Development of a Nanoparticle Time-Temperature Sensor for Passive and Active RFID

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Abstract—We have developed a thermally activated Time-Temperature Indicator (TTI) suitable for use in passive and active RFID applications. In general, this type of indicator can be used in the cold chain to monitor the thermal age of foods and pharmaceuticals as well as provide more accurate estimates of shelf life. In this manuscript we have sought to design an indicator with a time-temperature profile similar to that of milk. The sensor is composed of a dc resistance measurement of Cobalt/Antimony (Co/Sb) multilayer films. Upon deposition and processing, the Co self-assembles into single domain magnetic nanoparticles approximately 20 nm in-plane and 4 nm perpendicular to the plane resembling nano-pancakes embedded within the Sb matrix. At ambient temperatures the Co nanoparticles ensemble magnetization decreases. This is a thermally activated process that follows an Arrhenius like behavior. As the sample magnetization decreases, the resistivity of the material also decreases following the same thermally activated behavior as the magnetization.

I. INTRODUCTION

There is a clear desire in the RFID community to be able to monitor temperature profiles of individual products along the supply chain. [1] Monitoring can lead to more efficient processing of perishable goods as well as provide end users with an assurance of quality. Currently, the most common type of monitoring includes active RFID sensors that monitor and store temperatures at discrete time intervals for downloading and signal processing, providing users important information along the supply line. This monitoring provides a temperature record that can be analyzed in situ or by RFID readers. The main drawback of these type of sensors is that they require a power source and, to date, are limited to active RFID or semi-passive RFID.

Several techniques have been developed for measuring different aspects of temperature characteristics of passive RFID labels. Sample et al. presented a Wireless Identification and Sensing Platform (WISP) which could sense and feed back temperature readings within a UHF RFID platform. [2] Bhattacharyya et al. have designed several novel temperature threshold sensors using shape based polymers that actuate for temperature changes through a particular temperature such as the freezing temperature. [3][4][5] Hassan et al. evaluated the possibility of using a Reflected Electro-Material Signature (REMS) sensor to sense the electrical properties of thermotropic liquid crystals embedded in a polymer substrate to sense and store a temperature profile as a function of time. [6]

A type of time-temperature indicator using thermally activated chemical reactions have found efficacy in both drug and food monitoring. [7] [8] This type of indicator relies on time and temperature dependent chemical reactions to provide an indicator, usually a color change. The time-temperature properties of these chemical reactions are governed by an Arrhenius law. This is the same law that governs the degradation in many foods and pharmaceutical products and the reactions can often be designed to fit a particular application. There are several distinct advantages to this type of sensor. First, these types of indicators require no external power source as they rely on energy from the surrounding thermal bath for activation. Second, they are relatively inexpensive to produce and therefore can be applied to individual production units.

Attempts have been made to produce a Time-Temperature Indicator (TTI) for passive RFID applications similar to the chemical sensors mentioned above. In 2005, a European consortium including government, industry and academia formed the “Chill-On” project. [9] One of the primary goals of this project was to develop a TTI indicator with the time-temperature and energy characteristics of the above described chemical indicators but with changing electronic characteristics (as opposed to color changes) suitable for use in passive RFID.

An ideal time-temperature indicator for truly passive RFID cold chain applications demands several properties. First, it is required that the indicator have well defined time-temperature characteristics that span both the necessary time and temperature regimes (i.e. minutes to months for temperatures ranging from ∼ 50 °C to -10 °C). Second, these time-temperature properties should manifest within the electronic properties of the material used. Third, the time temperature properties should be driven by the thermal energy of the surrounding heat bath negating the need for an onboard power supply. Fourth, they must be able to be probed at low power (< 20μW). Finally, for application to individual consumer items, the indicator must be very inexpensive to produce.

In this paper we report a thermally activated time-temperature indicator suitable for use in passive RFID applications. The material we have developed displays thermally activated time-temperature dependent resistivity changes. It has been reported that in Co/Sb (1.5 nm/ 2.5 nm) multilayer samples, both the magnetization and the resistivity have similar
time-temperature dependencies. The authors observed a magnetization decay of almost 80% of the initial magnetization and a resistance decay of almost 50% of the initial resistance. In this paper we have produced samples with three different thicknesses of Co. As the Co thickness decreases the time-temperature dynamics change. We use these results to attempt to produce a material with time-temperature dynamics similar to that found in milk.

