New Capacitive Thermal Age Tag for Predicting Remaining Thermal Life of Multiple Products

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ABSTRACT

This project will demonstrate that a thermal age tag, incorporating two capacitive sensors of different activation energy, can be used to determine the effective temperature (T_{eff}) of complex thermal environments to predict the condition of a wide range of thermally degradable products and materials. Correlation of the thermal age of the tag at the monitored product's degradation activation energy provides estimated remaining thermal life of the product. The thermal age tag requires no batteries or electronic memory required in data-logging approaches, resulting in reduced size, weight and cost resulting in a totally passive tag. These passive tags are potentially maintenance free for the life of the product. The capacitive thermal age (CTA) sensors incorporated in the tag consist of a polymeric dielectric between two conductive plates to create a tiny capacitor. Capacitance of the sensor increases during thermal aging due to shrinkage of the polymer. Additives such as catalysts are used to adjust the activation energy (Ea) of the capacitance change~~s~~ with thermal age. By incorporating two CTA sensors of different activation energies in the tag the effective temperature of a complex thermal environment can be determined at any (or multiple) target activation energies for products or materials. This paper describes the development of a universal thermal age (UTA) tag incorporating capacitive thermal age sensors~~-~~and presents preliminary co-aging trial~~s~~ data with a variety of selected polymeric products to demonstrate feasibility of this approach.

INTRODUCTION

Identifying when a product will fail in a real-world environment is a complex and daunting task. Environmental conditions including; temperature, humidity, chemicals, atmospheric pressure, and radiation may affect materials and components during transport, storage and operations. This may significantly decrease expected product life. Knowledge of the current properties of these products without the need to perform difficult and/or destructive testing, as well as the prediction of remaining life under known or assumed thermal environments is a valuable tool in many industries from pharmaceutical to defense applications. Use of Arrhenius methodology for prediction of target product condition in variable thermal environments is a common approach. This approach requires monitoring of the product thermal environment throughout product life, and traditionally is done with thermal data logging.

Substantial progress in prognostic methodology has vastly improved prognostic health monitoring (PHM) capabilities in regards to finding the remaining useful life (RUL) of products. (Vichare et al., 2006) New types of sensors, computation models and algorithms, as well as destructive and non-destructive testing capabilities have vastly improved PHM. Unfortunately, the sheer magnitude of comprehensive monitoring remains impractical for many products and materials. Shelf-life dating remains one of the most common PHM methods for determining the RUL of products and materials is a testament to this. Long term accelerated aging studies are still very normal for determining expected life of materials but no matter how many studies are conducted this does not simulate the true use environment in most cases. Many items and products in the field cannot be monitored for actual thermal aging and are returned and refurbished before the end of their lifetime based on accelerated aging studies that are usually more extreme than actual conditions.

Even single-environmental stressor monitoring of products such as thermal history can become complex when monitoring large numbers of individual products. In the cases of individual food or pharmaceutical products, or for products with long lifetimes such as munitions, propellants, and explosives the complexity makes producers continue to rely on highly accelerated aging data and assumed environments. In order to monitor actual environmental conditions, data loggers require batteries and/or energy harvesting means with incumbent battery life and experience replacement issues. Data storage and handling issues including memory size, sample rates, data storage location and retrieval become cumbersome and make this approach impractical.

Measuring thermal exposure provides a relatively simple and effective means to adjust RUL based on integrated time and temperature. For example, "smart tags" incorporating thermal data-loggers are small enough to monitor many individual products. The thermal exposure sensed by the tag is incorporated into kinetic models to project remaining thermal life based on thermal age testing of the product or material being monitored (Roduit et al., 2019).

A passive thermal age sensor significantly simplifies product thermal life monitoring since data logging and energy needs disappear. A handheld reader provides the electrical power required to read the tag sensor and provide a present indication of remaining thermal life of the product. A pass/fail reader such as a simple red/green light is easily used in the field.

A resistive thermal age (TA) sensor and method to project remaining thermal life (Watkins, 2018) discloses such a passive monitoring tag. The resistive thermal age sensor comprises a conductive composite sensor element consisting of a polymeric matrix and conductive particle filler. As the polymeric matrix shrinks during aging, due to crosslinking or other chemical or physical mechanisms, the resistance decreases. Selection of an appropriate matrix and filler provides a change in resistance with thermal exposure that follows Arrhenius behavior. The resistance of the TA sensor always represents the current integrated time-temperature of its environment at the sensor's characteristic activation energy. Correlation of sensor resistance to multi-temperature thermal aging data of target materials or products provides a means to project RUL.

The TA sensor is incorporated into a tag that is attached to, or thermally associated with a product or material that is to be monitored. The tag acts as a surrogate of the material from a thermal aging perspective. Tags incorporating passive TA sensors are smaller, lighter and reduce the initial and service life costs when compared to thermal data loggers in projecting RUL.

This paper describes a TA sensor which uses capacitance change in a thermal environment to predict remaining thermal life. This capacitive thermal age (CTA) sensor consists of a polymeric dielectric between conductive plates. As the dielectric shrinks from thermal aging, the plate separation decreases, thus increasing capacitance measured between the plates. As in the resistive thermal age sensor, the electrical property (capacitance in this case) always represents the integrated time-temperature of its environment at its characteristic activation energy. Arrhenius behavior of the capacitance in a variable thermal environment allows correlation modeling of RUL for materials and products for which thermal aging data is available.

