Latest advances in Lubricant and Grease Instrumentation for Oxidation Testing:

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Overview

Oxidation of oil occurs when there is a presence of oxygen (air) and heat. Atmospheric oxygen reacts with hydrocarbons which then form carboxylic acids. Over time, the concentration may rise and ultimately lead to corrosion of the machine to which the lubricant is applied. Thus, the lubricant would be doing more harm than good. antioxidant additives are included in formulations to protect the lubricants. Furthermore, the oil must be kept clean, dry, and as cool as possible in order to manage oxidation rates. To test this resistance of lubricating greases to oxidation, the testing method ASTM D942 is often utilized. A test using this method cannot predict whether grease will remain stable under dynamic service conditions, whether grease will remain stable when stored in containers for long periods, or whether films of grease will remain stable on bearings and motor parts. It should not be used to estimate grease types' relative oxidation resistance. This poster will discuss the developments in oxidation testing of greases and how these newly developed methods have been utilized in a recent study.

Recently Developing Test Methods

ASTM D942 is a standard test method which was first introduced in 1947 and has numerous characteristic limitations despite being a relatively simple assessment method. For instance, it is not suitable for comparing the relative oxidation stability of different greases, very labor-intensive and time-consuming requiring large sample sizes.

Fourier Transform Infrared (FTIR) analysis equipped with Attenuated Total Reflectance (ATR): This provides in-depth information on grease oxidation allowing comparison of oxidation stability of various greases.

ASTM D6186: Pressure Differential Scanning Calorimetry (PDSC): Consists of a TA 2920 thermal analyzer, a 2920 CE PDSC cell, an oxygen cylinder, a flow meter and a computer for data recording and further processing. Major companies use this apparatus to analyze how lubricating oils and greases resist oxidation. In these tests, an additional challenge is obtaining a smooth film on the sample in the test pan. This approach may not be adopted globally due to its cost and lack of availability to grease manufacturing companies.

ASTM D5483: The time it takes for oxidation to begin can be used to determine oxidation stability. This test method applies to lubricating greases exposed to oxygen at a pressure of 3.5 MPa500 psig and temperatures of 155 to 210°C.

ASTM D4742: This test method is used to determine the oxidation stability of lubricating base oils containing additives in the presence of chemistries that are similar to those present in gasoline engine service. Engine oils with viscosities ranging from 4 cSt to 21 cSt at 100°C, including re-refined oils, can be tested using this approach.

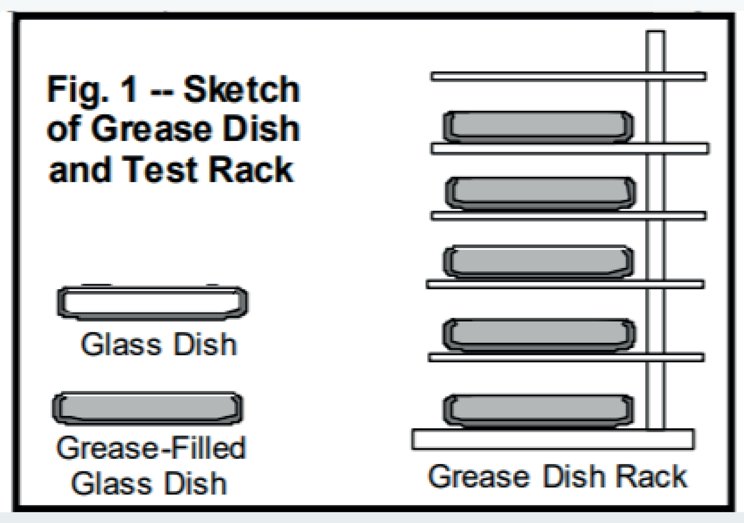
Oxidation Stability Apparatus adhering to ASTMD942

Oxidation of Lithium 12-hydroxystearate soap after (a) 0 hours (b) 36 hours (c) 72 hours

Comparative Study of Grease Antioxidative Performance

Oxidation can greatly diminish the tribological properties of all greases. As such, antioxidants are incorporated as an additive to the base-oil of greases to further increase oxidative stability. Once the efficacy of the antioxidants are depleted after a certain amount of time, the soap thickener in the base oil degrades and alters the physiochemical properties of the grease. Enhanced lubricant and grease instrumentation for oxidation testing enables the identification of optimal antioxidant additives for environmentally friendly lubricants and greases. The base oils which are derived from vegetable oils, such as castor oil, or even waste cooking oils often have an increased number of unsaturated bonding sites prone to oxidation. These specific subsection of lubricants are thus especially hindered in their poor low temperature performance and detrimental oxidation stability. A particular study done by Selby, Theodore W., et al. analyzed grease oxidation through the testing method ASTMD942. The results and testing method are highlighted in this poster.