A. Experimental Methods

The multilayer samples were prepared in an Edwards E306A e-beam evaporator with a starting pressure of 2x10\(^{-7}\) torr. Samples were deposited on the <100> face of silicon held at a temperature of 50 °C. Previous measurements have shown that similar time-temperature results can be obtained on mylar film and on glass. We have produced Co/Sb multilayer samples with Co layer thicknesses of 1.5nm, 1.0nm and 0.8 nm. All samples studied in the following resistance vs. time measurements had Sb layer thicknesses of 2.5 nm. For these samples, an initial 2.5 nm layer of Sb was deposited on the bare substrate followed by the layer of Co. This was repeated 10 times and the sample was capped with a final 2.5 nm layer of Sb. The deposition system is not automated, requiring manual gun rotation as well as manual deposition temperature control. After deposition, the master sample were removed from the chamber and stored in liquid nitrogen to effectively stop thermal aging. To prepare a sample for a resistance measurement the master sample was removed from liquid nitrogen and brought to room temperature in a desiccator to prevent water formation. An approximately 4 mm x 4 mm sample was cut for the measurement and the master sample placed back into the liquid nitrogen.

All resistance measurements in this study were conducted using the MagnetoResistance (MR) probe option on a LakeShore Model 7307 Vibrating Sample Magnetometer (VSM). After loading the sample onto the MR probe and measuring the room temperature resistance, the sample was inserted into the cryostat which had been preheated to the measuring temperature. All resistance measurements in this study were made with a dc technique. Measurements were conducted with both 2-probe and 4-probe dc resistance techniques. The 4-probe technique is an in-line measurement with the current leads set 3 mm apart and the voltage leads separated by 1 mm located in-line 1 mm from the respective current leads. The 2-probe measurement sensed the voltage drop across the current leads. The 2-probe measurement therefore measures a voltage drop across 3 mm of sample as opposed to 1 mm in the 4-probe measurements. The resistance decay results for the Co/Sb (1.5 nm/ 2.5 nm) and Co/Sb (1.0 nm/ 2.5 nm) samples were made with the 4-probe technique. The resistance decay results for the Co/Sb (0.8 nm/ 2.5 nm) samples were made with the 2-probe technique. We have also made both 2-probe and 4-probe measurements on the Co/Sb (1.5 nm/2.5 nm) samples, for comparison. Finally, for the resistance vs. temperature measurements a 10 layer Co/Sb (1.0 nm/2.0 nm) sample was produced.

![Graph showing decay of normalized resistance as a function of log time for Co/Sb samples](image)

**Table 1**

VALUES OF THE CHARACTERISTIC TIME \(\tau\) (TIMESCALE AT .85 \(R/R_0\)) AS A FUNCTION OF TEMPERATURE FOR CO/SB (0.8 nm/2.5 nm).

<table>
<thead>
<tr>
<th>Temp (K)</th>
<th>(\tau) (sec.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>290</td>
<td>8.9x10(^{5})</td>
</tr>
<tr>
<td>300</td>
<td>3.27 x 10(^{5})</td>
</tr>
<tr>
<td>310</td>
<td>9.87 x 10(^{4})</td>
</tr>
<tr>
<td>320</td>
<td>4.01 x 10(^{4})</td>
</tr>
<tr>
<td>330</td>
<td>2.20 x 10(^{4})</td>
</tr>
<tr>
<td>340</td>
<td>1.09 x 10(^{4})</td>
</tr>
<tr>
<td>350</td>
<td>3.04 x 10(^{3})</td>
</tr>
</tbody>
</table>

Variations in sample size produced notable changes in the initial room temperature resistance. In the 2-probe measurements, small samples (2 mm x 4 mm) had initial room temperature resistances as large as 700 \(\Omega\). Large cut samples 5 mm x 6 mm had much smaller initial resistances on the order of 200 \(\Omega\). The standard 4 mm x 4 mm had initial room temperature resistances of 380±20 \(\Omega\). 4-probe measurements produced resistances approximately an order of magnitude smaller.