Advantages of CTA sensors include the elimination of conductive fillers in the process. Addition of conductive particles in the resistive thermal age (RTA) sensor affects the chemical reactions which in turn affect matrix shrinkage. This shrinkage due to fillers therefore changes activation energy (Ea) of the sensor. Adjustment of the Ea of a capacitive TA sensor may use virgin polymers, or polymer/additive combinations can be chosen based on Ea and desired reaction rates alone. The conductive plates of a CTA sensor also provide good hermetic sealing, reducing sensitivity to humidity or corrosive gasses as compared to RTA sensors. And most importantly, CTA sensors appear to retain their aging sensitivity longer in thermal life than their RTA counterparts. Retention of aging sensitivity is important to provide reasonable resolution of aged properties near the end of life, especially in product having long thermal life.

Use of tags incorporating CTA sensors will be shown by analysis of the aging response of several CTA sensors, and correlation of CTA sensor capacitance with the condition of materials co-aged with the sensors. A method of predicting condition of multiple materials or products with a single tag comprising two CTA sensors will be demonstrated.

2. CAPACITIVE THERMAL AGE SENSORS

Figure 1 shows a prototype CTA sensor comprising a thermoplastic polyurethane (TPU) dielectric and copper top and bottom plates.

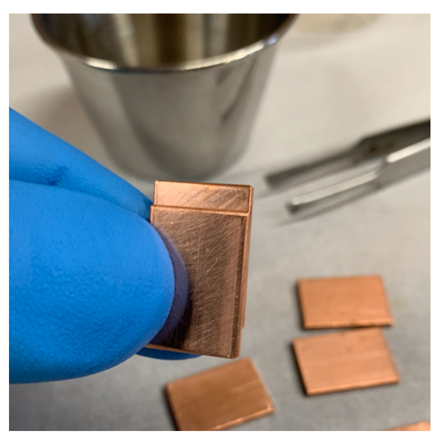

Figure 1. Prototype capacitive thermal age (CTA) sensor.

The capacitance of the sensor is shown in Eq. 1 by the following relationship:

$$C = k * \frac{A}{d} \tag{1}$$

Where:

C = capacitance, k = dielectric constant of the dielectric between the plates, A = surface area of the plates, and d = the spacing between the plates.

The capacitance of the sensor is directly proportional to changes in dielectric constant and surface area and inversely proportional to changes in the film thickness of the dielectric during aging. If the surface area of the dielectric between the plates is assumed to be constant, shrinkage of the dielectric decreases the thickness between the plates, increasing the capacitance. Changes in dielectric constant can be calculated by use of thickness and capacitance measurements during aging.

In order for CTA sensors to be of practical use in projecting target property condition from thermal aging, the sensors must show good Arrhenius behavior data derived from empirical multi-temperature aging data. The example below shows thermal aging data of a CTA tag comprising a commercial printed circuit board (PCB) as a bottom plate, a copper film top plate, and TPU resin as the dielectric. The use of a PCB as the bottom plate allows multiple capacitive sensors on a common tag platform. In Figure 2, two capacitive sensors are formed by the outside electrodes of the tag for the bottom of the sensor using a common ground top plate connector pad.

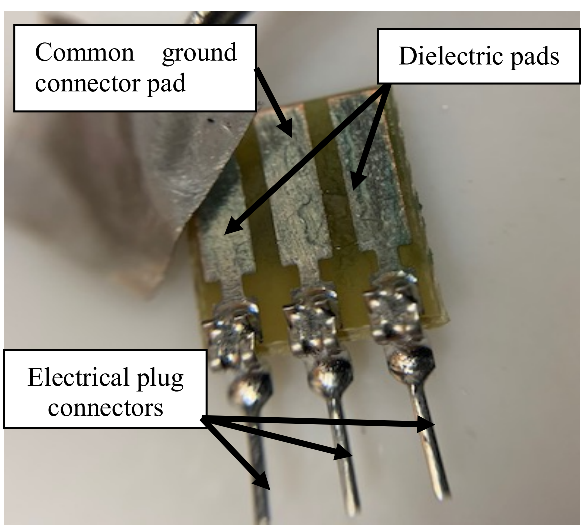

Figure 2. CTA tag bottom plate for two CTA sensors

A copper foil top plate covers the sensor (Figure 3). The top plate foil is electrically connected to the middle electrode of the PCB. Capacitance is easily measured as demonstrated in the image.

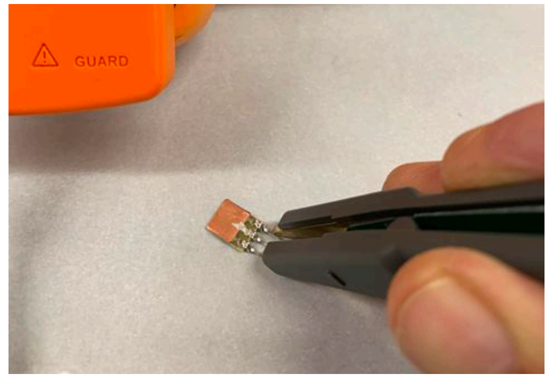

Figure 3. Measuring capacitance of a two-sensor CTA tag with a common copper top plate.

