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Design, Packaging and Characterization of a Langasite Monolithic Crystal Filter Viscometer

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Abstract: A two-port monolithic crystal filter (MCF) viscosity sensor has been developed using high coupling langasite material and advanced packaging techniques [1]. The present work details the evaluation of quartz and langasite MCF elements with varying shapes, contours and electrode designs. The study has resulted in a commercial design on 10.25 mm blanks, housed in a TO-8 header with support circuitry contained in a ½" NPT threaded bolt. *Copyright © 2007 IFSA.*

Keywords: Viscosity sensor, Monolithic crystal filter

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1. Introduction

There has been an unmet need for in situ viscosity sensing in numerous applications including condition based monitoring (e.g. lubricants) and process control (e.g. chemical manufacturing). To date no technology has completely satisfied the various requirements, resulting in fragmented markets with many unserved niches. Previous efforts to place mechanical instruments into this role have been costly and only partially successful. Conservation and economic considerations dictate longer service intervals whereas reliability considerations require constant monitoring to allow this. The present work demonstrates the design of a solid state sensor meeting the needs of *in situ* condition based monitoring of lubricants while overcoming end user reluctance to embrace new units of measure.

Previous efforts to apply acoustic wave technology to this requirement have met with varied obstacles. High frequency (160 MHz), shear horizontal (SH) acoustic plate mode (APM) sensors operate at shear rates of $10^5 – 10^7$ s^{-1} and time constants of 10^{-9} s [2]. While these parameters preclude good correlation with traditional measurement methods, the resulting sensors have addressed specific niches in the application space. Other offerings based on quartz crystal microbalances (QCM) ranging from 5 to 10 MHz exhibit shear rates from 10^3 to 10^4 s and 16 to 33×10^{-9} s time constants. These sensors were more capable of correlation to mineral oils for condition based monitoring (CBM) but ultimately faced limitations of metal corrosion/erosion, capacitive/conductive loading, and packaging, in addition to the well known dynamic range limitations of quartz. Low frequency tuning fork resonators have offered attractive performance in prototypes but also suffer the packaging constraints of the QCM sensors in addition to worsened shock and vibration issues as the resonant frequency approaches the vibration frequencies. SH surface acoustic wave (SAW) sensors combine the correlation problems of high frequency SH APM sensors and the packaging problems of the tuning fork and QCM.

Schweyer [3] presented a sensor geometry that (a) isolated the driven electrodes from the fluid, (b) allowed differential transmission measurements, (c) operated at moderately low frequencies, and (c) overcame the shunt capacitance limitations of traditional QCM sensors. The sensor was based on a two-port monolithic crystal filter (MCF) topology with sensing occurring on a passivated ground plane. The early quartz devices exhibited 15 dB of loss in air and 34 dB of loss in 64% glycerin solution. It was acknowledged that the thin aluminum electrodes offered inadequate energy trapping, but also surmised that quartz was inadequate.

Subsequent effort has compared plano-convex and plano-plano sensor elements and has compared AT quartz to Y+1.5° langasite. The work has also explored the electrode geometry and packaging requirements and has explored the role of packaging on sensor performance.

2. Results

A. Sensor Element Design Approach

A number of applications exist wherein the sensor would ideally be placed in the tip of a bolt ranging in size from 14 to 25 mm (e.g. M-14 automotive to ½” through 1” NPT). Initial effort sought to address the M-14 bolt with a custom package and an 8.9 mm (0.350”) crystal blank; however the present design has employed semi-standard TO-8 packages with a 10.25 mm (0.410”) diameter, 0.26 mm and .33 mm thick, crystal blank to expedite development. The design preference was biased towards quartz elements for cost considerations; however no viable quartz element was obtained, plano-plano or contoured, for use in oscillator based instrumentation.

The designs sought to allow subsequent miniaturization to a 9 mm blank while allowing a 1mm bond ring around the perimeter of the sensor. Designs were therefore sought that maximized the electrode area (minimized the crystal resistance) in a 7mm “free” region with a mechanically clamped region extending to the edges. Modeling suggested that the benefits of increased electrode area (crystal resistance under fluid load) far outweighed the detriments of mounting losses and initial designs were therefore developed to fit within a 5mm active diameter with at least a 1mm decay length to the mounting structure.

