

Phase Transitions in Inorganic Ferroelectric and Piezoelectric Crystals by a Controlled Heating System

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Abstract

An experimental system has been developed to measure capacitance as a function of temperature (CT) within a temperature range of 23.0 Degrees Celsius to 200 Degrees Celsius. This CT characterization system represents one high-temperature-part of a composite system intended to measure CT curves above and below room temperature. The identification of nonlinear elements in CT curves is an important indicator in the characterization of piezoelectric and ferroelectric materials often referred to as nonlinear. Examples of well-known nonlinear ferroelectric materials are barium titanium oxide, BaO_3Ti (BTO) and triglycine sulfate, $C_6H_{17}N_3O_{10}S$ (TGS)[1,2,3,6]. The primary goal of this work is to obtain CT data for novel nonlinear materials such as bis(diisopropylammonium) cobalt (II) tetrachloride. For testing and characterization of this system BTO and TGS were used. CT data for BTO and its nonlinear nature and transition temperature is presented and compared to previous results [1,6].



Figure 1: Prepared sample of BTO Yellow Crystal
Scale: Millimeters



Figure 2: Prepared sample of BTO Grey Ceramic
Scale: Millimeters



Figure 3: Prepared sample of TGS White Crystal
Scale: Millimeters

Theory

Ferroelectric- Is of a body having a susceptibility to an applied electric field, the strength of which depends on that of the electric field. A fundamental characteristic of ferroelectricity is the spontaneous and remnant polarization of dipoles. [6] In this experiment, we search for high dielectric constants and curie points, which suggest large spontaneous polarization, another indication of a proper ferroelectric.[4]

Dielectric Constant $[\kappa]$ - A quantity measuring the ability of a substance to store electrical energy in an electric field. Materials with high $[\kappa]$ are useful in the manufacture of high-value capacitors.

Curie point- The temperature in which certain materials reach a critical point at which it's intrinsic magnetic dipole moments change direction.

Piezoelectric- The ability of certain materials to generate or have the appearance of an electrical potential across the sides of a crystal when subjected to mechanical stress. [5]

Capacitance $[C]$ - Ability of a system to store an electric charge.

$$C = \frac{\epsilon_0 \kappa A}{d} \quad \epsilon_0 = 8.85 \times 10^{-12} [F]$$

Capacitance reactance $[X_c]$ - Represents an opposition to the change of voltage across an element. For high-frequency signals, a charge does not have time to build up before the polarity of the signal changes; therefore, less resistance to the applied signal will be encountered. Frequency applied in experiment: 20KHz.