Figure 4 shows the correlation trial measuring capacitance of one of the thermal age sensors (C1A) of CTA Tag (C1) during thermal aging. Included are the error bars for each data point. The sensor/sensor variation is believed to be caused by the variability in the current prototype sensor manufacturing process and will be evaluated at later date to attempt to minimize the variability. The CTA tag, similar to that of

Figure 2, utilizes dielectric films comprising a TPU matrix (comprised of Estane 5703) between the PCB bottom plate and a copper film top plate. The C1A sensor utilizes a catalyst identified as CAT 2 (dibutyltin dilaurate), which provides a higher reaction rate of the sensor as compared to virgin polymeric dielectric. The catalyst also provides a means of adjusting the effective activation energy of the capacitance response to thermal aging of the sensor.

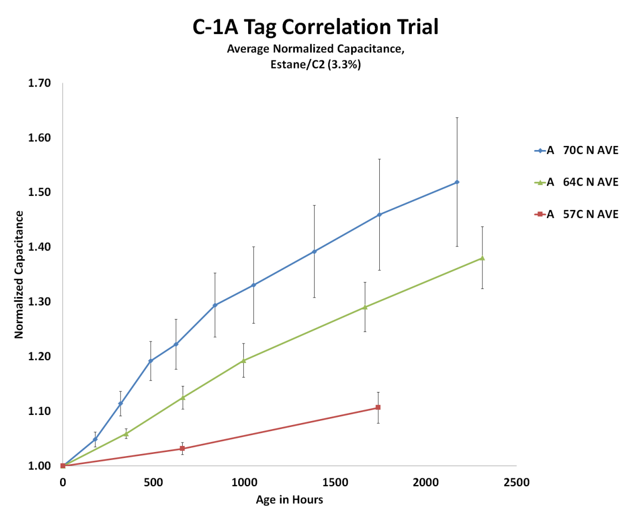

Figure 4. Normalized capacitance of C1A sensor of C1 tag vs. time at 70C, 64C and 57C aging. Note: error bars are +/- one standard deviation.

Time-Temperature-Superposition (TTS) disclosed by Gillen et al. (Gillen, K., Bernstein, R., & Celina, M., 2017) is used to determine the acceleration factor for each aging temperature as compared to a reference temperature. TTS is a preferred method of determining the acceleration factors

(AFs) since it includes all of the data instead of single point determination as is common in other methods. Figure 5 shows a TTS graph of the aging data with normalized capacitance used to determine the AFs for the C1A sensors for the TTS for each aging temperature.

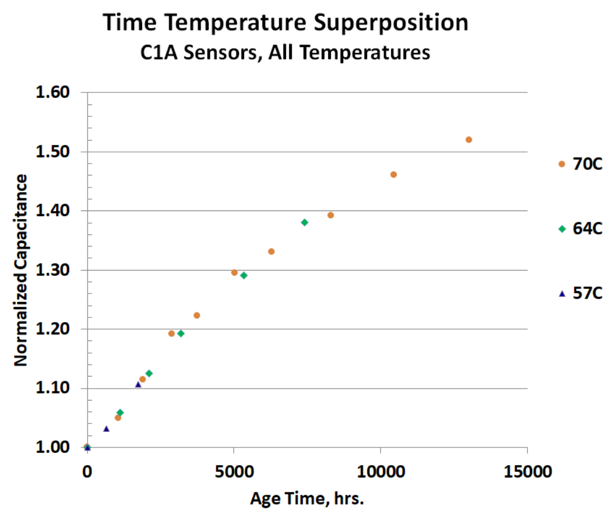

Figure 5. Time Temperature Superposition (TTS) graph of 70C, 64C and 57C temperature aging capacitance of C1A sensors.

Table 1 summarizes the calculated AFs for the C1A sensors determined by TTS for each aging temperature based on Figure 5.

Table 1. Acceleration Factors (AFs) for normalized C1A sensor capacitance for each aging temperature.

Temperature (C)	Acceleration factor
57	1.0
64	3.2
70	6

Although fewer data points are available for the lower temperature analysis during early aging as compared to the higher trial temperatures, less extrapolation to typical operating temperatures makes selection of the lowest aging temperature more representative of expected real-time aging. Therefore the 57C data was used as the reference temperature for determination of acceleration factors and sensor activation energy.

Figure 6 shows the natural logarithm of the reaction rate of the sensors (represented by the AF of normalized capacitance) vs. the inverse absolute temperature of aging. The linear correlation value calculated using the three temperature data (R^2 > 0.98) demonstrates the Arrhenius behavior of the sensor is consistent over the tested temperature range. The activation energy (Ea), related to the slope of the line, was determined to be 31 kcal/mol.