Initial candidates consisted of a rectangular electrode MCF having 1.5x3.5mm electrodes separated by a 0.5mm gap and a circular electrode MCF having 2.5 mm radius semicircles separated by 0.5 mm. The area per electrode was 5.25 mm^2 for the rectangular device and 9.8 mm^2 for the circular device.

Round 8.9 mm diameter 6.4 MHz quartz devices (0.26 mm thick) with X-alignment of rectangular electrodes using 490 nm Au electrodes exhibited 32dB of insertion loss. Aligning the devices 90° from X yielded 25 dB losses, while circular electrodes, having higher area and better input resistance, yielded

20 dB along X and 15dB at 90°. Increasing the gold to 790 nm offered no improvement, suggesting that energy trapping was as good as possible in contrast to the aluminum devices of Schweyer. Liquid loading these sensors to a viscosity-density product of 50 AV (acoustic viscosity units, 1 AV = 1 cP-g/cm^3 = 1 Pa-s-Kg/m^3) resulted in an insertion loss of nearly 50 dB. The target operating range is >200 AV (min) over temperature for oil CBM and a target range of >500 AV is desired.

Clearly a quartz plano-plano device is not feasible within the geometric constraints of the planned applications. The use of 5 MHz (0.33 mm) plano-plano blanks could only result in increased insertion loss in air since crystal resistance would increase as frequency squared for the same electrode area. The data did indicate that round electrodes were preferable since they maximized electrode area within the constraints of the geometry.

5 MHz contoured resonators with circular electrodes on 8.9 mm blanks were studied with contours of 0.5, 1.5, 2.5, 3.5 and 4.0 diopters. The plano-plano crystals exhibited nearly 40 dB of loss while contours of 2.5, 3.5 and 4 diopters exhibited 6-8 dB of loss. Data is shown in Fig. 1.

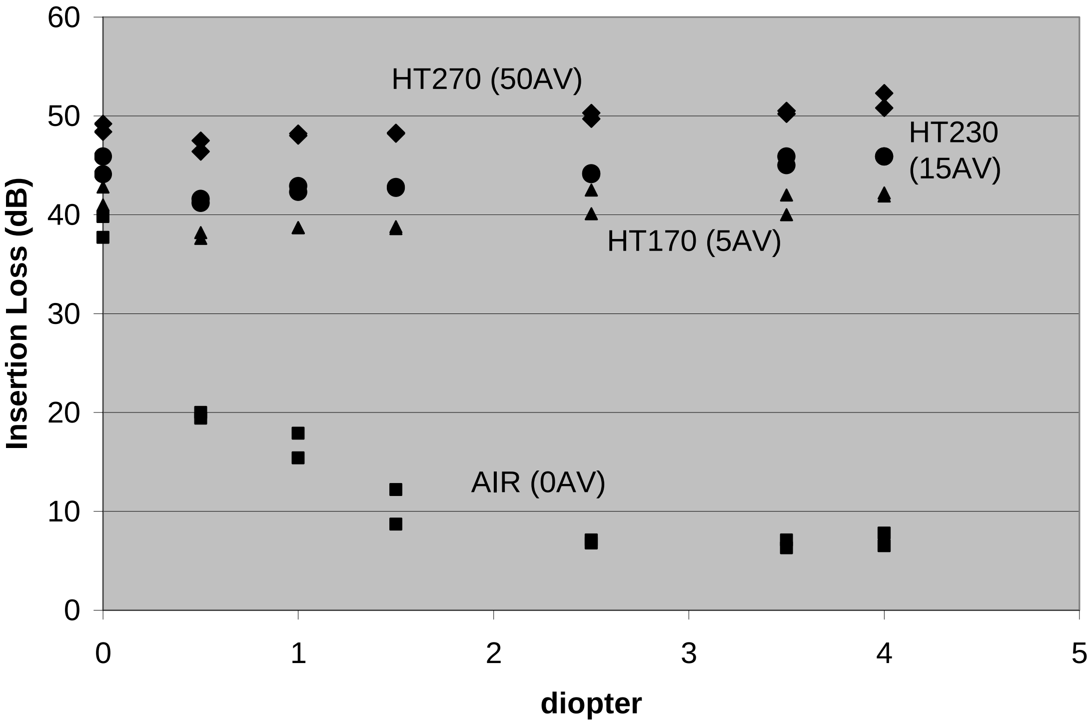

Fig 1. Insertion loss of 5 MHz quartz sensors in air and three low viscosity liquids vs. contour curvature shows unacceptable insertion loss.