A sample ASTM D942 testing method is shown below: This test necessitates the exposure of 20 grams of grease in five glass dishes, each with an exposed surface area of roughly 25 cm2 and 125 cm2 per test. As indicated in Fig. 1, the dishes are stacked with a space of roughly 5 mm between them.



The grease-filled stack is then placed in a cylindrical pressure chamber and subjected to oxygen with a purity of at least 99.5 percent at an initial pressure of 100 pounds per square inch (PSI = 690 kPa) at ambient temperature, which is then increased to 99 0.5°C. The oxygen pressure is gradually reduced to maintain a maximum of 1102 PSI at this elevated temperature.

The test is usually run for 100 or 200 hours, and the consequent drop in oxygen pressure as a result of grease oxidation is considered the test result.

Results of ASTM D942 Tests of Five Greases

ASTM D942 grease oxidation tests were run for 100 hours and additionally, in some cases, 200 hours. For each test, pressure change with time was continuously recorded. This is shown in figures 2, 3, 4, 5, and 6.

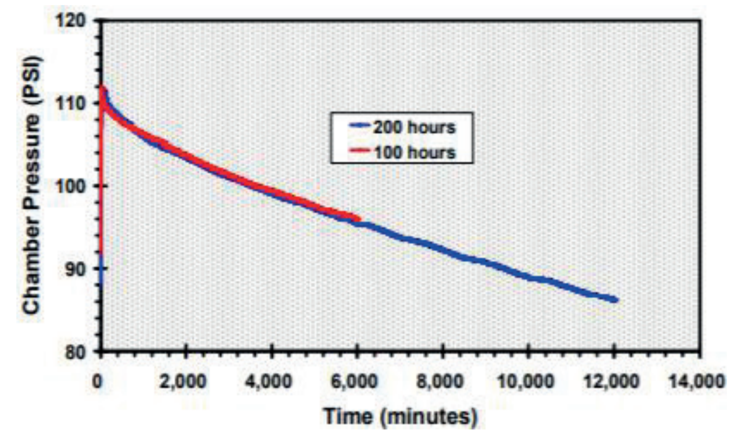


Fig. 2: Response of Grease Sample #1 to ASTM D942 Oxidation Test

The oxidation curves are notably comparable, indicating that ASTM Method D942 is capable of generating reliable outcomes. At 100 hours, the oxygen pressure in Test 1 was 96 PSI. It was 86 PSI after 200 hours of testing.

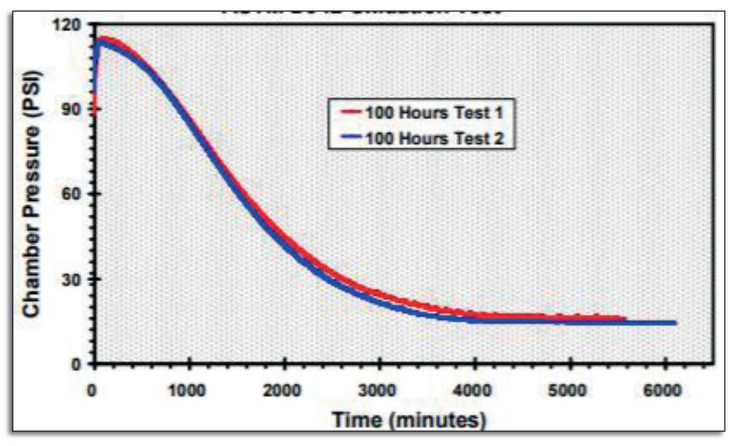


Fig. 3: Response of Grease Sample #2 to ASTM D942 Oxidation Test

ASTM D942 elicited a significant yet highly repeatable reaction. Even before the chamber temperature reached its maximum of 99°C, the grease may have began to oxidize. The final pressures in the chamber, which dropped to atmospheric pressures of roughly 15 PSI during the test, revealed entire oxygen uptake during the test.