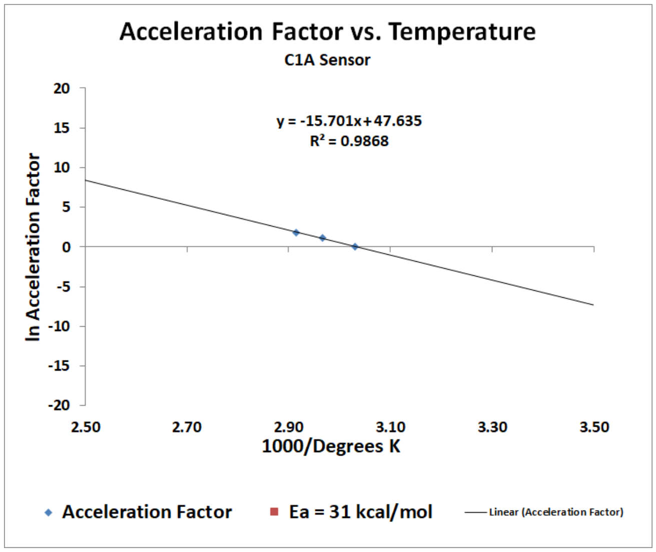

Figure 6. Linearity of the natural logarithm of acceleration factors with respect to inverse absolute temperature demonstrates Arrhenius behavior of the C1A sensor.

3. TARGET MATERIAL PROPERTY ANALYSIS

In order to determine aging through use of a thermal sensor a property that changes over time is selected that shows repeatable change. Most products comprising polymers generally have at least one trait that demonstrates aging of the material. For example, most polymeric adhesives, coatings and encapsulants demonstrate significant changes in mechanical properties, hardness, or molecular weight changes during thermal curing and thermal degradation during life.

In a constant thermal environment such as a laboratory oven, prediction of thermally-induced properties of a product can be accomplished by a simple correlation of time in the environment to measured properties of the material at selected intervals.

Most products and materials in consumer, industrial and military applications are subject to a more complex thermal history from the time of manufacture, including transportation, storage and operation. Since product condition properties are affected by chemical reactions, which by their nature are non-linear, simple time predictions are inadequate.

Since the capacitive thermal age sensors of this method follow Arrhenius behavior as discussed in the previous section, determination of the Arrhenius behavior of a product to be monitored for thermally-induced properties provides a means to correlate sensor reading with current product condition. With assumed future product conditions, such as continuation of the historical environment, prediction of remaining thermal life can be made.

In the current method, characterization of the target thermal property behavior is similar to that used in characterization of the thermal age sensors. Product or product materials are thermally aged under multiple constant temperature environments. Product properties of interest are measured periodically during aging. Arrhenius behavior properties are determined by TTS or other methods. A minimum of two temperature aging is required for this analysis, but a minimum of three is needed to demonstrate Arrhenius behavior.

Several target components for correlation with thermal age were selected for this project. O-rings and gasket samples of several different materials were thermally co-aged together with the capacitive thermal age sensors discussed in the previous section. Durometer (Shore A hardness) was selected and measured during aging at each temperature since durometer is a property frequently used to evaluate the suitability for O-rings, gaskets, hoses and other elastomeric products.

Figure 7 shows the increase in durometer reading (Shore A hardness) of nitrile O-rings during aging at the same temperatures as the CTA tags. Note the indicator line at the 5 point Shore A hardness increase, indicating a common end-of-life condition for O-rings. Error bars of one standard deviation are included to show the error within the early stages of the study. End of life indicator in the figure shows a hardness increase of 5 Shore A units.

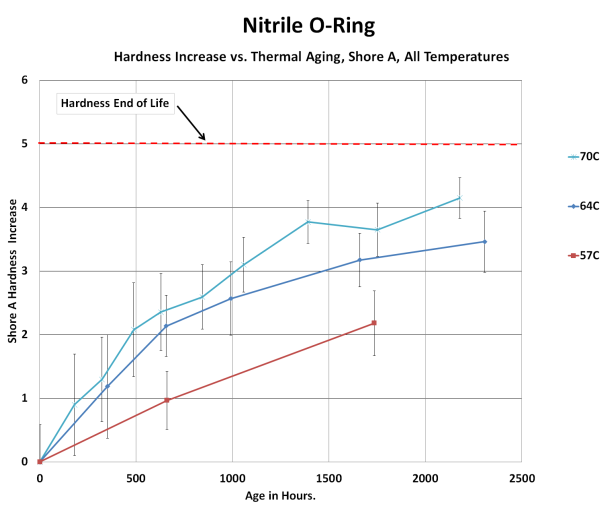

Figure 7. Increase of Shore A hardness with thermal aging for nitrile O-rings measured during co-aging with CTA sensors at three temperatures.

The same TTS approach was used with the Nitrile O-ring hardness increase as with the sensors. The superposition chart (Figure 8) shows reasonable overlay and similar shapes of the target material at each temperature.

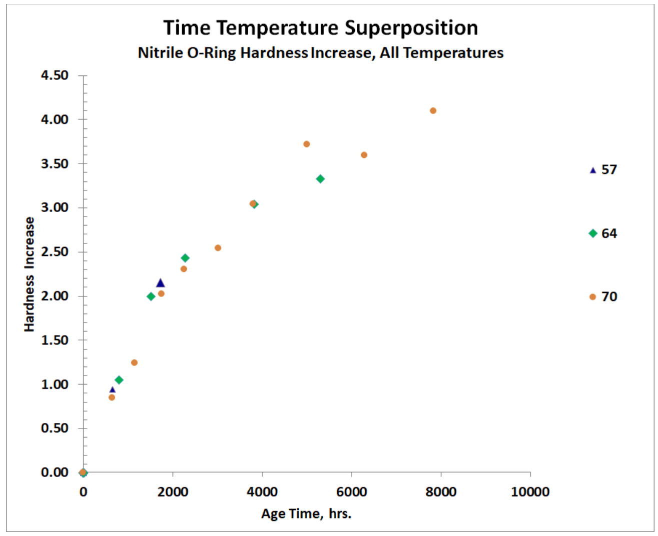

Figure 8. Time-temperature-superposition (TTS) results of multi-temperature aging of Nitrile O-rings.

