自動車用潤滑油の還元添加剤によるトライボロジー性能に及ぼす影響

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Effects on Tribology Performance of a Reducing Additive for Automobile Lubricant

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KEY WORDS: **Heat engine**, **Power Transmission**, Lubricating Oil/Engine Oil, Lubricating Oil/Transmission Oil, Additive Lubrication/Tribology (A1)

We developed a unique reducing additive composed primarily of polyolester, diester, and vegetable oil-based ester compounds in order to switch from boundary to fluid lubrication and improve tribology performance, and chemically and experimentally investigated its effects on tribology performance. To confirm the lubrication effects of this reducing additive, we added it to oil and chemically investigated the cleaning and dissolving of the ultra-fine particle layer of contaminants, etc., adhering to lubrication pathways and sliding surfaces, and performed commercialization testing using actual vehicles. We found that this additive washed away and dissolved sludge, varnish, and other contaminants from lubricated surfaces, improving their lubrication properties and their tribology performance by reducing abrasion and friction.

1．ま え が き

近年の自動車の高性能化に加えて，省資源と環境負荷低減のため潤滑油のトライボロジー性能向上に対する要求が一段と増してきている．これらの要求に応える手段として潤滑性能の向上, 摩擦, 摩耗の低減のためエステル系合成潤滑油が開発されてきたが，まだ，十分とは言い難い．このエステル系合成潤滑油は有機 (脂肪) 酸とアルコールを原料としているが, 特にポリオールエステル系合成潤滑油[(1),(2)]は元来，ジェットエンジンの潤滑油として広く使用されており，最近，自動車用として注目されている．

この合成潤滑油は鉱物系潤滑油と比べて，低温流動性, 熱, 酸化安定性に優れ，高粘度で使用温度範囲が広く，潤滑性が良好で清浄，分散性や生分解性を有するなど多くの特長を有する．しかし，欠点として加水分解しやすく，吸湿性があり，ゴム，シール材，樹脂や塗料で制限され，またコストが高い点を有するが，合成潤滑油の中でトライボロジー性能，省資源と省エネなどに対して最も機能的に優れている[(1),(2)]と言われている．

合成潤滑油は各種潤滑油添加剤[(3),(4)]を混合して製造されているが，潤滑油添加剤の主な種類として清浄分散剤，酸化防止剤，極圧剤，防錆剤，粘度指数向上剤，流動点降下剤，消泡剤や摩擦低減剤などがあり，これらの添加剤の化合物は自動車の発展とその環境とともに変革してきている．このような状況の下で最新の自動車技術に対応できる潤滑油はエステル系合成潤滑油としてポリオールエステル系やジエステル系添加剤を添加剤とした合成潤滑油が不可欠と考えられる．

本研究はエンジン，トランスミッション，デファレンシャルギアなどの自動車用潤滑油の潤滑, 摩耗, 摩擦性能などをより改善するため，２次的合成添加剤としてポリオールエステル（POE），ジエステル（DST）や植物油系エステル（VOE）化合物などを主成分とした独自の還元添加剤 (SOD-1)を開発し，トライボロジー性能に及ぼす影響を化学的作用と実用試験等により確認したので報告する．

2．最近のエンジンにおける潤滑油添加剤の傾向

最近のエンジンにおける潤滑油添加剤の傾向[(3)]を見てみると，ディーゼルエンジンは不完全燃焼による煤（C）が，燃料の燃焼によって発生する NO や SO_3 と反応してできる生成物がデポジットの主成分で，ラッカー，ワニスは圧縮行程の末期近くで発生する低分子の燃料酸化物と考えられている．燃焼室内のデポジット低減に燃料添加剤と潤滑添加剤を供用した報告[(5)]がある．また，排ガスの後処理として煤や灰の捕捉を目的とした DPF の灰分蓄積を少なくするために，カルシウム硫酸塩をベースにした潤滑油添加剤[(6)]があり，クランクケース内の潤滑油中の煤の存在がバルブ系とライナー摩耗に影響を与えるので，適切な潤滑添加剤を用いることにより両者の摩耗を制御して実用化できた報告[(7)]がある．

ガソリンエンジンのデポジット生成は，高温領域での基油の酸化劣化が主原因と言われ，低温領域では燃料とその酸化物が主原因となり，これらの物質はブローバイガスの成分としてクランクケース内で樹脂状やカーボン状物質を形成して，エンジン内部の潤滑油経路や各部の堆積物となる．また，ガソ

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リンエンジンの低摩擦持続性を延長可能な新規の潤滑油添加剤[8]や硫黄を含まないジアルキル燐酸亜鉛（ZnDTP）や燐酸エステルなどの耐摩耗性向上添加剤[9]も開発されている．したがって，両エンジンにとって共通のデポジット対策は，潤滑油添加剤の清浄分散，分解性と酸化防止が最も重要な作用と言える．

３．還元添加剤

3.1　還元添加剤のコンセプト

自動車エンジン，変速機，デファレンシャルギアなどの潤滑摺動部表面に固着したデポジット，ワニスやスラッジなどのコンタミ類は，時間の経過とともに積層して，トライボジー性能（潤滑，摩耗，摩擦）に悪影響を与えている．これらを改善するために通常，オイル交換時に灯油系清浄剤でフラッシングを実施する方法があるが，図 1 に示すように上から３層の一般の汚れの膜，吸着分子層と酸化膜からなるコンタミの完全除去は困難で，除去されたコンタミ類が潤滑油経路に詰まったりして清浄効果は少なくなり，潤滑不良を発生するなど逆にリスクを伴う．

したがって，本還元添加剤は POE，DST や VOE 系化合物等を主成分に各種添加剤をブレンドした還元添加剤をエンジン，手動変速機，パワーステアリングとデファレンシャルギア・オイルに 10 vol.%，自動変速機オイルは 7 vol.%混合して，潤滑作用をしながらコンタミ類を清浄，除去し，最初の金属加工面に戻す還元作用により，摩擦損失を大幅に低減し十分なオイルクリアランスを保持することで潤滑性を改善して摩耗，摩擦を減少できる独自の作用である．

よって，本還元添加剤のコンセプトは潤滑摺動部のコンタミ類を清浄，分解，除去して，最初の潤滑面（金属加工面）を再生，還元することにより，潤滑，摩耗，摩擦といったトライボロジー性能の改善を示唆している．本還元添加剤の最大の欠点は加水分解しやすさと吸湿性であり，またゴム，シール材を膨潤，硬化する特性があるが，この対策として石油系油脂のグリース成分等を添加することによって対応しているが，逆に良好な結果を得ている．

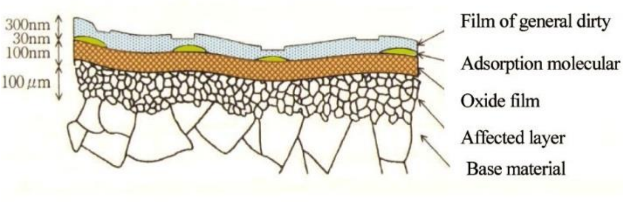


Fig.1 Components of Solid Surface

Table 1 Oxidation Stability Test of Reducing Additives (SOD-1)

