Statistical tools such as test modeling are invaluable in quickly and efficiently understanding the effects of additives on grease performance. These tools can also save time and capital investment in expensive rig or field testing. In addition, insight can be gained about the properties a grease formulation needs to perform well in an application.

BY RICHARD BEERCHECK

Formulating high-temperature greases is particularly challenging, Joseph Kaperick, Afton Chemical Corp., told delegates at ELGI's Annual General Meeting in Barcelona in April. "Modern machinery continues to be designed to meet higher efficiency and power density targets, resulting in higher operating temperatures that punish lubricants with increased oil oxidation, decreased ability of the grease thickener to hold oil in place, increased oil viscosity and overall reduction in hydrodynamic protection." Combined with the use of lower viscosity oils to meet efficiency goals, these factors can lead to higher torque, increased wear and less protection for grease-lubricated bearings.

"A properly-designed high-temperature grease formulation results in a lubricant that is better able to withstand the effects of high heat while providing less wear and tear on bearings and increased lubrication intervals," continued Kaperick, who is based in Richmond, Virginia. This enables bearings to run more efficiently while maintaining long useful life without increasing overall lubricant use. He then described several screening models that Afton developed to evaluate the high-temperature performance of greases.

What's Significant?

Kaperick explained that the analysis was divided into three sections, each representing a different aspect in understanding high-temperature performance. In the first, Afton researchers developed a model to correlate simple and inexpensive bench tests to the FAG FE9 bearing test run at 160 degrees C.

Greases used in this example were lithium complex formulations incorporating an ISO 220 polyalphaolefin and diluted to NLGI 2 consistency. Test conditions are described in DIN 51821. Bearing failure occurs when torque rises above a preset limit, indicating that the grease is losing its ability to effectively lubricate the bearing.

"We calculated 10 percent (L10) and 50 percent (L50) grease life values using Weibull statistical analysis of five bearing runs," Kaperick said. These numbers indicate when 10 percent or 50 percent of the bearings are expected to have failed under the test conditions. According to DIN 51825 and ISO 6743, the maximum operating temperature of a grease is determined by the temperature at which the L50 value in the FE9 test is greater than 100 hours.

Kaperick noted that previous work had determined that five bench tests were statistically significant and could be used to model the FE9 test. From these properties, a statistical model was built based on the L50 data for each grease formulations from the FE9 tests in unshielded bearings and the significant bench test properties were determined.

"This testing showed that rheological properties – yield point of the aged grease and modulus at flow point for the aged grease – account for over one-half of the total model while dropping point and bearing torque of the aged grease along with some lab-to-lab variability make up the rest," Kaperick reported. Mini Traction Machine friction was not statistically significant in this model.

