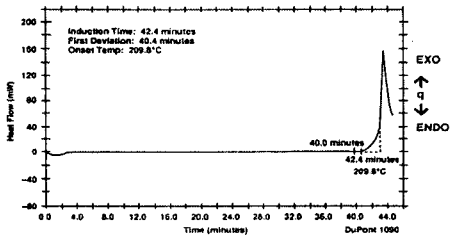
➢ ASTM D 5483 PDSC Method





➢ OIT can be used as an indication of oxidation stability
➢ Used for R&D, QC and specification purpose
➢ But no correlation has been determined between OIT result and service performance

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Table TAN(mgKOH/g) data of greases before and after life test

Grease	A	B	C
Before	0.29	0.47	0.30
After	3.79	2.78	2.15

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Table Orthogonal array L12 [2(11)]

Factorial	Level 1	Level 2
Thickener type	Amine - 1	Amine - 2
Thickener content(wt%)	20	17
AO Type	Amine	Phenol
AC Type	Metal - 1	Metal - 2
AW Type	AW - 1	AW - 2
AO content (wt%)	0.5	1.0
AC content (wt%)	1.0	1.5
AW content (wt%)	1.0	1.5
Reaction condition (℃)	160	180
Reaction condition (rpm)	30	15
Roll milling(a time)	2	4

※ AO : Anti oxidation , AC : Anti corrosion , AW : Anti wear

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Table Kind of grease additives

Additive	Level 1	Level 2
Anti Oxidation additive	Amine (Benzenamine) (C_6H_7N)	Phenol (B H T) $C_6H_2(OH)(CH_3)[C(CH_3)_3]_2$
Anti Corrosion additive	Metal (Barium Sulfonate)	Non metal (2-heptadecenyl-4, (5H)-Oxazoledimethanol)
Anti wear additive	Metal (Zn-DTP)	Non metal(Amine Phosphate)

Table Result of anderon test(6203UU)

One-way ANOVA: LB, MB, HB

Analysis of Variance

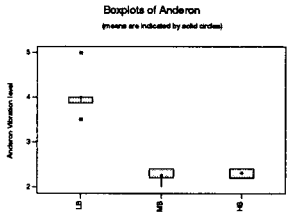
Source	DF	SS	MS	F	P
Factor	2	19.5120	9.7560	149.67	0.000
Error	27	1.7600	0.0652		
Total	29	21.2720			

Individual 95% CIs For Mean Based on Pooled StDev

Level	N	Mean	StDev
LB	10	4.0000	0.4082
MB	10	2.2600	0.1350
HB	10	2.3200	0.1033

Pooled StDev = 0.2553

2.40 3.00 3.60 4.20



One-way ANOVA: LB, MB, HB

Analysis of Variance

Source	DF	SS	MS	F	P
Factor	2	19.0587	9.5293	173.85	0.000
Error	27	1.4800	0.0548		
Total	29	20.5387			

Individual 95% CIs For Mean Based on Pooled StDev

Level	N	Mean	StDev
LB	10	3.9200	0.3458
MB	10	2.2000	0.1886
HB	10	2.2600	0.0966

Pooled StDev = 0.2341

2.40 3.00 3.60 4.20

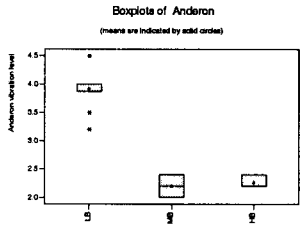


Table Result of grease dropping point test

RED – Raw Data – Synthesis of Optimum Grease(Dropping point)

Date: 2004-02-11 Time: 1:35:21 오후
Input File Name: gredpdata Output File Name: red1.out
Data Type: Continuous Data Characteristics: Larger-the-Better
Inner Array Interactions: 0 Outer Array Interactions: 0