Kinematic Viscosity　40℃　mm^2/s	610.0
100℃　mm^2/s	92.4
Viscosity　Index	243
Pour Point　℃	-42.5
Flash Point　(PM)　℃	170.0
Ash　mass %	2.305
Oxidation Stability　(ISOT)	
Viscosity Ratio	1.06
Increased Oxidative　mgKOH/g	-0.80
Lacquer Rating	No Deposits

3.2　エンジンオイル化学的性状における還元添加剤の影響

還元添加剤の化学的性状とエンジンオイルに及ぼす影響を表 1 に示す．動粘度は 40℃と 100℃で各々610，92.4 mm^2/s，粘度指数は 243，引火点は 170℃と高く，流動点は-42.5℃と低い値を示しており，高粘度，温度による粘度変化が小さく耐燃性，低温流動性に優れる POE，DST 系化合物の特色を表している．酸化安定度試験（ISOT）は粘度比 1.06 で酸化前後の粘度変化が少なく，酸化の増加の -0.80 mgKOH/g は加熱後の酸化が少なく，ラッカー度は付着物なしで酸化物やスラッジが混入していないことを示している．

3.3　還元添加剤をエンジンオイルに添加時の化学的性状

還元添加剤をエンジンオイル（SN5W-30）新油に 10 vol.%添加時の化学的性状の結果を表 2 に示す．動粘度は 40℃，100℃において還元添加剤無しのエンジンオイルの 60.9，10.5 mm^2/s に対して，還元添加剤の添加後は 77.6 と 13.5 mm^2/s で各々27.6 と 28.6%の増加率であった．粘度指数は添加前の 162 に対して添加剤の添加後は 179 で 10.5%の増加，ファレックス焼付荷重は同様に 750 に対して 1000 lbs で 33.3%の増加，オイルの耐熱性と洗浄分散性の程度を 10 段階で判断するホットチューブテストの評点は還元添加剤無しが 7 で添加後は 8 で 14.3%の上昇であった．ゴム膨潤度は同様に各々5.6 と 8.2%で 46.4%の増加であった．

以上のように還元添加剤の効果は大きく，特に動粘度とフ

Table 2 Effects of Reducing Additive Chemistry on Engine Oil (SN5W-30)

	SOD-1	SN5W-30	SN5W-30+SOD-1(10 vol.%)	Progress Rate %
Kinematic Viscosity　40℃ mm^2/s	610.0	60.9	77.6	27.4
100℃　mm^2/s	92.4	10.5	13.5	28.6
Viscosity　Index	243	162	179	10.5
FALEX Seizure Load　lbs	―	750	1000	33.3
Hot Tube Test　289℃　Mark	―	7	8	14.3
Rubber Swelling Degree　vol.%	―	5.6	8.2	46.4

Table 3 Effects of “SOD-1” on ATF Oil Chemistry and Shear Stability (Ultrasonic Method, JPI-5S-29-88)

	New Oil (ATF)	New Oil (ATF)+SOD-1(7 vol. %)	Progress Rate %
Kinematic Viscosity 40℃ mm^2/s	33.7	42.2	25.2
100℃ mm^2/s	7.21	8.96	24.3
Viscosity Index	185	200	8.1
Acid Value mgKOH/g	1.72	1.48	-14.0
Shell 4-Ball Wear Test (ASTM D4172) mm	Seizure	0.46	―
Shear Stability (Ultrasonic Method)			
Kinematic Viscosity 40℃ mm^2/s (Before Test)	33.6	42.0	25.0
40℃ mm^2/s (After Test)	30.8	36.3	17.9
Rate 40℃ %	-8.10	-13.6	―
Kinematic Viscosity 100℃ mm^2/s (Before Test)	7.21	8.97	24.4
100℃ mm^2/s (After Test)	6.50	7.59	16.8
Rate 100℃ %	-9.83	-15.4	―

ァレックス焼付荷重の増加率は約 30%前後あり，これらの上昇率が後述するように還元添加剤が潤滑，摩擦，耐熱性と洗浄分散性などの複合作用とゴム，シール材の硬化防止といったトライボロジー性能の改善に寄与していることを示唆している．

3. 4 ATF オイルに及ぼす還元添加剤の影響と剪断安定度試験

ATF オイル（新油）に及ぼす還元添加剤（7 vol.%添加）の影響と剪断安定度試験の結果を表 3 に示す．動粘度は 40℃，100℃においての還元添加剤無しの ATF 油の 33.7，7.21 に対して還元添加剤添加後は 42.2，8.96 で各々25.2 と 24.3%の増加で，粘度指数は同様に ATF 油の 185 に対して添加後は 200 で 8.1%の増加であった。酸価は同様に 1.72 と 1.48mgKOH/g で-14.0%と減少した。シェル四球摩耗試験において還元添加剤無しの ATF オイルは焼付いたが，添加後は 0.46mm の摩耗径であった．よって ATF においても還元添加剤の動粘度に対する影響は約 25%増加し，粘度指数は 8.1%増加し，酸価もマイナスを示しており，これらの特性が還元添加剤の高粘度，耐熱，耐酸性安定性と耐摩耗性の向上に対する影響を示している．

さらに，剪断安定度試験は超音波試験法により 40℃と 100℃で測定した．40℃の動粘度は試験前で還元添加剤無しの ATF オイルで 33.6，還元添加剤を添加して 42.0 mm^2/s を示し，増加率は 25.0%で，試験後は同様に 30.8 と 36.3 mm^2/s を示し，増加率は 17.9%を示した．よって，40℃の動粘度は還元添加剤無しで -8.10%，還元添加剤を添加して-13.6%だった。100℃の動粘度は試験前で同様に 7.21 と 8.97 mm^2/s を示し，添加による増加率は 24.4%で，試験後は同様に 6.50 と 7.59 mm^2/s を示し，添加による増加率は 16.8%を示した．また，100℃の動粘度は還元添加剤無しで-9.83%，還元添加剤を添加して-15.4%だった。

したがって，剪断安定度試験により試験前の 40 と 100℃の動粘度の増加率は両者とも約 25%で，試験後においては同様に動粘度の増加率は両者とも約 17%で，試験前後における 40 と 100℃の動粘度の増加率は，各々約 63 と 57%を示しており，以上の結果から，本還元添加剤の添加によって粘度変化による剪断安定度の影響は無かった．

3. 5 エンジンオイルによるシェル四球摩耗試験おける還元添加剤の影響

某自動車メーカの純正オイル 5W-30（新油）を用いて還元添加剤によるシェル四球摩耗試験の摩擦，摩耗の影響を測定した．試験方法は回転速度 1200 rpm，負荷 40 kgf，温度 75℃，時間 60 min で実施した結果，図 2 に示すように添加剤無しの摩耗痕径は 0.46 mm で添加剤有りは 0.33 mm と還元添加剤の添加が 0.13 mm 少なく 28.3%の減少率を示し，これらの数値から本還元添加剤が摩擦，摩耗に対して有効な特性を有しており，トライボロジー性能の改善の可能性を示唆している．