While this data is initially encouraging, Fig. 1 also shows the insertion loss values for three perfluoropolyether calibration standards with 5, 15, and 50 AV nominal acoustic viscosity. The loss in these relatively low-viscosity liquids increases with increasing contour. It is clear that even contoured quartz MCF sensors are incapable of operating to the required viscosity range. However, a 2.5 diopter contoured quartz resonator could offer a very sensitive device in the 0-5 AV range, e.g. addressing fuel grade or contamination monitoring applications. Nonetheless, in terms of the present requirement, it is determined that quartz sensors are incapable of addressing the size and viscosity constraints.

The development of a langasite sensor followed the same evaluation of rectangular and circular electrodes aligned along X and at 90°, initially on 8.9 mm diameter 5.3 MHz (0.26 mm) plano-plano blanks. The unpackaged insertion loss was between 2.7 and 4 dB with minimal dependence on metal thickness, alignment or style of electrode. Whereas the best quartz sensor elements exhibited >35 dB with only 5 AV of liquid loading, langasite sensor elements exhibited <30 dB in a 636 AV mineral oil (Cannon N350) with <20dB in the 50 AV (HT270 perfluoropolyether). Subsequent effort determined that a 4.5mm diameter electrode offered the best compromise between viscosity range and packaging effects. Prototypes were developed with 10.25mm blanks at 5.3 MHz to simplify development of the packaging process.

B. Sensor Element Packaging Approach

The sensor concept is ultimately to employ a hermetic seal of the sensor ground plane to the TO-8 lid. Existing prototypes have all employed a wrapped ground electrode to the driven electrode face with epoxy die attach. A ground wrap (conductive polyimide or evaporated CrAu) brings the ground plane electrical contact to the front face of the crystal. The crystal is affixed to a machined TO-8 lid (kovar+Ni+Au) with a defined aperture of 7.25, 7.9, or 9 mm. The four lead TO-8 package was modified to have springs extending the leads.

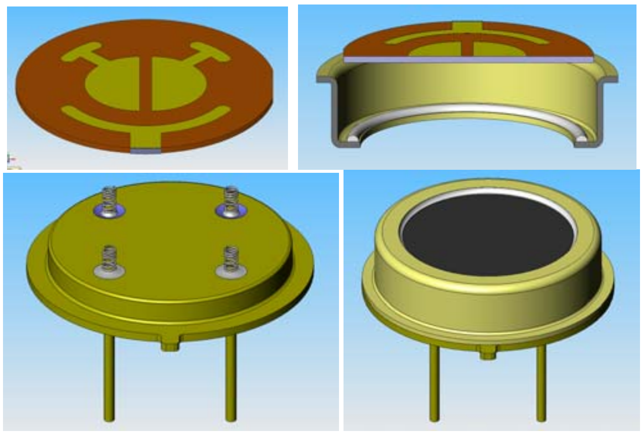

Fig 2. Packaging of the langasite MCF sensor starts with a polyimide wrap of the ground contact (top left) followed by epoxy die attach (top right). The package is affixed with springs (bottom left) that connect to the sensors pads via conductive epoxy. The sensor element is vacuum baked and then weld sealed (bottom right).

Presently the unpackaged parts exhibit <2dB of insertion loss in a probe fixture, <2.5dB with the polyimide wrap, <3.5dB after die attach, and an average of 4.5 dB after attaching the leads to the electrodes. Reducing the lead attachment losses is an active project.