Factors and Levels

Controllable(Inner) Factors: (*): Current or Standard Level

Symbol	Name	Level 1	Level 2
A	Thick	Amine-1(*)	Amine-2
B	Thick co	20(*)	17
C	ACtype	Amine(*)	Phenol
D	ACtype	Metal-1(*)	Metap-2
E	AWtype	AW-1(*)	AW-2
F	ACcon	0.5(*)	1.0
G	ACcon	1.0(*)	1.5
H	AWcon	1.0(*)	1.5
I	Re con℃	160(*)	180
J	Re rpm	30(*)	15
K	Roll	2(*)	4

ANOVA Table (Raw Data)

Factor Sym	(DoF)	S (Variation)	V (Variance)	Fo (F-Value)	Test	Contribution
A	1	38.028	38.028	5.7	**	5.3 %
B	1	132.25	132.25	19.9	***	21.3 %
C	1	17.36	17.36	–	pool	
D	1	0.02778	0.02778	–	pool	
E	1	17.36	17.36	–	pool	
F	1	2.25	2.25	–	pool	
G	1	124.69	124.69	18.8	***	20.0 %
H	1	0.6944	0.6944	–	pool	
I	1	38.028	38.028	5.7	**	5.3 %
J	1	34.028	34.028	5.1	**	4.6 %
K	1	30.25	30.25	4.6	**	4.0 %
e(p)	5	37.6944	7.53889	–	–	7.7 %
e(2)	24	154.67	6.4444	–	–	31.7 %
(e)	(29)	(192.36)	(6.63311)	–	–	(39.4 %)
Total	35	589.639	–	–	–	100.0 %

Estimate of Optimum Conditions (Raw Data)

Factor	Level
(A) Thick	(1) Amine-1
(B) Thick co	(1) 20
(G) ACcon	(2) 1.5
(I) Re con℃	(1) 160
(J) Re rpm	(2) 15
(K) Roll	(2) 4

Mean of data when all factors set at current or standard level
260.333

Mean of data of all experiments
260.194

Predicted Optimized Mean at Estimated Optimum Conditions

267.917 (+/- 4.02) (2.91% Improved mean value over standard run)

Table Result of grease OIT test

RED – Raw Data – Synthesis of Optimum Gtrase(OIT Value)

Date: 2004-02-11 Time: 1:35:21 오후
Input File Name: new.in Output File Name: greaseop.out
Data Type: Continuous Data Characteristics: Larger-the-Better
Inner Array Interactions: 0 Outer Array Interactions: 0

Factors and Levels

Controllable(Inner) Factors: (*): Current or Standard Level

Symbol	Name	Level 1	Level 2
A	Thick	Amine-1(*)	Amine-2
B	Thick co	20(*)	17
C	AOtype	Amine(*)	Phenol
D	ACtype	Metal-1(*)	Metap-2
E	AWtype	AW-1(*)	AW-2
F	AOcon	0.5(*)	1.0
G	ACcon	1.0(*)	1.5
H	AWcon	1.0(*)	1.5
I	Re con℃	160(*)	180
J	Re rpm	30(*)	15
K	Roll	2(*)	4

SSRED

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ANOVA Table (Raw Data)

Factor Sym	(DoF)	S (Variation)	V (Variance)	Fo (F-Value)	Test	Contribution
A	1	9.1875	9.1875	89.2	***	20.6 %
B	1	9.9008	9.9008	96.1	***	22.2 %
C	1	0.00083340.0008334		–	pool	
D	1	4.4408	4.4408	43.1	***	9.8 %
E	1	0.80083	0.80083	7.8	*	1.6 %
F	1	1.6875	1.6875	16.4	**	3.6 %
G	1	7.5208	7.5208	73.0	***	16.8 %
H	1	0.3008	0.3008	–	pool	
I	1	1.8408	1.8408	17.9	**	3.9 %
J	1	8.5008	8.5008	82.5	***	19.0 %
K	1	0.0075	0.0075	–	pool	
e(p)	3	0.30916	0.103053	–	–	2.6 %
(e)	(3)	(0.30916)	(0.103053)	–	–	(2.6 %)
Total	11	44.1892	–	–	–	100.0 %