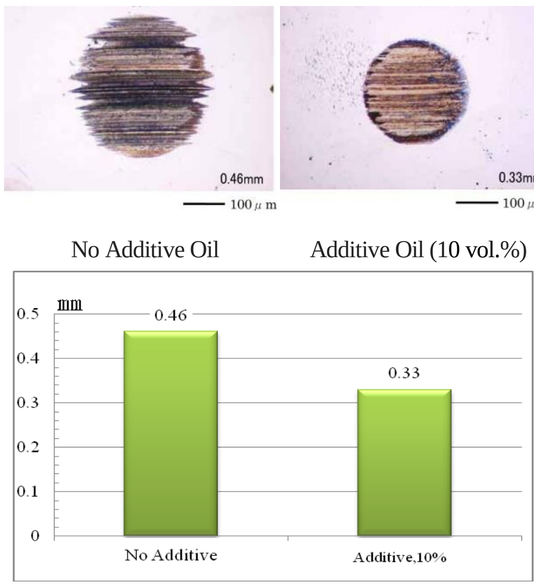


Fig. 2 Shell 4-Ball Wear Test (SN 5W-30)

4．還元添加剤を用いた実用例

4. 1 還元添加剤の添加によるカムシャフトの洗浄作用実験

還元添加剤の洗浄作用を確認するため，ガソリン車のカムシャフト（約 12 万 km 走行）を用い，図 3 に示す洗浄作用確認装置を製作して実験をした．図 3 に示すようにエンジンオイル 720cc に還元添加剤を 80cc（10 vol.%）添加した大気開放のビーカー内にカムシャフトを垂直に固定し，温度を 80℃±

0.1℃に温調器で制御し，撹拌羽根付きモーターでオイルをビーカー内で強制循環させた．その結果を図 4 に示すが，開始時と 481 時間後のカム軸端部を観察すると，481 時間後は開始時と比べて表面のコンタミ類がかなり洗浄され，カムフェイスも清浄されているのが認められる．今回は実機に比べるとオイルの作動圧力は 1/5 以上低い点が異なるが，本実験により還元添加剤の洗浄効果が確認された．

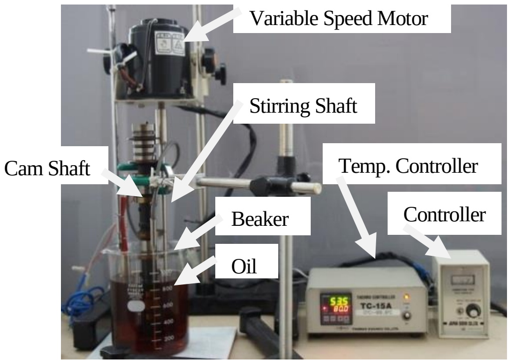
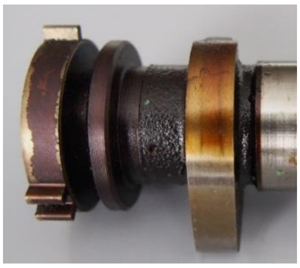
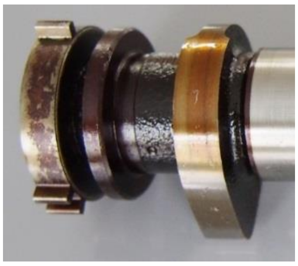


Fig. 3 Cleaning Action Confirmation Apparatus



Before Starting



After 481 hours

Fig. 4 Effects of Reducing Additive on Cam Shaft Cleaning

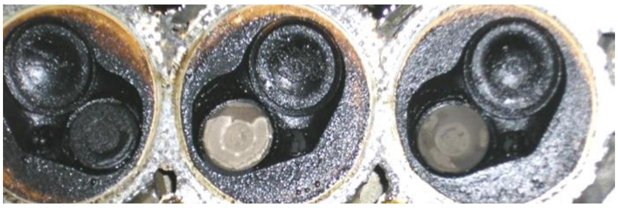
4. 2　還元添加剤よるガソリンエンジンの白煙対策例

沖縄県自動車整備振興会から軽自動車の白煙対策の依頼があり，100 km に約 1 L のエンジンオイル消費でバルブステムシールを交換しても図 5 に示すように白煙が止まらないとの状況であった．この症状から判断すると，オイルリング固着の可能性が考慮されるのでエンジンを分解したところ，図 6 に示すようにピストンリング，オイルリング固着と燃焼室のカーボン，デポジットの固着が認められた．よって，エンジンオイルに還元添加剤を 10vol.%添加したところ，白煙は数分後に止まった．その後，約 500 km 走行後にエンジンを再分解したところ，図 6 に示すようにピストンリングとオイルリングの固着解消と燃焼室のカーボン，デポジットが洗浄，除去され，プラグも清浄されているのが認められた．当然ながら，ピストンリング類の張力復元により，燃焼圧力も上昇してオイル消費，出力，燃費，排気ガスの改善効果が認められた．

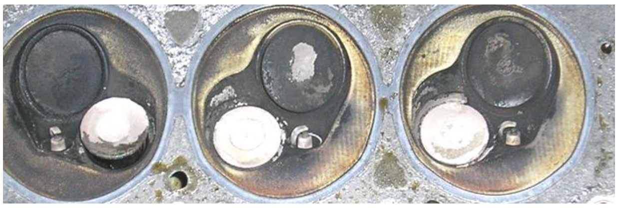
Fig. 5 Emission of White Smoke

Before Reducing Additives

After 500 km Driving



Combustion Chamber with Carbon and Deposit



Removal of Carbon and Deposit in Combustion Chamber

Fig. 6 Engine Disassembly Picture on White Smoke Car

4. 3　還元添加剤の添加よるガソリンエンジンの内部洗浄例

中古軽自動車の車検整備後，純正エンジンオイルに交換して約 1,000 km 走行後にエンジン分解をして内部点検をした．その後，エンジンを組み立てて還元添加剤を 10 vol.%添加して平均 50 km/h で 5089 km 走行後にエンジンを再分解して 1 回目の比較点検をした．さらに，再組み立て後に同条件で 5,032 km，合計 10,121 km 走行後に 2 回目の比較点検をした．その結果，1，2 回目とも還元添加剤の添加後は各所に清浄化が進行しており，特にシリンダボディのオイル通路孔に蓄積していた閉塞気味のヘドロ状のスラッジがほぼ清浄，除去されていた点は注目の成果であった．

還元添加剤の添加前後のシリンダヘッド上面内部とピストン側面の比較を図 7 に示す．シリンダヘッド上面内部は 2 回

目の比較で示すが，カムシャフトやカム駆動部分の表面に黒く固着していたスラッジ，カーボンのコンタミ類が分解，清浄化されて金属面が現れているのが分かる．ピストンについては，図７に示す１，２回目の側面写真からクラウン部はもとよりトップランドのスラッジ類が著しく分解，清浄化され，1st と 2nd ピストンリングの固着解放による復元とオイルリングの清浄化が認められた．さらに，ピストンリングとクラウン部を試験前と拡大比較すると，清浄化の状況がより明確に認められる．

排気ガスの改善例として，1500 cc の普通乗用車に還元添加剤を添加前後におけるアイドル時の CO，HC の測定値を表 4 に示す．この表から CO が 0.03 ％，HC が 45 ppm と大巾に改善されたことが分かる．このように還元添加剤はエンジン内のスラッジ，カーボン，ワニス，デポジット類を分解，洗浄し最初の金属加工面を還元でき，燃焼も改善されることが立証できた．