C. Sensor Surface Materials

Devices employed CrAuCr sandwiches with the capping chromium layer inset to allow good electrical contact of the ground plane. An ongoing project, to be reported in the future, is addressing high reliability sensor coatings. The ultimate sensor will offer abrasion and chemical resistance with excellent thermal shock and cycle resistance. The package reliability is enhanced by the excellent match of thermal expansion between the kovar and langasite. The sensing films being developed also offer good thermal expansion match to this system.

D. Sensor Circuitry

The sensor instrumentation is oscillator based using a common emitter amplifier with active bias control. The input impedance of the feedback amplifier is typically 400 Ω, reducing the effective insertion loss of the sensor element from that measured in 50 Ω. This yields some possibility of attaining the 500 AV target despite a 30 dB loss compared to 18 dB of amplifier gain in 50 Ω.

The radio frequency (RF) signal levels are measured at the input and output of the MCF using two temperature compensated diode detector circuits [4], allowing an approximate measurement of the insertion loss. The ability to operate diode detectors with minimal temperature sensitivity over the entire operating temperature is critical to the operation of the sensor system.

In addition to the RF level measurements and bias control circuitry, the circuitry also incorporates either a Pt resistive temperature device (RTD) or an integrated circuit thermometer. The probe obtains +5V@30mA and a bias control signal from the control module and returns three analog voltages representing temperature (VT), the sensor drive signal level (VDH), and the sensor output signal level (VDL).

One format mimics the form and function of the SH APM sensor reported elsewhere. In this format, these signals are monitored by a control module on a second PCB, removed from the extreme thermal environment of the fluid under test. The diode voltages are interpreted as power levels and employed to approximate insertion loss of the sensor element. The insertion loss is compared to tabulated data for the probe in air and the difference is used to compute the viscosity density product based on previously measured calibration data.

Of more interest are the complete bolt format sensors. The standard TO-8 sensor element cannot fit inside an M-14 bolt and therefore resides outside the bolt but fits through the receiving threaded hole (not shown) and further miniaturization will be required for a practical M-14 version. Fig. 3 shows the commercial rectangular format while Fig. 4 shows the commercial in-line threaded bolt format. In the ½" NPT version of Fig. 4 the sensor element is completely within the bolt.

The long term goal of this development is an integrated system in which the sensor intelligence is also incorporated directly into the sensor housing. Prototypes have been developed in which the RF circuit is integrated with an analog to digital converted (ADC) and microprocessor with controller area network (CAN) data communications. Current effort is addressing the development of a CAN Open compliant interface.

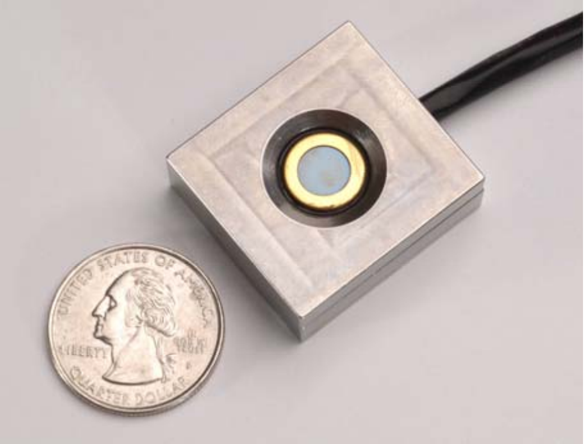

Fig 3. Rectangular format sensors allow direct replacement for an existing commercial SH APM sensor (left) while development continues on miniaturization and integration to a bolt format (right).

Fig 4. Vectron solid-state ViSmart™ sensor.

E. Sensor Performance

While a number of sensor, system and calibration/analysis improvements are ongoing, it is possible to evaluate the performance of the sensors. Fig. 5 shows data for the ½" bolt format sensor measuring 5W30 motor oil with various levels of diesel fuel dilution. Addition of diesel fuel slightly lowers the density and also lowers the viscosity. Sensor to sensor tracking is good and it is possible to readily discern the change in acoustic viscosity vs. temperature for 3% fuel dilution.