The acceleration factors for each temperature determined by the TTS chart of Figure 8 is shown in Table 2.

Table 2.Acceleration Factors (AF) for nitrile O-ring hardness increase vs. age time for each aging temperature.

Temperature (C)	Acceleration factor
57	1.0
64	2.3
70	3.6

Utilizing the acceleration factors (AF) from the table above, an Arrhenius chart of the natural logarithm of the acceleration factors at inverse absolute temperature is created as shown in Figure 9. Linearity (R2 > 0.95) of the data indicates Arrhenius behavior of hardness increase with Nitrile O-ring aging, and suggests the ability to correlate thermal age sensor readings with thermal aging of nitrile O-rings.

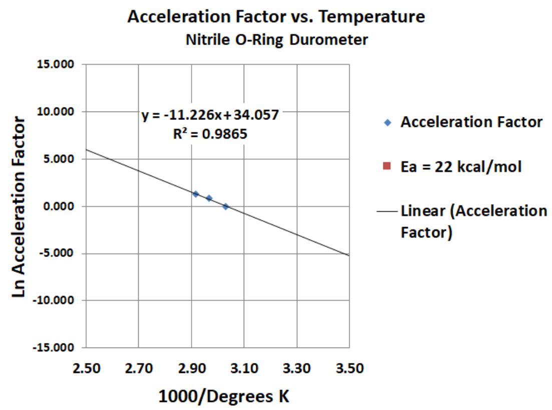

Figure 9. Arrhenius chart of Nitrile O-ring hardness change during thermal aging using the acceleration factors obtained from Figure 8.

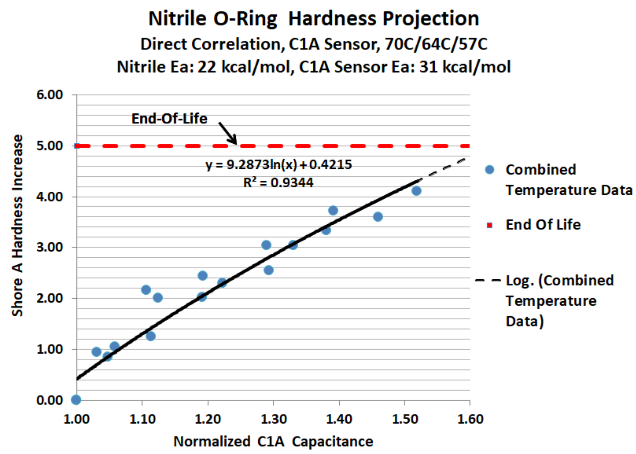

Figure 10. Direct correlation of C1A thermal age sensor normalized capacitance with Nitrile O-Ring hardness increase.

4. EMPIRICAL CORRELATION MODELS

Several empirical correlation models used in this project provide a projected target condition based on capacitive thermal age sensor readings. These include direct correlation, effective temperature (T_{eff}) and multi sensor T_{eff} correlation and will be discussed in the following sections.

4.1 Direct Correlation

Direct correlation is a best-fit curve of thermal age sensor capacitance, representing the integrated time and temperature of the product it is associated with, to product condition. The correlation requires product and sensor aging data at the same times and temperatures.

As discussed previously, the sensor is co-aged with the target product to provide the aging data. However, where co-aging data is not available, Arrhenius methodology may be used to adjust the times and temperatures of either the product or sensor aging data so that the same data points on the correlation curve define points of equivalent thermal age.

Figure 10 is a direct correlation model for nitrile O-ring hardness increase as a function of thermal age as measured by the C1A capacitive thermal age sensor discussed in section 2. These sensors were co-aged with nitrile O-rings discussed in Section 3. Average values of normalized sensor capacitance were correlated with average values of O-ring hardness increase at the same thermal exposure (same time at each temperature).

It is worth noting that results of Figure 10 demonstrate reasonable correlation despite a significant difference in the Ea of the sensor (31 kcal/mol) and Ea of the target degradation mode (22 kcal/mol).

Three temperature (70C, 64C, 57C) data was used in this correlation model. A straight line curve fit provided reasonable fit ($R^2 > 0.90$), and the dotted line continuation of the correlation curve provides predicted values to end-of-life.

This trial is still in process, and will be continued until the highest temperature data reaches or exceeds end-of-life conditions. The correlation curve is expected to become non-linear later in life as the reaction rate of the sensors, represented by the normalized capacitance rate of change, begins to decrease with increasing thermal age.

A significant advantage of the direct correlation method is that once the correlation model is established for a target product or material, only the sensor reading of a CTA sensor associated with the target material is needed to predict the condition of the product.