4. 4　CVT のジャダー対策に及ぼす還元添加剤の影響

最近，同一メーカの普通乗車の CVT 式オートマチックトランスミッションにおいて，発進時にジャダーを発生するクレームがあり，その対策として還元添加剤を 7 vol.%添加したところ，ジャダーが改善できることを確認した．その後，同社の複数の車種についても同様なクレームがあり，その概要を表５に示すが走行距離は約 47,000～12,2000 km で発生しており，特に 2002 年式が最も多く発生している．このクレーム対策として還元添加剤の効用が販売店に認められ，現在ではジャダー対策のディラー純正部品として認められている．

この改善理由は CVT のクラッチ表面に積層した金属の微細摩耗粉を含むコンタミ類が還元添加剤により分解，洗浄されて，最初の金属表面に復元して滑りが減少し，摩擦力が回復したためと考察される．このように還元添加剤は CVT のジャダー対策に対しても有効であることが立証された．

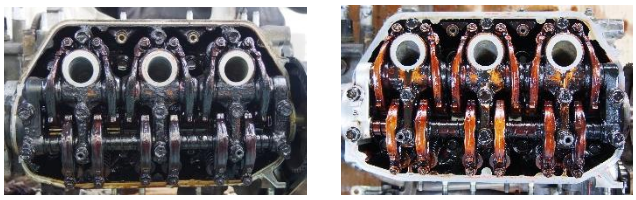
その他の改善例として過去 10 年間にわたり，ドライスター時の異音発生，変速ショック，エンジン異音，白煙，オイル消費，パワーステアリングの異音や AT クレームなどの対策として還元添加剤の添加により，全て解決できた実績を有する．

4. 5　D1 グランプリドリフト競技車のミッションオイルにおける還元添加剤の効果

最近，ドリフト競技が脚光を浴びているが，従来のモータースポーツと比較してかなり過酷なレースで，スタートから 500 m を 10 sec 前後で約 200 km/h に急加速後，急減速に耐えるエンジンとトランスミッション等に対する高度な耐久性と耐摩耗性を要求される．この車両のミッションオイルに還元添加剤の有無について各々2 時間走行して，そのオイルのシェル四球摩耗試験と元素分析を実施した結果を表 6 と図 8 に示す．この結果，摩耗痕径は還元添加剤無しで 0.93 mm，添加剤有りで 0.38 mm で 59.1 ％少なく，元素分析で最も顕著な有意差のあった元素は Fe で，添加剤無しで 15 mass%，添加剤有

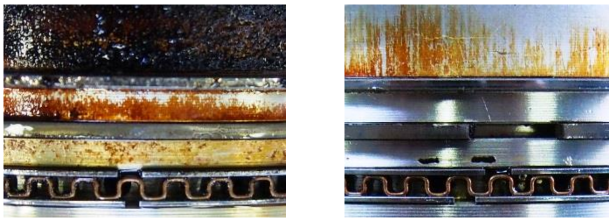
Table 4 Effects of Reducing Additives on Emission of CO, HC

	CO %	HC ppm
Before Addition	0.03	54
After Addition	0.00	9



Before Test of　　　After 10,121 km Driving

Cam Shaft Assembly in Cylinder Head



Before Test　　　After 10,121 km Driving

Enlarged View of Crown and Piston Rings

Before Test

After 5,089 km Driving　　　After 10,121 km Driving

Piston Side View of Before Test and After Addition

Fig. 7 Effects of Reducing Additive on Camshaft Assembly, Piston Side and Piston Rings

りで 4 mass%を示し，73.3 ％の改善率により摩擦と摩耗が著しく減少することを示した．さらに，Cu, Al, P などについても同様に 60.0～97.8%の改善が見られた．また，ドライバーによると添加剤有りの時，変速の UP，DOWN 時のショック音が静かでスムーズであったとの感想であった．

以上の結果により，本還元添加剤は過酷なミッションなどの潤滑状況においても，摩擦，摩耗などのトライボロジー性能を著しく改善ができ，高耐久性を明らかにした．その他，潤滑

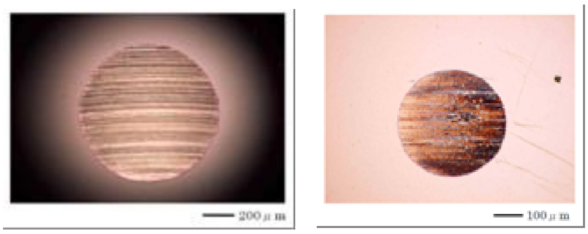
油中の異物の成分，形状などを観察し，状態の劣化状況を評価する手法[10]を用いることで，本添加剤の有効性をメカニズムとして示すことが期待できるが，そのことは今後の課題として検証を加える．

Table 5 Improvement of CVT Judder

Car Model	A	B	B	C	B
Model Year	2001	2002	2002	2002	2004
Mileage ×10^3km	122	47	113	72	57
Symptom	Judder at CVT Starting				
After Addition	Improvement of CVT Judder				

Table 6 Effects of Reducing Additives on Mission Oil in Drift Car Engine (Shell 4-Ball Wear Test)

Mission Oil	85W-250	85W-250 + SOD-1
Diameter of Wear Trace mm	0.93	0.38
Fe Element mass %	15	4



No Additive Oil　　Additive Oil (10 vol.%)

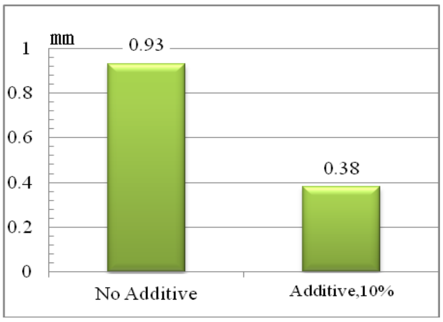


Fig. 8 Shell 4-Ball Wear Test (レース用 MT オイル 85W-250)

5．結　論

合成添加剤としてポリオールエステル（POE），ジエステル（DST）や植物油系エステル（VOE）化合物などを主成分とした独自の還元添加剤を開発し，トライボロジー性能に及ぼす影響を化学的作用と実用試験等により確認した結果，下記の結論が得られた．

1) ポリオールエステル，ジェステル系と植物油系エステル化合物などを主成分とした独自の還元添加剤は，潤滑面のスラッジ，ワニスなどのコンタミを洗浄，分解することにより潤滑面を再生し，摩耗や摩擦を低減させてトライボロジー性能を改善できる．
2) 本還元添加剤の化学的，物理的特性からトライボロジー性能の改善に寄与できることを立証することができた．
3) 本還元添加剤の効果は，実車を用いた分解，点検測定，潤滑油の耐久性試験や運転性試験により明らかになった．
4) 本還元添加剤はエンジンの摩擦損失，排気ガスや燃費低減のみならず，オートマティック・トランスミッションやデファレンシャルギアなどのトライボロジー性能にも画期的な効果を示す．
5) 本還元添加剤は自動車における過酷なモータースポーツであるドラフト競技において，摩擦，摩耗を著しく低減できることを明確にし，高耐久性を立証した．

あ と が き

本添加剤の化学的作用については，さらに工学的な解明を加えて実施する．最後に，本実験や測定に支援を得た（有）D1 ケミカルの園田智之取締役社長，竹川秀男技術部長を始め社員の方々に深甚の謝意を表します．また，実験，測定にご協力を得た久留米工業大学大学院自動車システム専攻長森　和典教授及び福岡県整備振興会山口和彦常務理事に謝意を表します．

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Effects on Tribology Performance of a Reducing Additive for Automobile Lubricant

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ABSTRACT

We developed a unique reducing additive composed primarily of polyolester, diester, and vegetable oil-based ester compounds in order to switch from boundary to fluid lubrication and improve tribology performance, and chemically and experimentally investigated its effects on tribology performance. To confirm the lubrication effects of this reducing additive, we added it to oil and chemically investigated the cleaning and dissolving of the ultra-fine particle layer of contaminants, etc., adhering to lubrication pathways and sliding surfaces, and performed commercialization testing using actual vehicles. We found that this additive washed away and dissolved sludge, varnish, and other contaminants from lubricated surfaces, improving their lubrication properties and their tribology performance by reducing abrasion and friction.