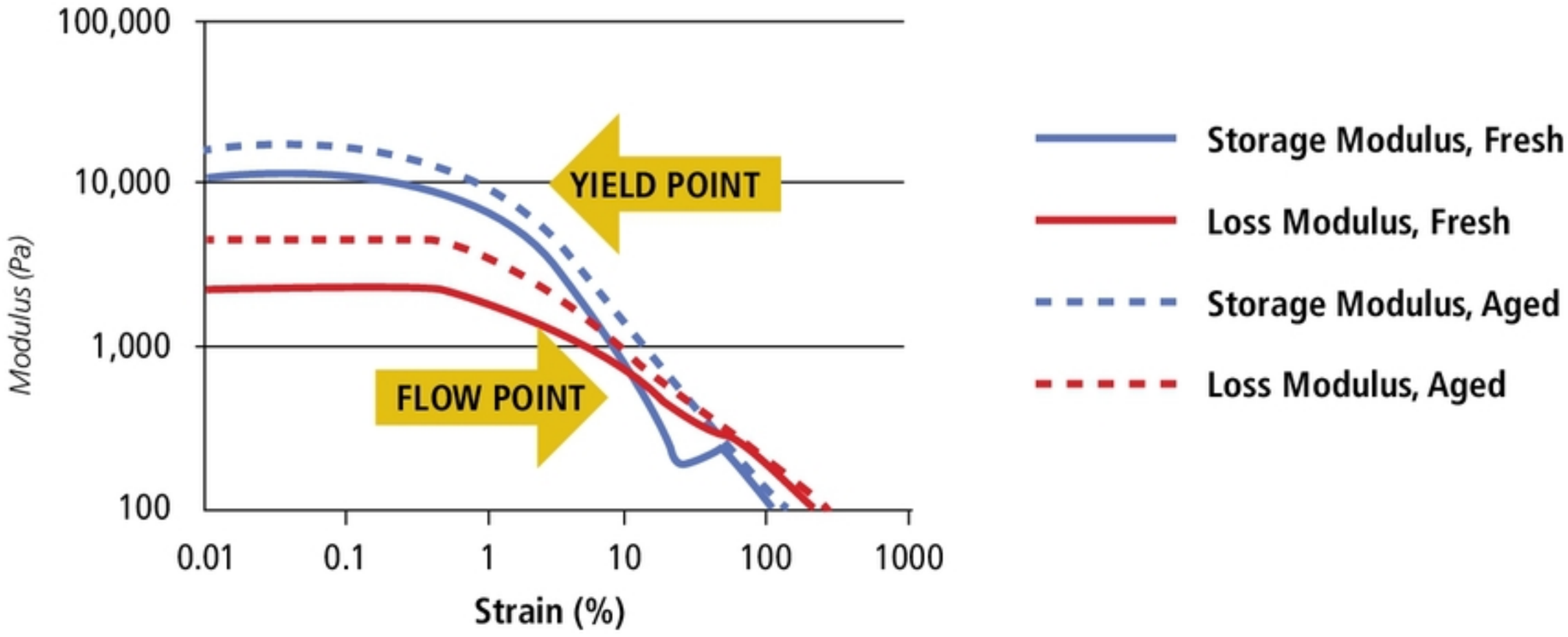
"Surprisingly, the model predicted better results at the high end (L50 greater than 100 hours) than were actually achieved when the model was tested," he said. Additional work showed that the significance of the factors varied with the amount of time in the FE9 test. This led Afton to examine two additional models.

Significant Factors in FAG FE9 Model

Attribute	Test	Grease Condition	Property
Dropping Point	ASTM D2265	New	Hold reservoir of oil at high enough temperature to replenish the contact
MTM Friction	Mini Traction Machine (70 N, 140°C, 500 mm/s, SRR-20%)	New	Maintain low torque in rotating bearing
Modulus	Rheometer (strain sweep @ 160°C) measured at flow point	Aged 100 h @ 160°C	Grease stiffness -Loose enough to release oil -Stiff enough to hold in place
Yield Point	Rheometer (strain sweep @ 160°C) calculated	Aged 100 h @ 160°C	Strong enough to hold grease & oil in bearing
Bearing Torque	Rheometer with bearing attachment, FE9 bearing torque @ 160°C	Aged in FE9 bearing for 100 h @ 160°C	Ability of aged grease to maintain low torque

Source: Afton

Determining Yield Point & Modulus at Flow Point



Source: Afton

The 50 Hour Frontier

Kaperick explained that the findings from this modeling seem to match well with discussions in the literature. "There are roughly two phases for grease lubrication in bearings running under constant conditions, the churning phase and the bleeding phase." Afton termed these phases the less than 50-hour model and the greater than 50-hour model.

"In the less than 50-hour model, MTM friction is statistically significant and rheological factors of the aged grease (yield point and modulus