In general, for the first 7 to 10,000 seconds a resistance measurement was taken every 200 seconds. For times longer than 7000 seconds, a measurement was taken every hour or every three hours; for very long time measurements that extend out to approximately two weeks. All resistances were measured in zero magnetic field.
B. Experimental Results

Fig. 1 shows a complete set of resistance versus time decay curves for Co/Sb (0.8 nm/2.5 nm) with temperatures ranging from 290 K to 350 K. It can clearly be observed that at higher temperatures the sample ages more rapidly. The resistance decay process is irreversible so each curve corresponds to a different sample cut from the master sample. In each sample the resistance decays from its initial resistance value of $R_o$ to a final resistance of approximately’s 0.6 $R_o$. The final resistance ratio $R/R_o \sim 0.6$ for the samples with Co thickness of 0.8 nm is similar to the final resistance ratios of 1 nm Co samples $R/R_o \sim 0.6$ and greater than the 1.5 nm Co samples, $R/R_o \sim 0.5$.

For the Co/Sb multilayer samples with Co layer thicknesses of 1.5 nm and 1.0 nm, we have used the inflection point of the decay curves plotted on a log scale to determine a characteristic timescale $\tau$ for each individual decay curve. This type of analysis is common in the analysis of aging curves, dating back to analysis of polymers by Struick [11] with application to spin glasses by Lundgren et al. [12]. The inflection point is a natural place for determining a characteristic timescale as it is a peak in the logarithmic derivative function $S(t) = dM_{TRM}(t, t_o)/d\ln t$. If one has a continuous set of measurements then this timescale makes sense. If however the samples is being probed intermittently, such as in RFID detection along a supply chain, there may not be enough data to make a full decay plot or a logarithmic derivative plot. To simplify this analysis for the Co/Sb (0.8 nm/2.5 nm), we acquired the time scale associated with a resistance value of $0.85 R_o$. This value is a little larger than the inflection point for all of the curves generated. Fig. 2 displays the resistance curves in the vicinity of $0.85 R_o$. From this graph we extract the timescale associated with the value of each individual resistance curve at $0.85 R_o$. While simplifying this analysis to extract a single timescale is useful for Arrhenius law fitting, this technique does not preclude systematic or intermittent measurements of the decay for more detailed analysis.

In Fig. 1 the resistance vs. time plots are plotted on a log scale within which it may be difficult to appreciate the wide range of time scales spanned for the temperature range. In Table I we list values of Temp. (K) vs $\tau$ (sec.). At 350 K the time to decay to $0.85 R_o$ is $3.04 \times 10^3$ sec. or about 50 minutes. At 320 K the time to decay to $0.85 R_o$ is $4.01 \times 10^4$ sec. or about 11 hours. Finally at 290 K the time to decay to $0.85 R_o$ is $8.9 \times 10^5$ sec. or about 10.3 days.

C. Thermal Activation Analysis

The time and temperature dependence of the decays in these samples indicate that the process is thermally activated. In nature when a system in one state can transition to another state by thermally activated hopping over an energy barrier, the system can generally be described by an Arrhenius law.

In chemical reactions the Arrhenius law is often written as

$$k = Ae^{-\frac{E_a}{RT}},$$

(1)

where $k$ is the reaction rate $E_a$ is the activation energy, $R$ is the ideal gas constant and $T$ is the temperature in Kelvin. $A$ is the frequency factor and gives the rate for zero activation energy or infinite temperature. In a chemical reaction, the activation energy is the energy needed to be overcome the energy barrier that separates an initial chemical state from some final chemical state.