INTRODUCTION

In recent years demand has grown for improved lubricating oil tribology performance in automobiles, to provide not only greater vehicle performance, but also to reduce their resource usage and environmental impact. Ester-based synthetic lubricating oil has been developed to provide greater lubrication performance, and to reduce friction and wear, in order to satisfy this demand, but there is still a great deal of room for improvement. These ester-based synthetic lubricating oils are made from organic (fatty) acids and alcohol, but attention is increasingly being drawn to the use of polyol ester-based synthetic lubricating oil[(1),(2)], originally widely used in jet engines, and in automobiles as well.

This synthetic lubricating oil offers many features, such as greater low-temperature fluidity, thermal stability, and oxidation stability than mineral lubricating oils. It can be used at high viscosity levels over a wide range of temperatures. It offers excellent lubrication and is clean, dispersible, and biodegradable. However, it is prone to hydrolysis, absorbs humidity, and is limited regarding which rubber, sealing materials, resins, and paints it can be used with. It is also expensive, but it is said to offer the best tribological performance, require the least resources and energy, and be the most functional of all of the synthetic lubricating oils[(1),(2)].
Synthetic lubricating oils are made by mixing lubricating oil additives[(3),(4)], including detergent dispersants, antioxidants, extreme pressure agents, rust-preventive agents, viscosity index improvers, pour point depressants, antifoaming agents, and friction-reducing additive. The compounds made of these additive are undergoing tremendous advances in line with the development of automobiles and their environments. The ester-based synthetic lubricating oils believed to be essential for the latest automobiles are synthetic lubricating oils which use polyolester and diester additive as their additive.

In this research, in order to develop automobile lubricating oil with improved lubrication, wear, and friction performance for use in engines, transmissions, and differentials, we developed a unique reducing additive (SOD-1) composed primarily of polyolester (POE), diester (DST), and vegetable oil-based ester (VOE) compounds as secondary synthetic additive, and chemically and experimentally investigated its effects on tribology performance.

LUBRICATING OIL ADDITIVE TRENDS IN THE LATEST ENGINES

Looking at the trends in the lubricating oil additive used in the latest engines[(3)], incompletely combusted soot (C) in diesel engines reacts with NO and SO_3 created by fuel combustion, accounting for the majority of deposits. Lacquer and varnish are believed to be low molecular fuel oxidation compounds formed near the end of the compression stroke. There are reports of fuel and lubrication additive contributing to reductions in the amounts of deposits in combustion chambers[(5)]. In exhaust gas post-processing calcium sulfate-based lubricating oil additive[(6)] is used in order to reduce the accumulation of DPF ash, collecting soot and ash. The presence of soot in lubricating oil in crank cases causes friction in valves and liners, so there are reports[(7)] of commercialization of products reducing friction for both valves and liners through the use of appropriate lubricating additive.

The main cause of deposits in gasoline engines is said to be, at high temperatures, oxidation of base oil, and at low temperatures fuel and oxidization products. These substances form resin-like or carbon-like substances inside crank cases as components contained in blow-by gas, accumulating inside engine lubricating oil pathways and parts. Wear resistance improving additives(9), such as new lubricating oil additives(8) extending the low friction properties for gasoline engines, zinc dialkyldithiophosphate (ZnDTP), containing no sulfur, and phosphate ester, have also been developed. Therefore, the key attributes of lubricating oil additive required to counter deposits in both types of engines are detergent dispersal, solubility, and oxidation inhibition.

REDUCING ADDITIVE

THE CONCEPT BEHIND THIS REDUCING ADDITIVE

Contaminants such as deposits, varnish, sludge, etc. which adhere to the lubricated and sliding surfaces of automobile engines, transmissions, and differentials, etc., accumulate over time, having a negative impact on their tribology performance (lubrication, wear, and friction). Normally this is improved by flushing them with kerosene-based cleaning agent when performing oil replacement, but as Figure 1. shows, it is not possible to completely remove all contaminants, which build up three layers of general dirt, adsorbed molecules, and oxidation film. The removed contaminants also often clog up lubricating oil pathways, so this cleaning method is not very effective, and has its own risks.

Therefore, this reducing additive, a blend of additive made primarily with POE, DST, and VOE-based compounds, is added to engine, manual transmission, power steering, and differential gear oil (10vol.% mix) and to automatic transmission oil (7vo.l% mix), lubricating while also washing off and removing contaminants. It exposes the initial processed metal surface, greatly reducing friction loss and ensuring sufficient oil clearance. This unique method improves lubrication while reducing wear and friction.

The concept behind this reducing additive is that it washes away, dissolves, and removes contaminants from lubricated sliding components, exposing their initial lubrication surfaces (the face of the processed metal), using reducing action improve its tribology performance (lubrication, wear, and friction characteristics). The main problems with this reducing additive are that it is prone to hydrolysis, absorbs humidity, and swells and hardens rubber and sealing materials. These problems have been countered by adding petroleum-based oil and grease components. This approach has been highly effective.

CHEMICAL STATE OF REDUCING ADDITIVE AND ITS EFFECT ON ENGINE OIL

Table 1. shows the chemical state of the reducing additive and its effect on engine oil. Its kinematic viscosity, at 40°C and 100°C, is 610 and 92.4 mm^2/s, respectively. Its viscosity index is 243, its flash point is a high 170°C, and its pour point is a low -42.5°C. It offers the high viscosity, temperature-resistant viscosity, flame resistance, and low-temperature fluidity of POE and DST-based compounds. Indiana Stirring Oxidation Test (ISOT) testing found it to have a viscosity ratio of 1.06, with little viscosity change before and after oxidation. There was only a -0.80 mgKOH/g change in oxidation after heating, and its lacquer rating was found to have no deposits, indicating that there were no oxidants or sludge mixed in.

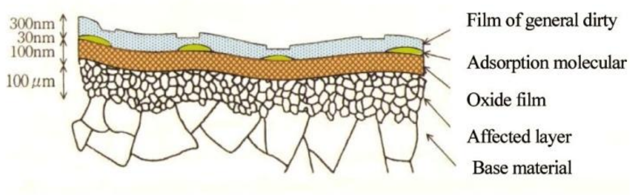


Figure1. Components of Solid Surfaces

Table 1. Oxidation Stability Test of Reducing Additive (SOD-1) JIS K2514

Kinematic Viscosity 40°C mm^2/s	610.0
100°C mm^2/s	92.4
Viscosity Index	243
Pour Point °C	- 42.5
Flash Point (PM) °C	170.0
Ash mass %	2.305
Oxidation Stability (ISOT)	
Viscosity Ratio	1.06
Increased Oxidative mgKOH/g	- 0.80
Lacquer Rating	No Deposits

CHEMICAL STATE WHEN REDUCUCING ADDITIVE WAS ADDED TO ENGINE OIL

Table 2. shows the chemical state when 10vol.% by volume of reducing additive was added to fresh engine oil (SN5W-30). The kinematic viscosity of the engine oil without reducing additive at temperatures of 40°C and 100°C was 60.9 and 10.5 mm^2/s respectively, but after adding reducing additive was 77.6 and 13.5 mm^2/s, respectively, increases of 27.6 and 28.6%. Its control viscosity index of 162 rose by 10.5% to 179 after adding additive. FALEX seizure load, likewise, rose by 33.3%, from 750 to 1000 lbs. Hot tube testing, which evaluates oil heat resistance and cleaning dispersal on a 10 point scale, rose 14.3%, from 7 to 8, after adding reducing additive. Rubber swelling, likewise, rose by 46.4%, from 5.6 to 8.2%.