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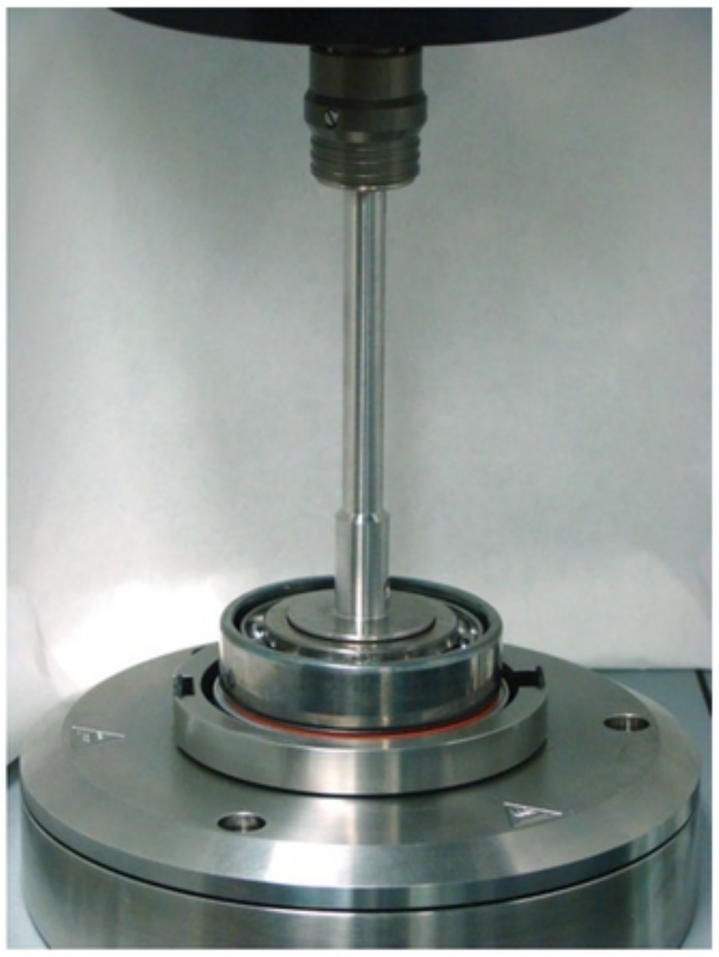

FAG FE9 bearing test set-up.

at flow point) are even more significant, correlating to almost two-thirds of the model," he said. Dropping point and bearing torque of the aged grease are not significant.

In the greater than 50-hour model, MTM friction and yield point of the aged grease are not significant. "Dropping point explains a little over one-third of the variation in the model while bearing torque and modulus at flow point of the aged grease combined cover about the same," Kaperick said. Differences between labs are significant to about the same extent in both models.

Additional findings are related to the shielding of the bearing. "Shields tend to be used in applications running at higher temperatures to help hold the grease in place," said Kaperick. Test results showed that shielding helped offset the lower rheological performance that reduces L50 grease life at higher temperatures. "Because it is held in place by the shield, the grease does not require a higher dropping point and aged modulus that apparently help hold it in place in unshielded bearings," Kaperick concluded.

What It All Means

"Models like these not only allow

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Photo courtesy of Afton

relatively easy prediction of rig test performance using quick and easy bench tests," said Kaperick, "they also provide the formulator with more insight into the properties that affect various aspects of grease performance in that test." By separating these aspects, individual properties such as friction or dropping point can be addressed in the appropriate fashion.

"Building such a model does not result in a one-size-fits-all solution to modelling any grease for a rig test," Kaperick cautioned. "It is not probable that such a model would fit different base oil or viscosity modifier combinations, different types of thickeners or different high-temperature rig tests."

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As a further effort to develop screening tests that simulate full rig tests, Afton ran an experiment using a different lithium complex base grease blended with Group II base oil to NLGI 2 consistency. The grease contained an ashless additive package at 3 percent treat rate. Various antioxidant combinations were investigated to identify three candidates that were tested alongside a commercial high-temperature grease product.

The greases were aged for 80 hours at 160 degrees C to simulate heating in the bearing test, and the modulus at flow point and yield point of the new and aged greases were measured. The testing showed that the yield point, modulus of the aged grease and dropping point do not appear to correlate to bearing life. "The modulus of the fresh grease does correlate and may be significant," Kaperick said.

"Pressurized Scanning Differential Calorimetry data is interesting in that the two greases with the most reserve antioxidancy gave the lowest bearing life," he added. Also, infrared scans showed that each grease undergoes about the same amount of oxidation in the high-temperature bearing test. "This may merely show that once the grease has oxidized to this extent it is likely to fail. The PDSC data shows that all four greases have some reserve antioxidancy," Kaperick said, "but we do not yet understand if oxidation is a driving force in the failure mode of these greases."

Antioxidant Effect

The two screening models focus on understanding grease behavior in rig tests, which can provide a better understanding of performance such as friction and high-temperature stability. Antioxidants can be a critical part of a formulation by extending the useful life of the grease or increasing protection at higher temperatures.

Therefore, Afton studied the performance and potential synergies of antioxidant combinations. "The earlier studies implied that oil degradation leading to sludge formation or viscosity increase will have a negative impact on aged bearing torque or the ability of a grease to maintain the rheological properties that may be critical to high-temperature performance," Kaperick explained.