For the time temperature analysis used in this paper, we will use the time dependence for the state transition given by the Neel-Arrhenius form [13]. This form was originally used to describe the characteristic spin flip time (barrier hopping time) of superparamagnetic nanoparticles.

$$\tau = \tau_o \exp\left(\frac{E_a}{RT}\right),$$

(2)

To make contact with the chemical reaction analysis of food products we will substitute in the ideal gas constant and analyze data for a straight line on a $\ln(\tau)$ vs. $1/T$ plot using the equation:

$$\ln(\tau) = \frac{E_a}{R}\left(\frac{1}{T}\right) + \ln(\tau_o),$$

(3)

In Fig. 3, we plot a graph of $\ln(\tau)$ vs. $1/T$ for three Co/Sb samples plus data for milk from ref. 4. In Fig. 4 the straight line fits to the activation energy are extrapolated to $1/T=0$ K$^{-1}$, $T=\infty$. The y-intercept on this graph give the fluctuation time scale $\tau_o$. Table II displays the values of the activation energy $E_A$ for three Co/Sb runs (varying thickness) and the fit parameter for the milk data. [7] Table II also displays the fluctuation timescales $\tau_o$ for the four samples. It is clear from Fig. 3 (slopes of the lines) and from Table II that all four samples have reasonably similar activation energies.
Fig. 3. Plot of $\ln(\tau)$ vs. $1/T$ for four samples Co/Sb (1.5 nm/2.5 nm), Co/Sb (1.0 nm/2.5 nm), Co/Sb (0.8 nm/2.5 nm) and milk from ref. [7].

Fig. 4. Same plot as in Fig. 3 but on an expanded scale. The straight lines are the Arrhenius law fits to the data. $E_a$ is extracted from the slopes of the lines. The y-intercept correspond to $\ln(\tau_o)$.

Observing the activation energies and fluctuation timescales ($\tau_o$) for the Co/Sb (1.5 nm/2.5 nm) and Co/Sb (1.0 nm/2.5 nm) samples we chose to make Co/Sb (0.8 nm/2.5 nm) as our first attempt at a milk sensor for the following reasons: First, the data have activation energies similar to milk. Second, the time-temperature characteristics shift towards the milk curve with decreasing Co thickness. In particular $\tau_o$ decreases as the Co thickness decreases. A further shift, we believed would put us near the approximate timescales of milk. The time-temperature characteristics of the deterioration of milk were observed in the acidification of milk increasing to a titratable acidity of 18°T. [7] While $\tau_o$ did shift down, as expected, and $E_a$ varied a little, we found that while the Co/Sb (0.8 nm/2.5 nm) is closer to the milk data it is still not an adequate overlap. In the future, we will endeavor to produce samples with even smaller thicknesses of Co to produce a perfect overlap.

Table II

<table>
<thead>
<tr>
<th>Sample</th>
<th>$E_a$(kJ/mol)</th>
<th>$\tau_o$(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co/Sb (1.5 nm/2.5 nm)</td>
<td>73.1</td>
<td>4.85x10$^{-7}$</td>
</tr>
<tr>
<td>Co/Sb (1.0 nm/2.5 nm)</td>
<td>73.9</td>
<td>1.13x10$^{-7}$</td>
</tr>
<tr>
<td>Co/Sb (0.8 nm/2.5 nm)</td>
<td>76.6</td>
<td>1.42x10$^{-8}$</td>
</tr>
<tr>
<td>Milk [7]</td>
<td>78.4</td>
<td>9.89x10$^{-9}$</td>
</tr>
</tbody>
</table>

D. Reproducibility and Power Analysis

We next analyzed the reproducibility of the resistance decays of Co/Sb (1.5 nm/2.5 nm) at a single temperature, 360 K. Using the 4-probe measuring system described in the experimental methods section, we found that there was some deviation in both the structures of the decay curves and in the characteristic timescale defined by the inflection point. In Fig. 5 we display resistance aging decay curves for the Co/Sb samples using the 4-probe technique. In Fig. 6 we display resistance aging decay curves for the Co/Sb samples using the 2-probe technique. The reasoning that led us to switch probing techniques is the belief that the mechanisms governing these decay curves are statistical. We estimate that there are approximately 2x10$^{10}$ nanoparticles in a single 4 mm x 4 mm sample. The voltage probes in the 4-probe measurement are located only one millimeter apart. This suggests that we are only measuring a small portion of the sample and not averaging over the whole sample. Hence, we are statistically averaging over many fewer nanoparticles. To test this we switched from the 4-probe to the 2-probe technique where the sensing probes (voltage) are now located 3 mm apart. The major change that we noticed going from 4-probe to 2-probe is that the decays of the 2-probe technique are much more reproducible both in time and structure. For example for 340 K, at .85 $R_o$ the time resolution of the three overlapping curves is approximately 10% of the average characteristic timescale for this temperature. This is comparable to chemical time temperature indicators. The increase in accuracy with larger sensing distance is suggestive that the accuracy of the technique may be increased with geometries that average over even more nanoparticles.