These results show that the reducing additive had major chemical effects. In particular, the kinematic viscosity and FALEX seizure loads improved by approximately 30%. These improvement rates indicate that the reducing additive contributed to tribology performance improvements through compound effects, such as improving lubrication, friction, heat resistance, and cleaning dispersion, and by preventing the hardening of rubber and sealing materials.

Table 2. Effects of Reducing Additive on Chemistry of Engine Oil (SN5W-30)

	SOD-1	SN5W-30	SN5W-30+SOD-1(10vol.%)	Progress Rate %
Kinematic Viscosity 40°C mm^2/s	610.0	60.9	77.6	27.4
100°C mm^2/s	92.4	10.5	13.5	28.6
Viscosity Index	243	162	179	10.5
FALEX Seizure Load (ASTM) lbs	-	750	1000	33.3
Hot Tube Test 289°C Mark	-	7	8	14.3
Rubber Swelling Degree vol.%	-	5.6	8.2	46.4

Table 3. Effects of “SOD-1” on ATF Oil Chemistry and Shear Stability (Ultrasonic Method, JPI-5S-29-88)

	New Oil (ATF)	New Oil (ATF) +SOD-1(7 vol.%)	Progress Rate%
Kinematic Viscosity 40°C mm^2/s	33.7	42.2	25.2
100°C mm^2/s	7.21	8.96	24.3
Viscosity Index	185	200	8.1
Acid Value mgKOH/g	1.72	1.48	-14.0
Shell 4-Ball Wear Test (ASTM D4172) mm	Seizure	0.46	—
Shear Stability (Ultrasonic Method)			
Kinematic Viscosity 40° C mm^2/s (Before Test)	33.6	42.0	25.0
40°C mm^2/s (After Test)	30.8	36.3	17.9
Rate 40°C %	-8.10	-13.6	—
Kinematic Viscosity 100°C mm^2/s (Before Test)	7.21	8.97	24.4
100°C mm^2/s (After Test)	6.50	7.59	16.8
Rate 100°C %	-9.83	-15.4	—

EFFECTS OF REDUCING ADDITIVE ON ATF AND SHEAR STABILITY TESTING

Table 3. shows the effects of reducing additive (7vol.% mix) on ATF oil (new oil) and the results of shear stability testing. Before adding reducing additive, the kinematic viscosity of ATF oil at temperatures of 40°C and 100°C were 33.7 and 7.21, respectively. After adding reducing additive, these increased by 31.2 and 24.3% to 42.2 and 8.96, respectively. Likewise, the viscosity index of ATF oil without reducing additive was 185, but rose by 8.1% to 200 after adding additive. The acid value fell by -14.0%, from 1.72 to 1.48 mgKOH/g. ATF oil without reducing additive seized during shell 4-ball wear testing, but after adding additive its wear diameter was 0.46mm. The reducing additive increased the kinematic viscosity of the ATF by roughly 25%, increased its viscosity index by 8%, and reduced its acid value. These property changes indicate the improvements the reducing additive made to high viscosity, heat resistance, acid resistance stability, and wear resistance.

Furthermore, shear stability testing using ultrasound was performed at temperatures of 40 and 100°C. The kinematic viscosity at 40°C was 33.6 for ATF containing no reducing additive, before testing. With reducing additive it was 42.0 mm^2/s, an increase of 25.0%. After testing it was 30.8 and 36.3 mm^2/s, an increase of 17.9%. Therefore, the kinematic viscosity without reducing additive at 40°C was -8.10%, but with reducing additive was -13.6%, an increase of 67.9%. Likewise, the kinematic viscosities before testing at 100°C were 7.21 and 8.97 mm^2/s. The additive increased the viscosity by 24.4%. After testing the viscosities were 6.50 and 7.59 mm^2/s, with the additive increasing viscosity by 16.8%. The kinematic viscosity without reducing additive at 100°C was -9.83%, but with reducing additive was -15.4%, an increase of 57.1%.

Therefore the shear stability testing found that the reducing additive increased pre-test kinematic viscosities by approximately 25%, both at 40 and 100°C, and increased post-test kinematic viscosities by approximately 17%, for both temperatures. The kinematic viscosity increase rates at 40 and 100°C were roughly 63 and 57%, respectively. Based on this, the reducing additive can be considered to provide sufficient shear stability, since there is no reduction of the kinematic viscosity.

EFFECT OF REDUCING ADDITIVE ON RESULTS OF ENGINE OIL SHELL 4-BALL WEAR TEST

Shell 4-ball wear testing was used to measure the friction and wear effects of reducing additive on a certain automobile manufacturer's oil 5W-30 (new oil). The testing was performed using a rotational speed of 1200 rpm, load of 40 kgf, temperature of 75°C, and time of 60 min. As Figure 2. shows, the wear scar diameter of the oil without additive was 0.46 mm, while for the oil with additive it was 0.33 mm. The reducing additive reduced the wear scar size by 0.13 mm, or 28.3%. These results show that the reducing additive is effective for improving friction and wear, and indicates the potential for it to contribute to improved tribology performance.

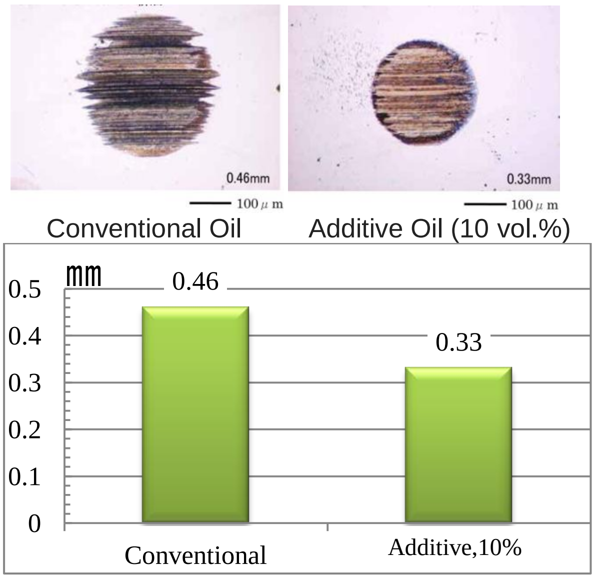


Figure 2. Shell 4-Ball Wear Test (SN 5W-30)

EXAMPLE OF PRACTICAL APPLICATION OF REDUCING ADDITIVE

REDUCING ADDITIVE CAMSHAFT CLEANING ACTION EXPERIMENT

In order to confirm the cleaning action of the reducing additive, a cleaning action confirmation device, shown in Figure 3, was fabricated and used to perform an experiment on a gasoline-powered automobile's camshaft (used for approx. 120,000km). As Figure 3. shows, 80cc of reducing additive were added to 720cc of engine oil (10vol.%) in an open beaker. The camshaft was then vertically suspended in the solution. The temperature was kept at 80°C±0.1°C, and a motor with an agitating blade was used to forcibly circulate the oil solution in the beaker.The results are shown in Figure 4. The camshaft end was observed at the start of the experiment, and then after 481 hours. Compared to its state at the start of the experiment, after 481 hours a great deal of the varnish and contaminants had been washed off, and the cam face was confirmed as being clean. The oil in the experiment was 1/5 heavier or more than that used in actual automobiles, but this experiment confirmed the cleaning effectiveness of the reducing additive.