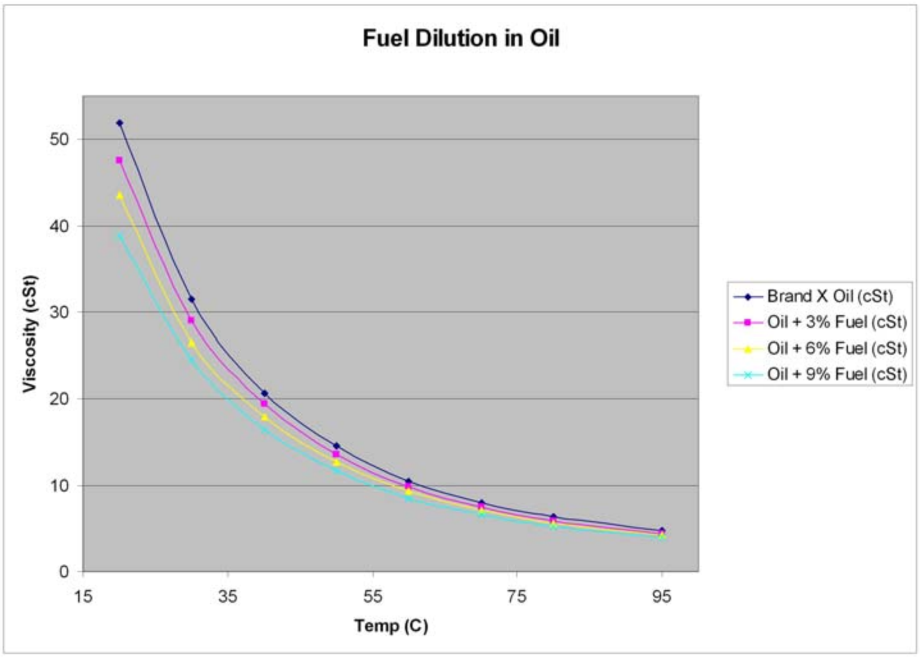

Fig 5. Tracking of percentage fuel dilution levels in viscosity of oil.

While oil condition based monitoring can be reliably performed by evaluating only the changes in reading of an arbitrary scale of measure, end users desire a sensor that reports in the units to which they are accustomed and with a direct correlation to OEM data sheets for the lubricants. This is troublesome in that acoustic wave sensors inherently measure a so-called acoustic viscosity (AV) as the product of viscosity and density whereas the preferred units in lubrication are the ratio of viscosity to density, called centistokes or mm^2/s. There exists a factor of ρ^2 corresponding to a typical error of 20 % or more.

This is compounded by the relatively high shear rates of the acoustic method, typically on the order of 10^2-10^4 for acoustic wave sensors based on the thickness shear mode. A further impediment to correlation exists due to Maxwellian effects of harmonic motion. These effects are illustrated and progress in obtaining a simple, solid state sensor that reports compensated viscosity data is detailed.

Rheometer data on perfluoropolyether fluids is shown in Fig. 6. The liquids exhibit no significant dependence on shear rate with the exception of the 100°C data. It is believed that turbulent flow characteristics within the rheometer distort the 100°C curves. Because of their excellent stability over time, temperature and shear rate, these fluids were chosen as calibration standards. Insertion loss measurements are taken in air, HT-200, HT-230, and HT-270 over temperature. Data is then curve fit such that the rheometer viscosity-density product is fit to a power law of the insertion loss increase from air.