While most of the measurements in this sample were made with a DC current of 1 mA some of the measurements were made at .1 mA requiring a power of $\sim 4\mu W$, well within the power requirements of passive RFID. We see no difference in curves measured with 1 mA and .1 mA. Tests have also been made with even smaller currents. With our system, currents less than .1 nA show resistance fluctuations larger than the sample resistance. This may be an equipment limitation. Probing a sample with 1 nA gave resistance fluctuations of approximately 1 $\Omega$ for a 400 $\Omega$ sample, producing an error of approximately .25 %. This is a very small error on the
approximately 40% change in \(R_o\). At 1 nA, the power required to probe the sample is less than a femto-watt.

E. Analysis

We previously described a simple model where the Co/Sb multilayer system can be thought of as two resistors in parallel. [10] One resistor consists of the antimony layers. Sb is a semimetal and has a bulk resistivity of \(417 \text{n}\Omega\text{m}\), almost seven times higher than that of Co \(62 \text{An}\Omega\text{m}\). The second resistor is initially composed of the cobalt nanoparticles. This contribution to the initial resistivity of the sample is very high. While the resistance of cobalt is much lower than antimony, electrical current would not only need to transit through the nanoparticles, it would also need to transit through the highly resistant barrier between the magnetic cobalt nanoparticles and the antimony, as well as transit through the antimony going from one nanoparticle to another. As the nanoparticles begin to coalesce the cobalt acts as a short through larger and larger regions of the antimony. Therefore, one can envision the resistor corresponding to the cobalt resistance decreasing as a function of time. While this simple model can explain the resistance and magnetization decays the resistance vs. temperature data of Fig. 7 suggest that the mechanism may be more complex.

The data of Fig. 7 imply that during the aging decay, the samples may undergo a Metal-Non-Metal (MNM) transition. This could be an important result for RFID implementation as described later. Fig. 7 displays the resistance vs. temperature results for a temperature cycle experiment. The un-aged samples are removed from storage at 77 K loaded onto the MR probe and inserted into the cryostat, precooled to 100 K. From 100 K the temperature was increased and resistance measurements made every 10 K up to 360 K. The resistance vs temperature in the initial ramp from 100 K to 300 K displays a negative slope \(\left(-\left|\frac{dR}{dT}\right|\right)\) suggestive of semiconductor behavior. At 360 K, the three samples were aged for three different times, 300, 6000 and 30,000 sec. The temperature was then decreased to 100K with resistance measurements taken at 10 K intervals. As the time aged at 360 K increases the amplitude of the negative slope \(\left(-\left|\frac{dR}{dT}\right|\right)\) decreases. Finally for the longest aging time (30,000 sec.) the sample has transitioned to a positive slope \(\left(+\left|\frac{dR}{dT}\right|\right)\) implying metallic behavior. At 30,000 sec. of aging, this sample is fully aged as determined from a measured resistance decay curve.

Sb is a semi-metal with a long history of being utilized in semiconductor technology particularly GaSb and AlSb. Kieftite (CoSb\(_3\)), is a well known diamagnetic semiconductor.
At first glance, looking at the resistance vs temperature graph (Fig. 7) and considering possible alloying during thin film deposition one might surmise that CoSb$_3$, a semiconductor, is formed on deposition and perhaps separates into Co and Sb during the aging process, producing a metal. This view is contradicted by STM, AFM, MFM and SEM scans which show nanoparticle formation upon deposition and MFM scans which show evidence of single domain magnetic nanoparticles in un-aged samples. [14] Also, magnetization vs temperature curves display a blocking temperature of $\approx 100$ K indicative of superparamagnetism, hence formation of magnetic nanoparticles on deposition. [15] Finally the magnetic aging signature including an approximately 80% reduction in sample magnetization, eliminates phase separation of CoSb$_3$ as a mechanism for aging. An opposing diffusion model where Sb diffuses into Co nanoparticles or Co atoms into the Sb to form CoSb$_3$ might explain the aging decays of the magnetization but would likely increase the resistance and would be at odds with the data of Fig 7.

