TRITON TECHNOLOGY

Application Note

Dielectric Thermal Analysis of Lubricating Fluid

Summary

The DS6000 DETA can be used to investigate liquids such as oils, waxes etc. The instrument comes complete with a comprehensive parts kit including a titanium cup and plate arrangement enabling the examination of such materials.
This Application Note illustrates that these materials can easily be characterised and examined to very low temperatures.

Introduction

Dielectric **Thermal** Analysis (DETA) is a powerful analytical tool for studying relaxation processes in materials or behaviour of polar species within a material. The glass transition (Tg) is a key process in any material, and can be observed with ease by DETA for many materials. This technique provides very revealing information about these relaxations through the tan δ vs temperature data. The form of the material can be anything from a thin film, sheet material, powder or a liquid.

Dielectric measurements are the electrical analogue of dynamic mechanical measurements. The mechanical stress is replaced by an alternating voltage across the sample (a.c.field) and the alternating strain becomes the stored charge (Q) in the sample. The sample in effect behaves as a simple capacitor. Q is always measured as its derivative dQ/dt = a.c.current.

The dielectric data is obtained from phase and amplitude measurements of current and voltage to resolve the components ε^* = Capacitance with sample/Capacitance with an identical air gap.

As in DMA, tan δ is the ratio of the loss factor(e″) to the storage component (e′ , dielectric constant or permitivity) . Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition. The size of this peak quantifies the amount of amorphous material present in the sample.

Experimental Conditions

Equipment

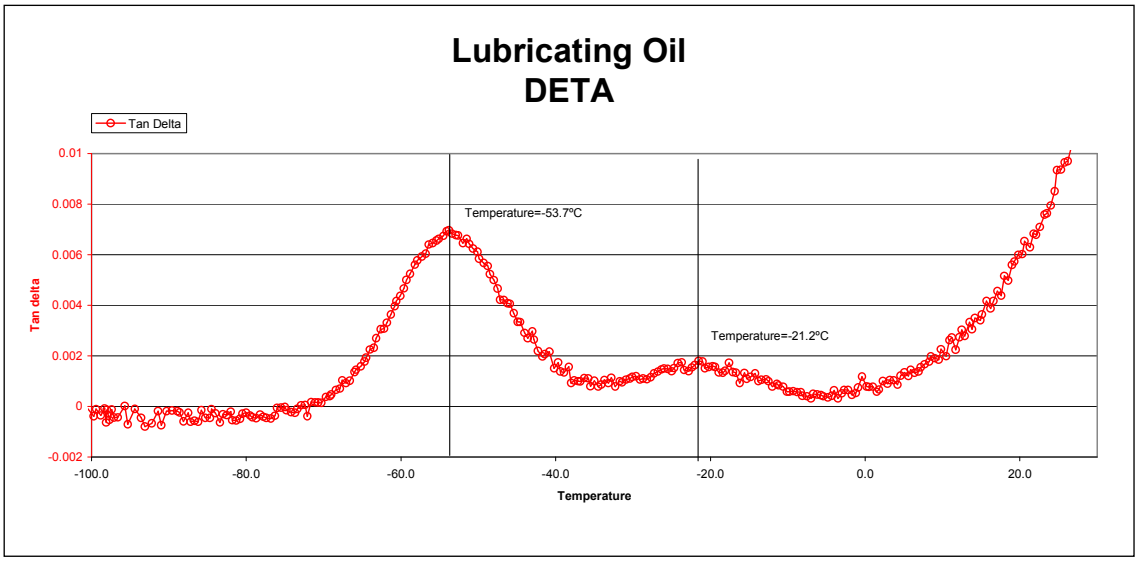
DS6000 DETA
1L 'Mini' Cryo

Sample:	Lubricating Oil
Geometry:	Cup and Plate 33mm diameter, thickness(gap) 0.3mm
Heating Rate:	3ºC/minute
Frequency	1 kHz

Experimental

Approximately 1ml of lubricating oil was placed in the cup and the top plate the fixed in place. A gap of 0.3mm was set using a micrometer and the sample was ready to run. The furnace was connected to the instrument as well as the 1 litre 'Mini' LN_2 cryo. The sample furnace was then cooled to approximately -100ºC using the fully charged 'Mini' cryo to the appropriate start temperature. The experiment was then started using the conditions indicated.

Result and Conclusion



The above Figure shows the result of the scan from -100ºC. This illustrates how very easy it is to fully characterise these types of material. This procedure could offer a very simple alternative quality control of the lubricating oil. The results can be directly correlated to the formulation used or the materials source.
In the above example, note that there appears to be two key fractions. The bulk of the material appears to relate to the process with a tanδ max at -53.7ºC , however, a significant fraction maximises at -21.2ºC.
As with most materials examined by this technique, conductive effects increase at higher temperatures and this is seen as we pass through room temperature.