4.2 T_{eff} Correlation Model

A second approach to correlate CTA sensor reading with product condition is to utilize the sensor capacitance reading to determine the effective or kinetic temperature of the environment it has been in since sensor association with the product. (Harrah et al, 1980)

T_{eff} is defined as the single constant temperature that will provide the same product degradation over the monitored time period as a variable temperature profile does over that same period. T_{eff} is different from average temperature in that chemical reactions that drive most degradation processes are highly non-linear. A relatively short temperature excursion may have a significant effect on overall degradation due to the non-linearity in reaction rate mechanisms. The only time that that T_{eff} is equal to average temperature is when the temperature is constant throughout the period, or when the activation energy of the degradation reaction that is being monitored is zero.

T_{eff} can be found using the acceleration factor of the calendar time to reach the present sensor value and the time required to reach the same value at a selected sensor reference temperature. The Teff is then found by entering the sensor Arrhenius curve with the calculated sensor AF which provides T_{eff} of the sensor environment at the effective activation energy of the sensor. This T_{eff} is then used to project the product condition from the reference degradation curve of the target at the target Ea. An example utilizing sensor (C1A) utilizing target aging data from 70C, 64C and 57C is shown in Figure 11, Figure **12** and Figure 13.

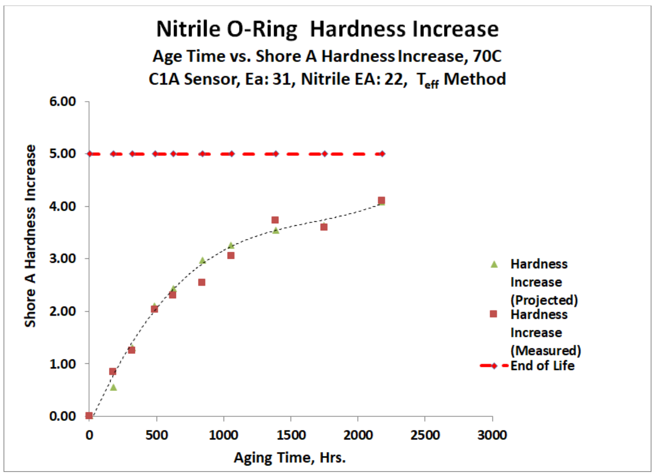

Figure 11. Nitrile O-ring hardness increase as a function of 70C age time, T_{eff} method.

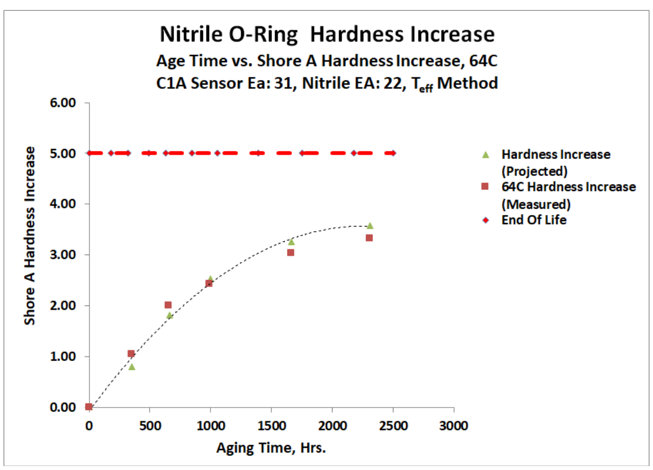

Figure 12. Nitrile O-ring hardness increase as a function of 64C age time, T_{eff} method.

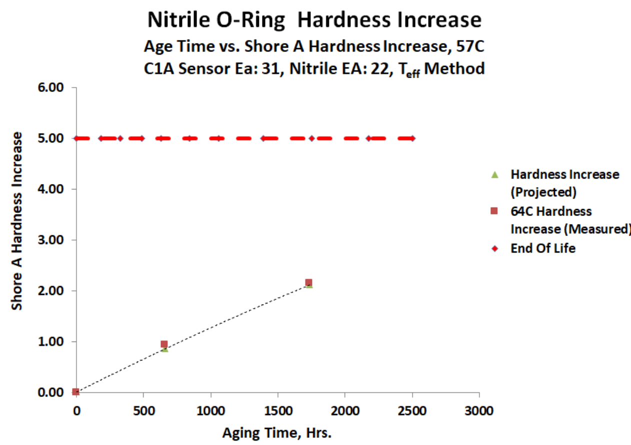

Figure 13. Nitrile O-ring hardness increase as a function of 57C age time, T_{eff} method.

The T_{eff} method improves projection accuracy as compared to the direct correlation method at the temperature extremes when the difference of activation energy between sensor and target is significant. However, calendar time (the elapsed time from sensor association with target) is required in calculating T_{eff}. Calendar time is not required for direct correlation models.

4.3 Multi-Sensor T_{eff} Correlation Model

Good accuracy of a modeling method for real-world (variable temperature) aging will necessitate adjusting Teff for the target Ea. A large difference between the sensor Ea and the product Ea will result in large projection errors in product condition, especially when the environmental temperature change is significant, as might occur during transportation, storage and operation. Designing sensors to achieve a specific thermal age Ea response is presently a trial-and-error process. Designing, manufacturing and deployment of the large number of different sensors required to match a wide range of target products represents a significant commitment of time and resources.

When sensor Ea and target degradation Ea are not similar, a multi-sensor T_{eff} approach can overcome this issue. In this project, multiple sensor types and target materials were co-aged in a variable temperature environment in order to demonstrate a model which adjusts the T_{eff} based on the Ea of the target degradation parameter.