EXAMPLE OF USE OF REDUCING ADDITIVE AS GASOLINE ENGINE WHITE SMOKE COUNTERMEASURE

A request was received from the Okinawa Prefecture Japan Automobile Service Promotion Association for minivehicle white smoke countermeasures. An automobile's valve stem shield was replaced, but it was going through approx. 1L of engine oil per 100km and producing continuous white smoke, as shown in Figure 5. Given these symptoms, it was determined that there was a possibility that the oil ring had become stuck, so the engine was disassembled. As Figure 6. shows, the compression ring and oil ring had become stuck, and there were carbon and deposits in

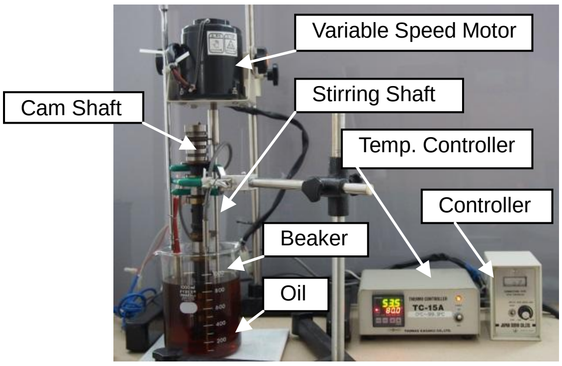


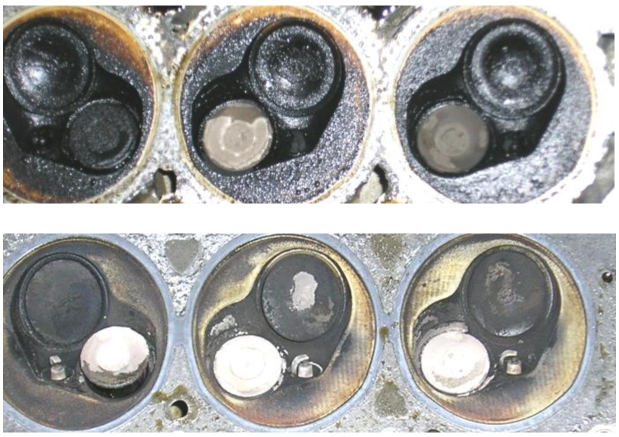
Figure 3.Cleaning Action Confirmation Apparatus

Before Starting After 481 Hours

Figure 4. Effects of Reducing Additive on Camshaft Cleaning

Figure 5. Emission of White Smoke

Before Reducing Additive After Driving 500 km



Removal of Carbon and Deposits in Combustion Chamber.

Figure 6. Disassembled Engine of White Smoke Car

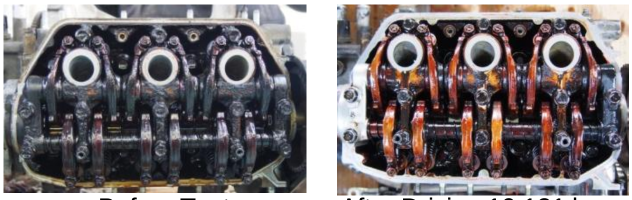
the combustion chamber. Reducing additive was added to engine oil (10vol.% mix), and the white smoke stopped after several minutes. The vehicle was driven for approximately 500km more, and the engine was disassembled again. As Figure 6. shows, the piston ring and oil ring were no longer stuck, the carbon and deposits had been cleaned off and removed from the combustion chamber, and the plug was also cleaned. The restoration of piston ring tension also resulted, of course, in increased combustion pressure, improving oil consumption, output, fuel efficiency, and exhaust gas.

EXAMPLE OF USE OF REDUCING ADDITIVE FOR CLEANING OF INSIDE OF GASOLINE ENGINE

After performing vehicle inspection and maintenance on a used minivehicle, and replacing the oil with new oil, the vehicle was driven for 1,000 km, and then the engine was disassembled and inspected. The engine was then reassembled and a 10 vol.% mix of reducing additive was added to the oil. The vehicle was then driven for 5089 km at an average speed of 50 km/h. After driving, the engine was disassembled and inspected again, and the inspection results were compared against those of the initial inspection. The engine was then reassembled again and driven, under the same conditions, for another 5,032 km, for a total of 10,121 km. The engine was disassembled and inspected again, and the results of the inspection were compared against those of the previous inspections. Both inspections performed after the addition of the reducing additive found that the interior parts of the engine had been cleaned, and, in particular, that the thick sludge which had accumulated and almost clogged the cylinder body oil passage ports was almost completely washed away.

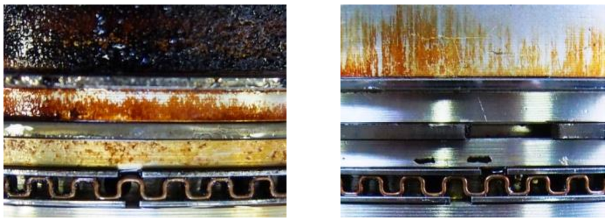
Figure 7. shows the interior of the cylinder head top and piston side after adding the reducing additive. The cylinder head top photo comparison with the second disassembly inspection results shows that the black sludge and carbon contaminants which had tightly adhered to the surface of the camshaft and cam drive were dissolved and washed away, exposing the metal surface. The 1st and 2nd disassembly inspection photos of the piston, shown in Figure 7, show that the sludge which had accumulated on the top land of the piston was significantly dissolved and washed away, the 1st and 2nd piston rings had been restored by re-exposing them, and the oil ring had been cleaned. Furthermore, comparing expanded photos of the piston ring and crown before and after testing made the extent of the cleaning results even clearer.

Table 4. shows an example of improvements to exhaust gas, comparing CO and HC measurements for a standard 1500 cc vehicle when idling, before and after adding reducing additive. As this table shows, both CO and HC were dramatically improved, CO by 0.03% and HC by 45 ppm. This evidence proves that the reducing additive can dissolve and wash away sludge, carbon, varnish, and deposits from engine interiors, restoring their initial processed metal surfaces and improving seizing.