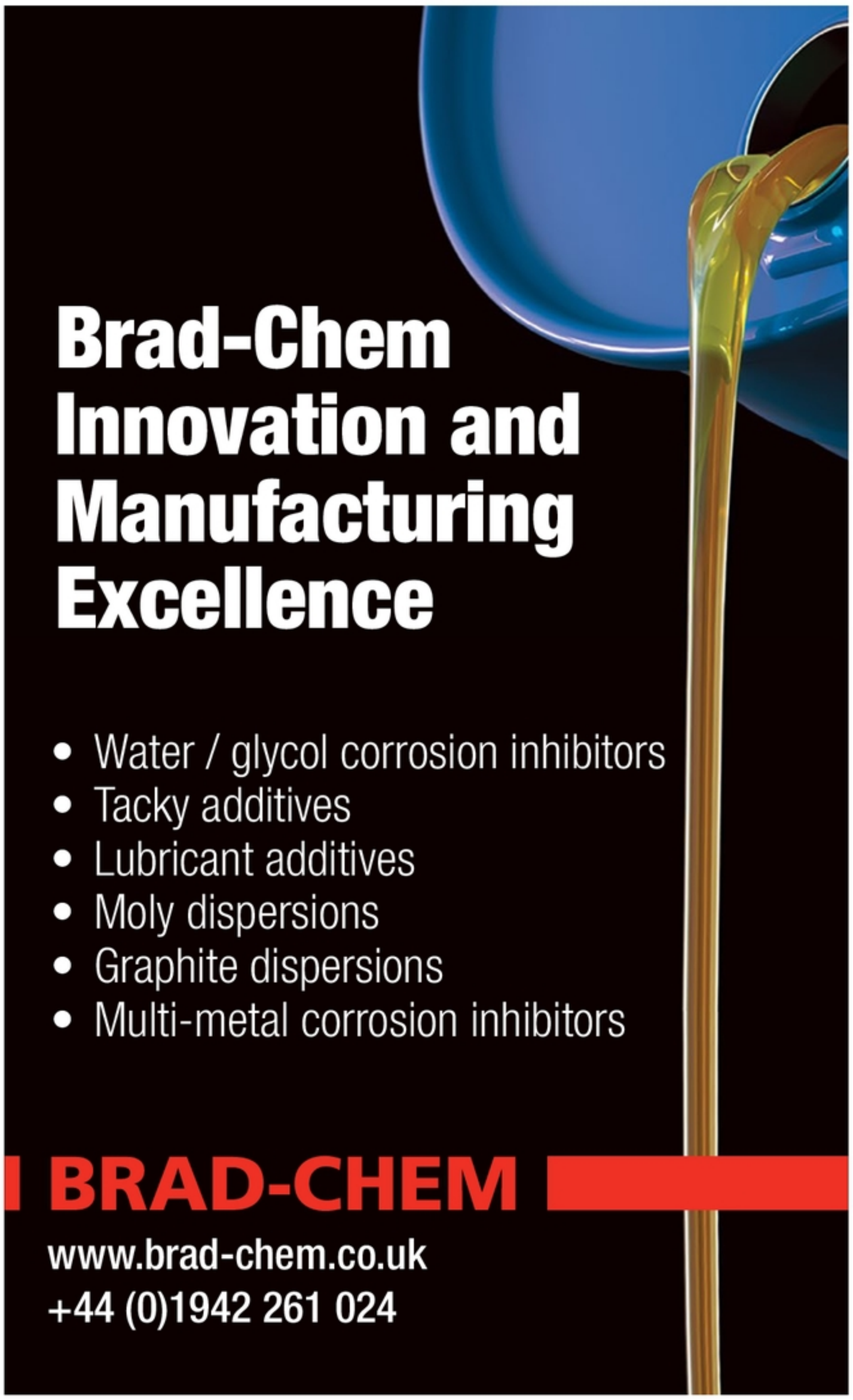
Afton studied six different antioxidants in combination with a multipurpose additive package containing zinc, phosphorus and sulfur. The study was conducted on an ISO 220 PAO blend with each sample aged using modified S-200 aging conditions as described in ASTM D2893-04.