The method correlates the activation energy of the sensor as described in section 2 with the effective temperature as described in section 4.2. A linear correlation is assumed with two sensors as in this example. The resulting algorithm provides a means to calculate the effective temperature of an environment at any other target product Ea.

The chart in Figure 14 shows a method of projecting T_{eff} based on different sensor Ea. This correlation predicts T_{eff} of degradation for a material of different Ea than either sensor.

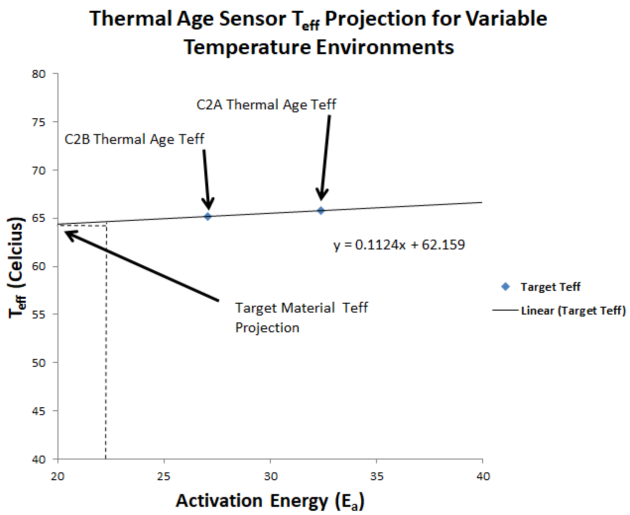

Figure 14. Correlation of T_{eff} to the Ea of a sensor.

The results of this analysis at three time periods during the variable temperature aging are shown in Figure 15. The method of Section 4.2 was used to determine T_{eff} for both sensors co-aged in the variable temperature trial. T_{eff} for the target material degradation measurements (Nitrile O-Ring hardness Ea) utilized the method of Figure 14.

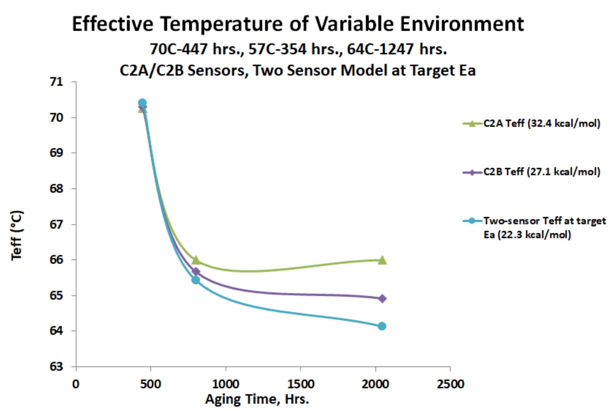

Figure 15. Effective temperature for a variable temperature environment as a function of activation energy. Note the target T_{eff}, with lowest activation energy, is lower than either T_{eff} calculated for the sensors.

Figure 16 below shows the projected hardness increase of nitrile O-rings determined at the effective temperatures of both C2A and C2B sensors and the projected Teff using the Ea of the target material.

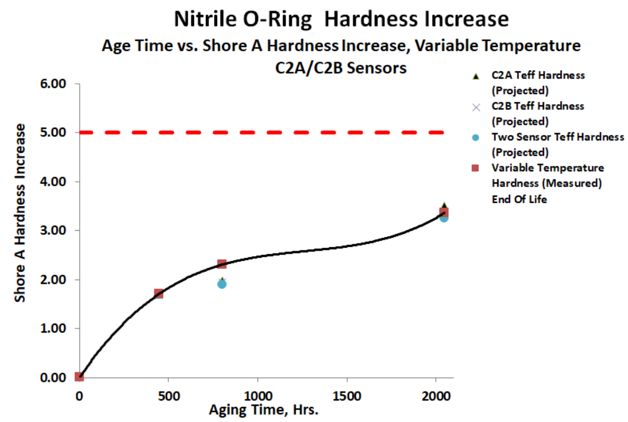

Figure 16: Comparison of nitrile O-ring hardness increase in a variable temperature environment utilizing the two sensor T_{eff} approach results of Figure 15 .

The relative differences between the individual and multi-sensor hardness projections of Figure 16 are consistent with those expected, i.e., the target projections are lower for the two sensor projection than those by the single sensors alone when adjusted for target Ea. Differences of projected hardness increase and measured hardness increase may be attributable to the relatively large variance in durometer measurements of the trial and the relatively small sample size of the variable temperature trial. Further investigation is required to confirm these results. Other thermo-kinetic models that compensate for thermal age sensor-target product Ea mismatches are being evaluated as part of this project.

5. APPLICATIONS OF THE APPROACH

Simplicity, low cost, small size and elimination of the requirement for complex data management and battery replacement issues associated with data loggers are the motivating factors for application of thermal age tags in applications. It is important where shelf life, fixed replacement and "run-to-failure" methods are insufficient. Passive thermal age sensors provide the simplest and lowest cost upgrade from simple shelf-life or operating-time methods and bridge the gap to extensive multi-sensor integrated vehicle health monitoring (IVHM) methods used in large, fixed asset applications. They offer significant cost reductions resulting from reduced unscheduled downtime for rapidly aged equipment, and reduced premature replacements where equipment is replaced on a fixed replacement time scenario.