Before Test After Driving 10,121 km

Camshaft Assembly in Cylinder Head



Before Test After Driving 10,121 km

Enlarged View of Crown and Piston Rings

Before Test

After Driving 5,089 km After Driving 10,121 km

Piston Side View Before Test and After Addition

Figure 7. Effects of Reducing Additive on Camshaft Assembly, Piston Side and Piston Rings

Table 4. Effects of Reducing Additive on Emission of CO, HC

	CO %	HC ppm
Before Addition	0.03	54
After Addition	0.00	9

THE IMPACT OF REDUCING ADDITIVE ON CVT JUDDER COUNTERMEASURES

Recently there have been complaints about judder when starting a CVT automatic transmission for regular passenger vehicles of a certain manufacturer. We confirmed that adding a 7 vol.% mix of the reducing additive reduced judder. Similar complaints were received for multiple models from the same manufacturer. Table 5. shows an overview. Judder generally began after driving 47,000 to 122,000 km, and was especially common in 2002 models. The effectiveness of the reducing additive at reducing

Table 5. CVT Judder Improvement

Model	A	B	B	C	D
Model Year	2001	2002	2002	2002	2004
Mileage $\times 10^3$km	122	47	113	72	57
Symptom	Judder at CVT Starting				
After Addition	Improvement of CVT Judder				

judder was recognized by dealerships, and the reducing additive is now recognized by dealers as an official judder countermeasure product of the manufacturer.

The reducing additive is believed to reduce judder by dissolving and washing away built-up contaminants on the surface of the CVT clutch, which include fine metal particles, exposing their metal surfaces again, reducing slipping and restoring their friction capabilities. In this way the reducing additive has also been proven effective at reducing CVT judder.

Other examples of improvements over the past 10 years include the use of the reducing additive to effectively reduce abnormal sounds during dry engine start-up, gear shift shock, abnormal engine sounds, white smoke, excessive oil consumption, abnormal power steering sounds, and AT complaints.

EFFECTS OF REDUCING ADDITIVE ON D1 GRAND PRIX DRIFT CAR TRANSMISSION OIL

In recent years, drift racing has gained wider recognition. Compared to conventional motor sports, drift racing is much harder on vehicles. Engines and transmissions must offer high levels of durability and wear resistance in order to withstand accelerating to 200 km/h in just 10 sec over the 500 m span after the start of the race, and then suddenly decelerating again.

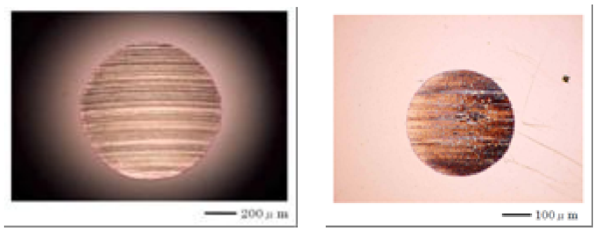
Figure 8, 9 and Table 6 show the results of oil shell 4-ball wear testing and elementary analysis performed on drift cars after two hours of driving, one with standard transmission oil and one with transmission oil containing reducing additive. The car with oil that did not contain reducing additive had a wear scar diameter of 0.93 mm, where as the one which contained reducing additive had a wear scar diameter that was 59.1% smaller, at 0.38 mm. Elementary analysis found the greatest difference in the amount of Fe. The result for the car with additive-free oil was 24.9 mass%, where as for the car with oil containing additive it was 1.2 mass%, an improvement of 95.2 %, indicating a significant reduction in friction and wear. Other elements were similarly affected, Cu 60.0, Al 93.5, Na 100, P 97.8 and Ca 80.0% of improvement.

The driver of the car whose oil contained additive noted that there was less shock noise when shifting up or down gear, and that shifting was smoother. The above results show that this reducing additive also greatly improves tribology performance, such as friction and wear, as well as durability, even in harsh transmission lubrication situations.

In addition, other benefits can be expected to be observed in the form of reduced foreign matter by proper oil analysis techniques[10]. This could be an area of further investigation to validate these assumptions.

Table 6. Acid Dissolution Metal Analysis of Reducing Additive on Transmission Oil in Drift Car Engine (Shell 4-Ball Wear Test)

Transmission Oil	85W-250	85W-250 + SOD-1	Progress Rate %
Diameter of Wear Scar mm	0.93	0.38	59.1
Element Mass %			
Fe	24.9	1.2	95.2
Pb	0.0	0.0	0.0
Cu	0.5	0.2	60.0
Cr	0.2	0.0	100
Al	3.1	0.2	93.5
Ni	0.0	0.0	0.0
Sn	0.1	0.0	100
Si	0.2	0.0	100
B	0.1	0.0	100
Na	0.4	0.0	100
P	4.5	0.1	97.8
Zn	0.5	0.5	0
Ca	0.5	0.1	80.0
Ba	0.1	0.0	100
Mg	0.0	0.0	0.0
Mo	0.0	0.0	0.0
V	0.0	0.0	0.0



Conventional Oil   Additive Oil (10 vol.%)

Figure 8. Wear Tracks of Shell 4-Ball Wear Test

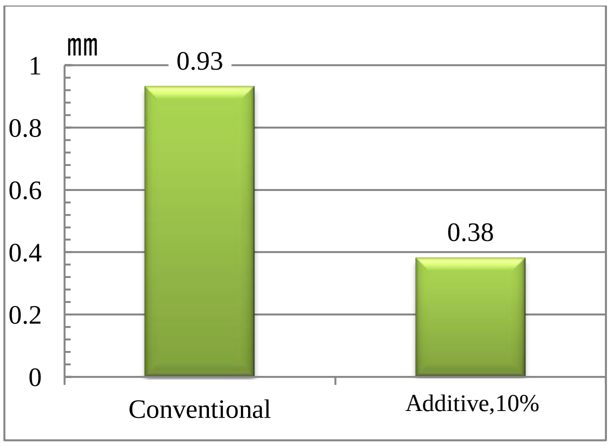


Figure 9. Shell 4-Ball Wear Test (SUNOCO 85W-250)

CONCLUSION

We developed a unique reducing additive composed primarily of polyolester (POE), diester (DST), and vegetable oil-based ester (VOE) compounds as synthetic additive, and chemically and experimentally investigated its effects on tribology performance. We reached the following conclusions.

1) The unique reducing additive composed primarily of polyolester, diester system, and vegetable oil-based ester compounds is effective at dissolving and washing away sludge, varnish, and other contaminants from lubricated surfaces, restoring them to their original conditions and improving their tribology performance by decreasing wear and friction.

2) The chemical and physical properties of this reducing additive have been confirmed as contributing to improved tribology performance.

3) The effects of this reducing additive have been proven through engine disassembly, inspection and measurement, lubricating oil durability testing, extension of interval of oil exchange and drivability testing on actual vehicles.

4) This reducing additive not only reduces engine friction loss, exhaust gas, and fuel consumption, but also has revolutionary tribology performance benefits for automatic transmissions, differentials, etc.

5) This reducing additive has been proven to offer high durability and exceptional reductions to friction and wear in drift racing, a motor sport which is punishing on cars' engines.

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DEFINITIONS/ABBREVIATIONS

ATF：Automatic Transmission Fluid
C: Carbon
CVT: Continuously Variable Transmission
DPF: Diesel Particulate Filter
DST: Diester
FALEX: FALEX Seizure Load Testing Machine
ISOT: Indiana Stirring Oxidation Test
NO: Nitric Oxide
POE: Polyolester
SO_3: Sulfur Trioxide
SOD-1: Unique Reducing Additive For Automobile Lubricant Oil
VOE: Vegetable Oil-Based Ester
ZnDTP: Zinc Dialkyldithiophosphat