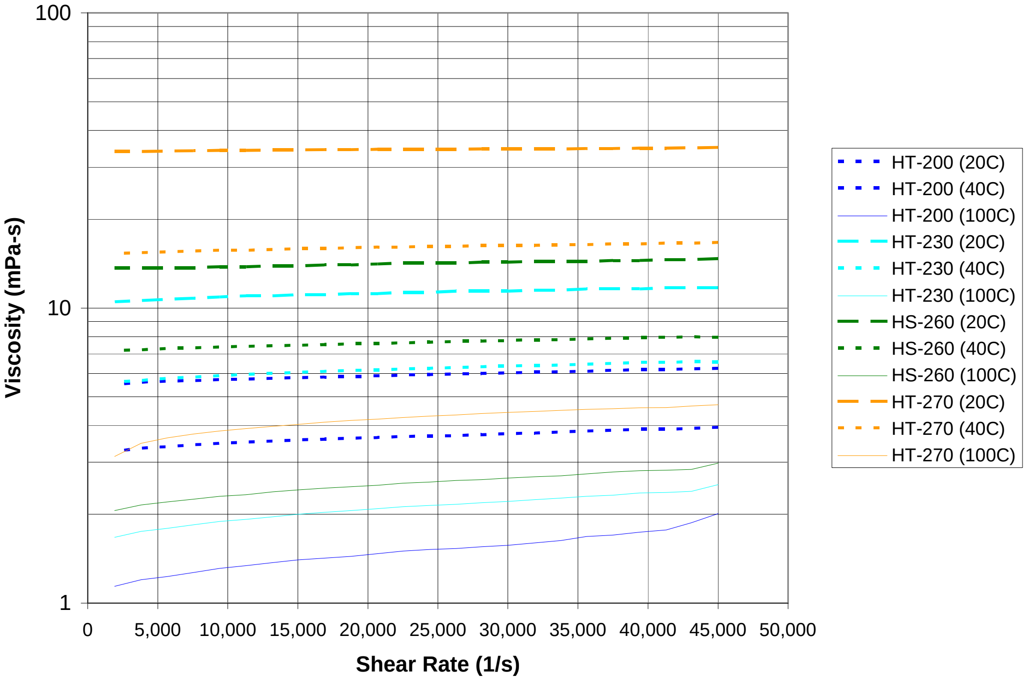

Fig 6. Perfluoropolyether viscosity vs. shear rate at 20°C and 40°C is very stable while at 100°C the low viscosity and high shear rate result in turbulent flow.

Fig. 7 indicates residual nonlinearity by measuring comparable standards and a fourth fluid of similar chemistry (HS-260). The curves are taken at 50 s^{-1} shear rate over temperature in a Couette cup rotating rheometer while the sensor was fit to capillary rheometer data. This nonlinearity has subsequently been identified and work is underway to increase the linearity.

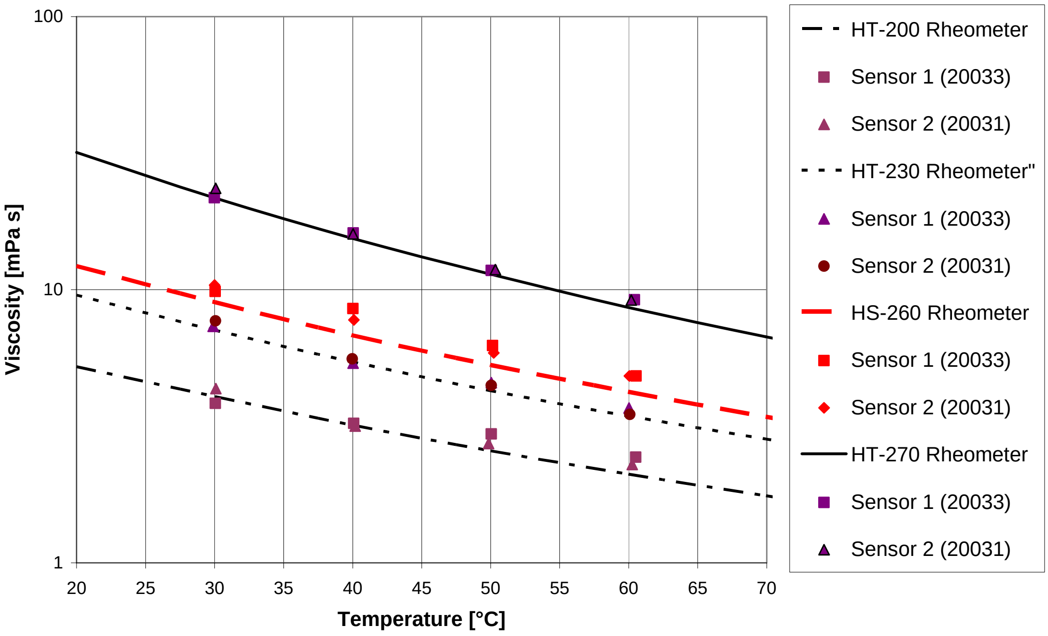

Fig 7. Two calibrated sensors reproduce the standards but show residual error to a fourth material.