Three samples showed the most beneficial impact, exhibiting low levels of volatility, sludge formation, viscosity increase and carbonyl increase. "It is interesting to note that using the high level of each antioxidant had a negative impact in each performance area," Kaperick said. Also, the additive package alone was actually more beneficial in many areas, lagging only slightly in viscosity increase and oxidation measurements against the best candidates. "This is likely due to the secondary antioxidant benefits of ZDDP antiwear components in that package."

The overall benefit of using additives was seen by comparing these results with a sample that contained no additives. "This sample generated large amounts of sludge as well as high viscosity and carbonyl increases," said Kaperick.

One other point for discussion is the elemental analysis of the oils at the end of test. "A little over one-half of the elemental concentration of sulfur and nitrogen remained in the oil at the end of the test," he reported. It is assumed that this is the result of some sludge formation, causing components containing these elements to drop out of the oil and not be measured at the end of the test.

"However, this effect is much greater with zinc and phosphorus, as about 10 percent of the phosphorus and almost none of the zinc were present at the end of the test," Kaperick related. "This would seem to indicate that under these conditions, almost all of the ZDDP has degraded and is no longer present in the oil." This result raises additional questions about whether this also happens in a synthetic grease formulation running in a bearing test at 160 degrees C. "Additional study is needed to determine whether ZDDP still helps maintain antiwear and oxidation protection under these conditions," Kaperick concluded. □



AFRICA'S LUBRICANT SUPPLY NETWORK

CHALLENGES AND OPPORTUNITIES

BY EMEKA UMEJEI

Poor road conditions, especially in the rainy season, are a severe barrier to intra-African trade.

"Most ports in the region currently operate near capacity and experience delays due to poor integration with other transport modes and slow clearance processes."
SAMER AKRAM

According to World Bank data, sub-Saharan Africa's growth increased moderately in 2014 to 4.5 percent, compared with 4.2 percent in 2013. The organization predicted that regional gross domestic product growth will remain broadly unchanged at 4.6 percent in 2015. Looking farther ahead, the bank reported, "Despite headwinds, growth is projected to pick up to 5.1 percent by 2017, lifted by infrastructure investment, increased agriculture production and buoyant services."

Despite this positive outlook on sub-Saharan Africa, lack of infrastructure remains a daunting challenge to intra-African trade. The World Bank report, "Defragmenting Africa," emphasized that the African market remains highly fragmented, resulting in the loss of enormous opportunities to exploit cross-border trade and, in turn, to generate new jobs. "Effective regional integration is more than simply removing tariffs – it is also about addressing the barriers that undermine the daily operations of ordinary producers and traders of both goods and services," the report stated.

Logistical Challenges

In a presentation at the Argus Africa Base Oils & Lubricants Conference in Johannesburg in June, Samer Akram, director-operations for Unichem South Africa, noted, "Intra-Africa trade is a mere 7 percent, compared to intra-European trade at more than 70 percent and intra-Asian trade, which exceeds 55 percent." The United Nations Conference on Trade and Development (UNCTAD) report, "Intra-African Trade: Unlocking private sector dynamism," noted a significant decline in intra-African trade. "The share of intra-African trade … rose from 19.3 percent in 1995, reached a peak of 22.4 percent in 1997 and fell to 11.3 percent in 2011."

The report noted that "African trade with the rest of the world grew much faster than intra-African trade. Over the period from 1996 to 2011, intra-African trade grew annually by 8.2 percent while trade with the rest of the world grew by 12 percent."

Of the 55 countries on the African continent, only 38 have ports; the remaining are either landlocked or have problems with accessibility. "Landlocked countries in the region require efficient transport links to enjoy competitive prices for landed goods and exports," said Akram. "Most ports in the region currently operate near capacity and experience delays due to poor integration with other transport modes and slow clearance processes."

Akram identified the main transshipment points for regional traffic as Abidjan, Cote d'Ivoire; Dar es Salaam, Tanzania; Djibouti; Durban, South Africa; and Mombasa, Kenya. However, he said, "They are not major hubs on the main international itineraries." He added that container transport in sub-Saharan Africa has an average annual growth rate of 7.2 percent – with a 13.8 percent growth in West Africa – but it is still far less than 1 percent of total global container traffic.

Challenges

At the Argus conference, Kamil de Villiers, lubricants manager, international business development for Engen, identified three critical