Estimate of Optimum Conditions (Raw Data)

Factor	Level
(A) Thick	(2) Amine-2
(B) Thick co	(1) 20
(D) ACtype	(1) Metal-1
(E) AWtype	(1) AW-1
(F) ACcon	(2) 1.0
(G) ACcon	(2) 1.5
(I) Re con℃	(1) 160
(J) Re rpm	(2) 15

Mean of data when all factors set at current or standard level
25.400

Mean of data of all experiments
26.242

Predicted Optimized Mean at Estimated Optimum Conditions

31.292 (+/- 0.885) (23.2% Improved mean value over standard run)

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Verification test

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Verification test of grease dropping point

Optimum Condition

Factorial	Level
(A) Thickener content	(1) 20 %
(E) A W Type	(1) AW-1
(G) A C Content	(2) 1.5 %
(I) Reaction condition (℃)	(1) 160 ℃
(K) Roll milling (a time)	(2) 4

Verification test

Estimated Value at Optimized condition	Test 1	Test 2	Test 3
267	265	267	264

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Verification test

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Verification test of grease OIT(Oxidation Induction time)

Optimum Condition

Factorial	Level
(A) Thickener type	(2) Amine-2
(B) Thickener content	(1) 20%
(D) AC Type	(1) Metal - 1
(E) AW Type	(1) AW-1
(F) AO Content	(2) 1.0%
(G) AC Content	(2) 1.5%
(I) Reaction condition(℃)	(1) 160 ℃
(J) Reaction condition(rpm)	(2) 15 rpm

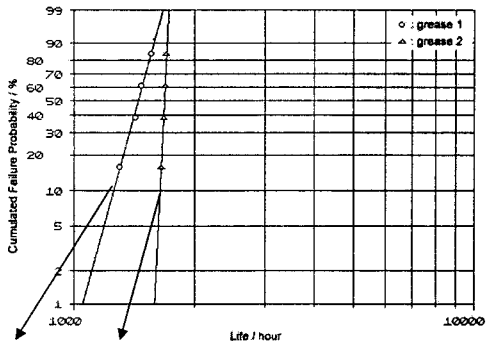
Verification test

(Unit : min)

Estimated Value at Optimized Condition	Test 1	Test 2	Test 3
31.29	32.34	33.57	33.97

Table Conditions of bearing endurance test

Test bearing	# 6203 UU
Amount of grease	1±0.1g (35% volume of free space)
Temperature	150℃
Rotation speed	10,000 rpm
Applied load	100 kg_f



1,255hr 1,638hr

Grease 1 : Optimum Grease (dropping point)
Grease 2 : Optimum Grease (OIT)

Fig. Results of bearing life test.

Conclusion

- Three urea/ether oil greases with different content of the thickener were synthesized and the performance of these greases were compared. The typical grease properties were investigated. And life test of these greases was conducted by FE-9 grease life tester(DIN 51821). The characteristics of the greases before and after life test were investigated by FT-IR, Micrograph, SEM, DSC-TGA, OIT(oxidation induction time) and TAN meter.
- Large differences in the grease performance depending on the content of the thickener were observed. The grease with higher content of the thickener showed higher performance such as long life time and low TAN value.
- Twelve greases were synthesized and evaluated the performance of dropping point and OIT.

- Optimized two greases were selected by SSRED (Six Sigma Robust Engineering Design)
- Characteristics of the optimized two greases by SSRED showed the same level with estimated values.
- The optimized grease by means of OIT value showed longer grease life in comparison with optimized grease by dropping point.
  Specially, the life time of the optimum grease using OITvalue was 1.5 times lomger than that of the optimum grease using dropping point
-Optimized two greases showed higher performance than typical urea/ether oil.

- The end -