Fig. 8 shows the correlation of the two sensor readings (AV = mPa-s * g/cm^3) to rheometer data. Sensor and rheometer data are paired within ±1°C, accounting for some of the spread. The aggregate data over a set of 9-10 samples representing both fresh and used oils are given. The linear fits were forced to intercept at zero and the slope represents a factor that includes the density but also a viscoelastic term,

$$AV \big/ \rho\eta_o = 1 \big/ (1 + 0.033\tau)^2 ,$$

where ρ is the density, η_o is the rheometer value (mPa-s), 0.033 is the sensor frequency in gigaradians/s, and τ is the viscoelastic relaxation time in ns. The linearity of the correlation indicates that viscoelasticity and density alone can account for the offset in lightly modified mineral oils on a class-by-class basis. Further work on lubricants with high levels of additives is ongoing.

3. Conclusions and Future Work

The data presented clearly indicates that 9-10mm diameter MCF sensors operate over viscosity ranges in excess of ~10 mPa-s (cP). Plano-plano langasite sensors operate to well in excess of 100 AV (mPa-s-kg/m^3) and appear to be capable of operation to as high as 500 AV.

The optimization of the lead attach placement and the die attach radius (package aperture) along with the use of improved die attach will reduce the magnitude and variability of packaging losses.

Further work is ongoing in developing a hard, smooth, chemically inert and thermally stable sensor coating. The existing CrAuCr parts are remarkably stable, however better systems are in development. Finally, there will always be fluid-specific differences between the viscosity reported by traditional laboratory techniques and that of a resonant sensor; hence, fluid-specific correlation curves are being created for quantitative analysis.

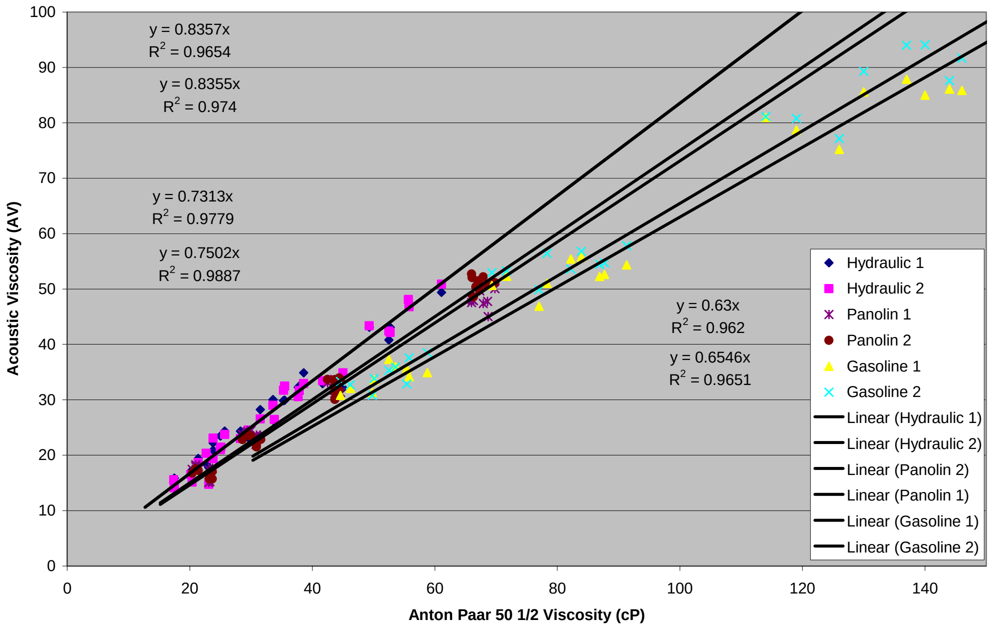

Fig 8. Composite correlation of two sensors to rheometer for nine fresh/used samples of three fluids.

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Guide for Contributors

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