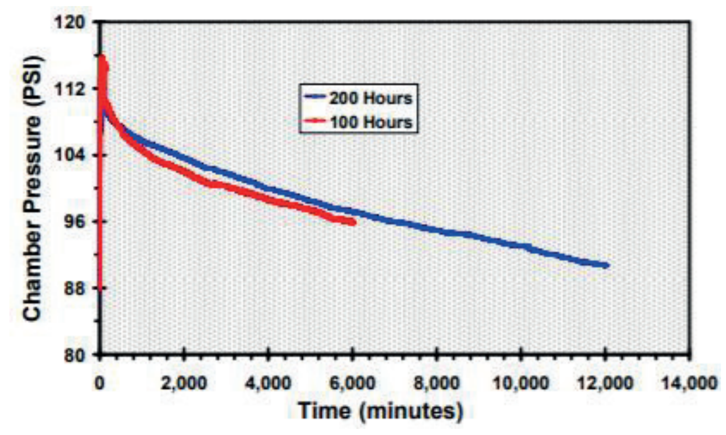


Fig. 4: Response of Grease Sample #3 to ASTM D942 Oxidation Test

This figure depicts the effects of failing to relieve the oxygen pressure in Test 1 for about 212 hours. Additional grease oxidation may occur with such preliminarily higher oxygen pressure, according to the findings. After 100 hours, Test 1 had a final oxygen pressure of 96 PSI, whereas Test 2 had a final oxygen pressure of 91 PSI after 200 hours.

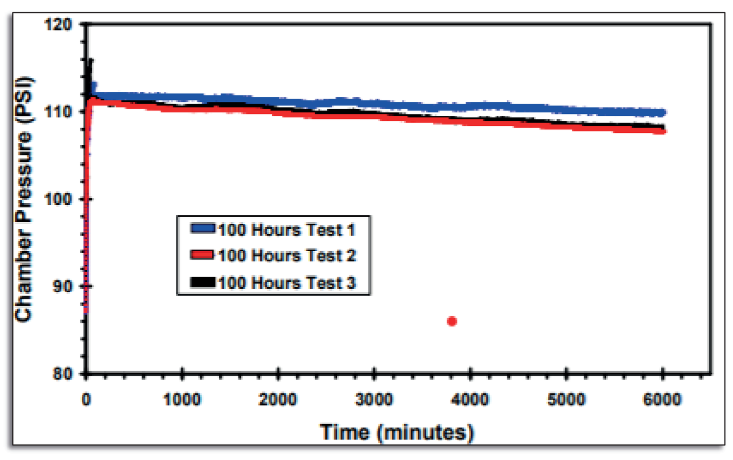


Fig. 5: Response of Grease Sample #4 to ASTM D942 Oxidation Test

The three replicate tests in the figure show that Grease #4 is highly resistant to oxidation in the D942 test. The lowest oxygen pressure after a 100-hour test was merely 107 PSI in these three experiments.

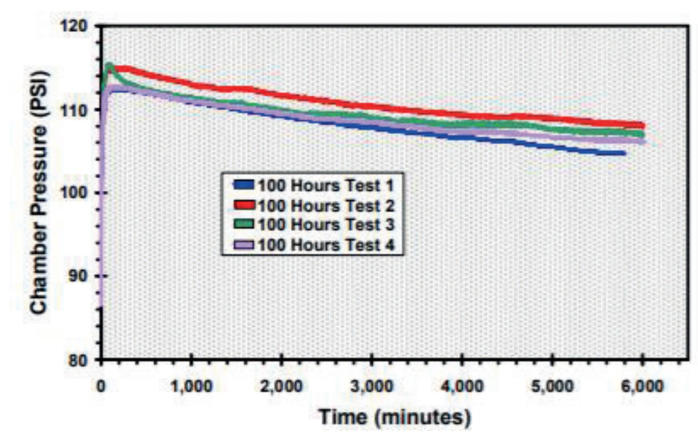


Fig.6: Response of Grease Sample #5 to ASTM D942 Oxidation Test

Grease #5, as illustrated in Figure 6, was likewise fairly resistant to oxidation after 100 hours of testing.

Conclusion

There have been many developments in oxidation testing of lubricating greases. Further improvements in the way tribological researchers analyze the physiochemical effects of oxidation on certain greases will allow for the identification of optimal antioxidant additives. A versatile instrument which can simulate industry-specific circumstances is therefore preferred, such as the Koehler Instrument Oxidation Stability Test Apparatus for Lubricating Greases conforming to the D942 standard testing method, along with IP 142, DIN 51808, and FTM 791-3453. It is an efficient machine in testing for oxidation stability of lubricating greases containing an oxidation bath, oxidation pressure vessel, pressure measurement and recording equipment, and Oxidata Pressure Measurement System. The key towards preventing the proliferated oxidation of greases relies on the success of oxidation stability testing, and the characterization of mechanisms leading to the phenomenon of oxidation.

References

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3. Azad, Samina, and Jonathan C. Evans. *An Advanced Technique for Grease Oxidation Measurement*. NLGI, 16 Dec. 2015, https://www.mtb.es/files/products/NLGI_Quantum_Grease_D942.pdf.
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5. Selby, Theodore W., et al. "A Comparative Study of Grease Oxidation Using an Advanced Bench Test Technique." *Tech. Akad. Esslingen, Ostfildern, Germany* (2014).