The versatility of this approach is shown in Figure 17 below, using one of the same sensors (C2B) used in predicting the degradation of nitrile O-rings to predict the end of shelf life of aspirin based on literature aging data (Al-Gohary et al, 2000). The direct correlation method of section 4.1 was used to model aspirin degradation data at three temperatures (70C, 60C and 50C). C2B sensor data was adjusted to the equivalent aspirin measurement temperature and times by the method discussed in section 4.

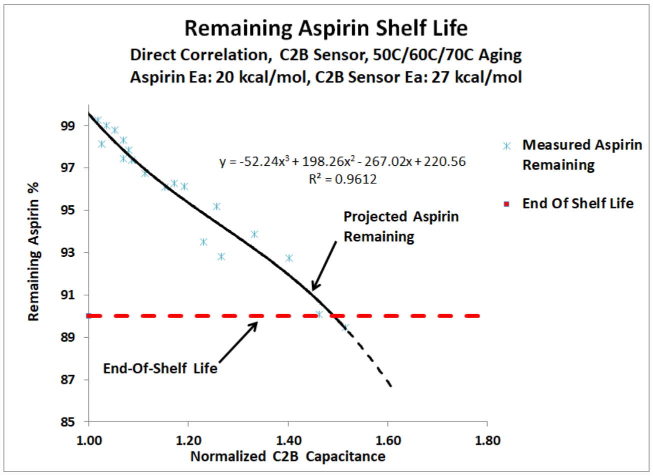

Figure 17. Direct correlation modeling of thermal age sensor C2B capacitance to predict the end of shelf life for aspirin.

Tiny, single sensor tags such as those shown in Figure 18 can be attached to individual products such as motors, generators, transformers, wire and cable insulation, elastomeric seals, gaskets and hoses, even food and pharmaceutical products. Resistive thermal age sensors, disclosed in a previous paper (Watkins, 2018) can be formed as films on products and read by contact readers.

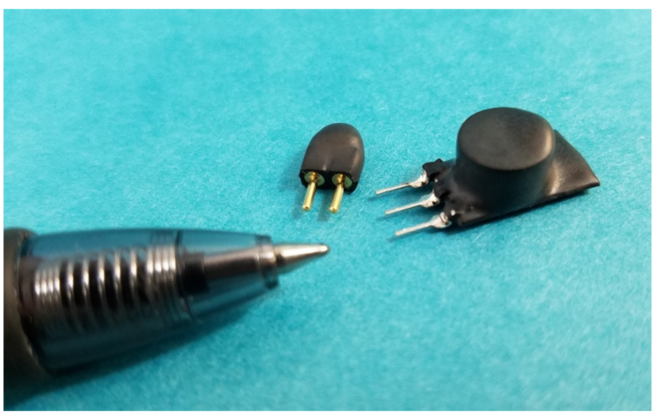

Figure 18: Small single and dual sensor thermal age tags can be used to project remaining thermal life of a wide variety of individual products.

The passive nature of thermal age tags lends well to passive Radio Frequency Identification (RFID) tag approaches where a thermal age sensor is utilized in a passive RFID tag to both track a product and predict remaining thermal life. An example of a passive RFID tag with thermal age sensor is shown in Figure **19**.

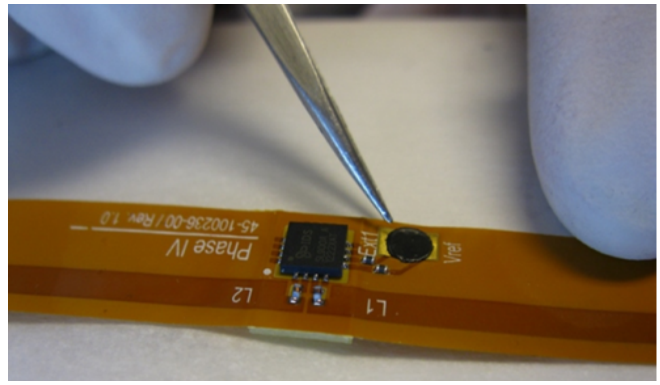

Figure 19. The passive (no battery) feature of thermal age tags allows full integration with passive RFID tags.

Additional tag capabilities can be expanded with additional sensor capabilities including chemical sensors and shock/vibration sensors. Polymer Aging Concepts has shown feasibility of a passive thermal/humidity age tag capable of predicting material properties under both variable thermal and variable humidity conditions.

6. CONCLUSIONS

Completion of this project is expected to demonstrate that thermal age sensors offer a simple, low-cost approach to identify prematurely aged materials and components and to extend the shelf life of materials and components which are optimally transported and stored.

Capacitive thermal age sensors offer a new simplified PHM approach that eliminates the need for conductive fillers used in conductive composite thermal age sensors while providing excellent thermal age resolution. And these sensors retain the completely passive (no battery, elimination of data logging requirements) and "life of the asset" benefits of resistive thermal age sensors.

Multiple empirical modeling approaches can be employed that correlate the capacitance of the thermal age sensor (representing the integrated time-temperature of the tag environment) to current target material or product condition. Accurate correlation requires multiple target material property data at multiple temperatures and property threshold data for "Red light/Green light capability. These empirical approaches will not detect product degradation or faulty engineering caused by product manufacturing defects.

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