Metal-Non-Metal transitions have previously been observed in Bi-Sb alloys. Fenton et al. have observed a magnetic field dependent MNM in Bi-Sb alloys. [16] Both Bi and Sb are semimetals. Heremans et al. have made resistivity and magnetoresistivity studies on Sb nanowires. Both the 10 nm and 48 nm diameter nanowires display a positive dR/dT in calculated resistivity curve for temperatures greater than 100 K. However, on application of a magnetic field dR/dT is negative for the 48 nm wires at least up to 70 K. It is therefore plausible that the negative dR/dT, observed in Fig. 7, for the un-aged or slightly aged samples is due to the Sb layers (2.0 nm layers, 20 nm total Sb thickness) under the influence of the strong magnetic fields exerted locally by the magnetic nanoparticles. As the magnetization decreases this effect also decreases.

There are several issues to be addressed in implementing these sensors in an RFID transponder. A preliminary issue is the presentation of the data sample resistance as a function of $R/R_o$. We cut our samples by hand with a diamond scribe and get variations in sample sizes. As previously mentioned size variations can give significant initial resistance variations without changing the time-temperature dependence. Therefore it is likely that by implementing a consistent geometry in the sensors, absolute resistances will suffice. A second solution would be to store $R_0$ on initialization of the sensor then feeding back both the initial resistance and the resistance at read time to the reader for further processing.

A plausible DC configuration within an RFID circuit might include rectification of the input signal coupled through the Co/Sb sample in series with a known resistance. A measurement of the voltage drop across the known resistance would determine the current through the Co/Sb sample and a measurement of the voltage drop across the Co/Sb sample would, coupled with the current, provide a determination of the sample resistance. These two values could be stored and sent to the reader for further processing. At present we have not yet analyzed the HF response of these samples. Hua et al. have described a capacitative anomaly over the frequency range $1 \rightarrow 3.1 \times 10^7$ Hz in materials undergoing an MNM transition. [18] They found that the capacitance increases markedly when the temperature approaches the MNM transition from the semiconductor side. The data of Fig. 7 indicates that during aging, the Co/Sb samples transition from possible semiconductor behavior to a metallic state. If the capacitative anomaly described by Hua et al. is universal for MNM transitions then the increase in the capacitance could be coupled to, within an LCR circuit. Bhattacharyya et al. have previously described frequency dependent RFID sensors.[19] Without ID logic and within a simple LCR circuit the transponder could be developed as a stand alone Radio Frequency Time Temperature Indicator (RFTTI).

With knowledge of the initial resistance, an in situ resistance reading, and a measurement of the ambient temperature, a good estimation of the shelf life can be obtained from fitted curves. We have obtained good fits to the data with sigmoidal functions. In general we have used sigmoidal function fitting of individual decay curves as a prelude to taking the logarithmic derivative inorder to obtain an S(t) function and $\tau$ from the maximum in that function. Since the RFID application does not require taking a derivative, it is possible that a polynomial fit would suffice. For accurate shelf life readings, for any temperature, a universal sigmoidal/polynomial function will need to be developed which can fit the two resistance points mentioned above for any ambient temperature thereby providing an extrapolated estimate of shelf life at that temperature.

II. Conclusion

There are many potential uses for Time-Temperature Indicators (TTI) in food monitoring and processing. We have presented a TTI material with many attributes necessary for passive RFID. First it has well defined time-temperature characteristics spanning the range required for monitoring of food in the cold chain. There are several significant parameters which can be used to tune the activation energy and fluctuation time scale $\tau_o$. We have looked at several of these parameters such as Co layer thickness and deposition temperatures but there are many other parameters such as composition, alloying etc., which we have not examined. The time-temperature characteristics manifest as a large change in the sample resistance. The effect is thermally driven so there is no need for an onboard power supply. Finally the Co/Sb multilayer system is very inexpensive to produce. Samples measured in this study contain a few micrograms of total material. The nanoparticles are self-assembling requiring no expensive processing and the effect appears to be independent of the type of substrate. [10]
III. ACKNOWLEDGMENT

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