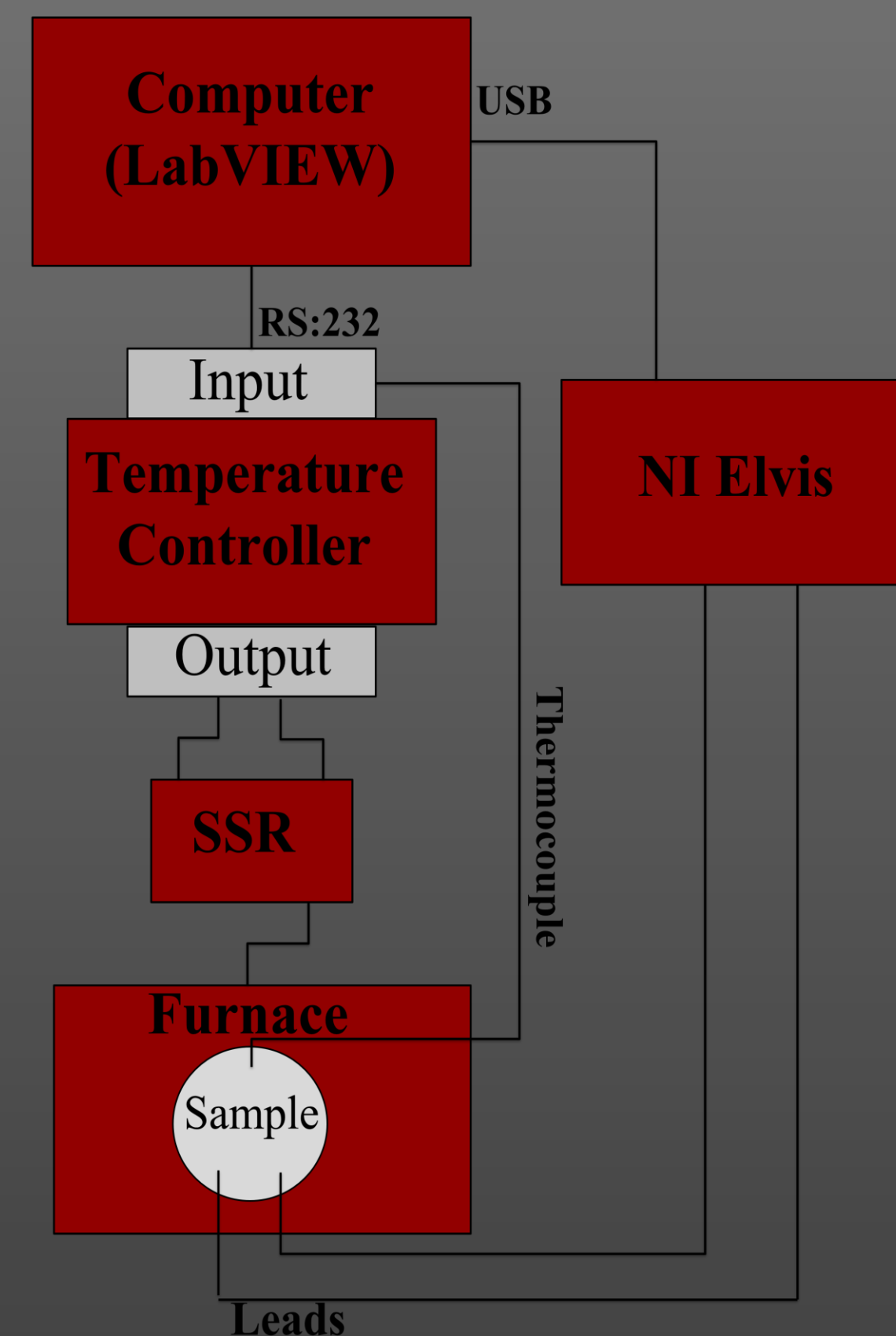
$$X_c = -\frac{1}{2\pi fC}$$

Procedures

Sample Preparation

1. Record thickness of crystal sample using the Starrett 230FL Outside Micrometer No. 230.
2. Place electrodes on the sample by painting a strip of silver paint on each side of the crystal.
3. Record the length and width of the electrodes with the Handheld Digital Microscope Pro and its software: Celestron Micro Capture Pro and Measure.
4. Prepare two lead wires: remove varnish at ends with a handheld blowtorch and clean with alcohol.
5. Adhere, with silver paint, the leads to the sample's electrodes. One on each side of the crystal.
6. Place sample, with leads, onto a glass slide and adhere each lead down onto the slide with superglue.

Closed System Diagram



Experiment Procedures

1. Check continuity and capacitance at each connection point with Handheld Multimeter and Capacitance meter.
2. Fasten the glass slide, with the sample, on the helping hand tool.
3. Place thermocouple directly on the sample and wrap Teflon tape around glass slide and sample.
4. Secure leads to N.I Elvis connection wires.
5. Position sample in furnace and cover furnace openings with aluminum foil to reduce heat loss.
6. Connect power to the system without connecting the furnace to calibrate manually with LabView and Impedance analyzer program.
7. Connect power to the furnace and adjust temperature steadily to assumed curie point with LabView. While monitoring Reactants vs. Temp graph
8. Collect data on the sample as temperature increases and decreases through assumed curie point.

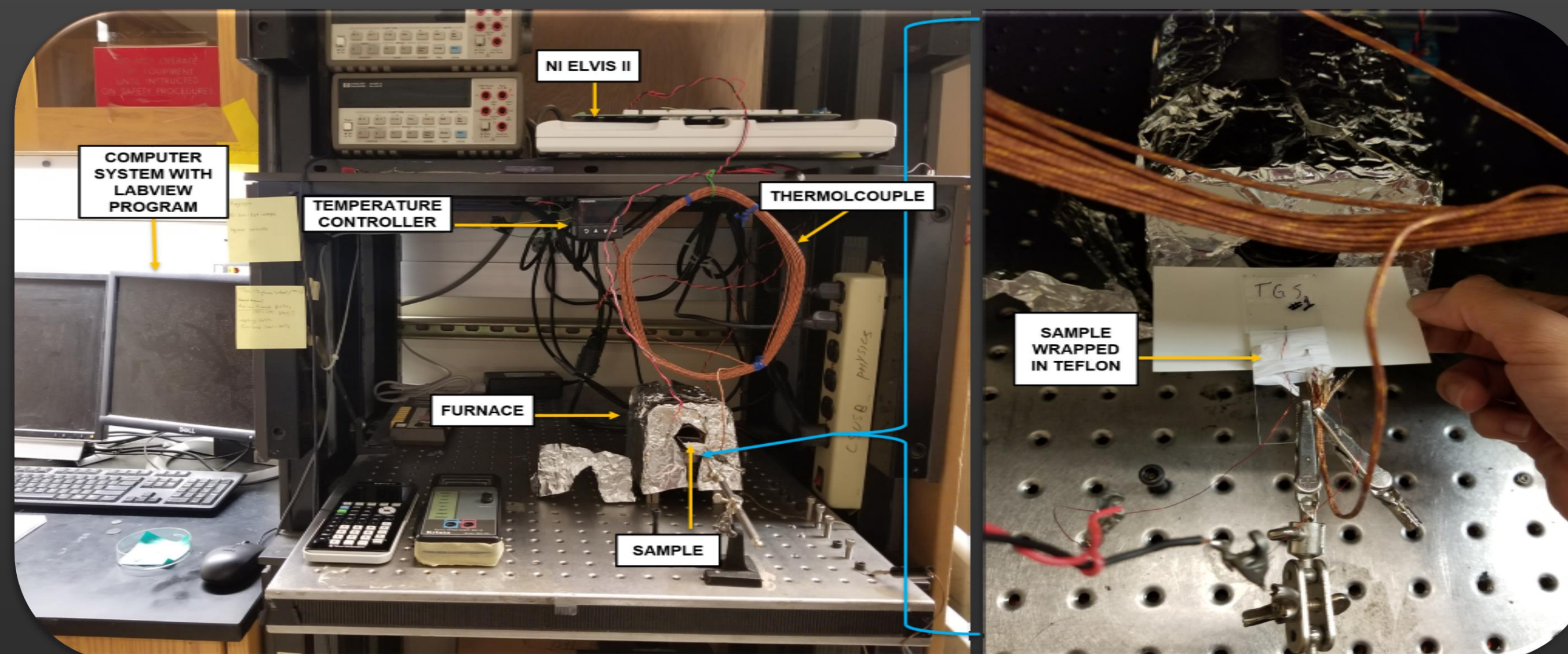


Figure 4: System overview: w/ Sample of TGS prepared and tested in the furnace. Lead wires from the sample are connected to the NI Elvis II, and an impedance analyzer measures Capacitance Reactants vs. temperature. Data is used to create Capacitance vs. Temperature graphs.

Results

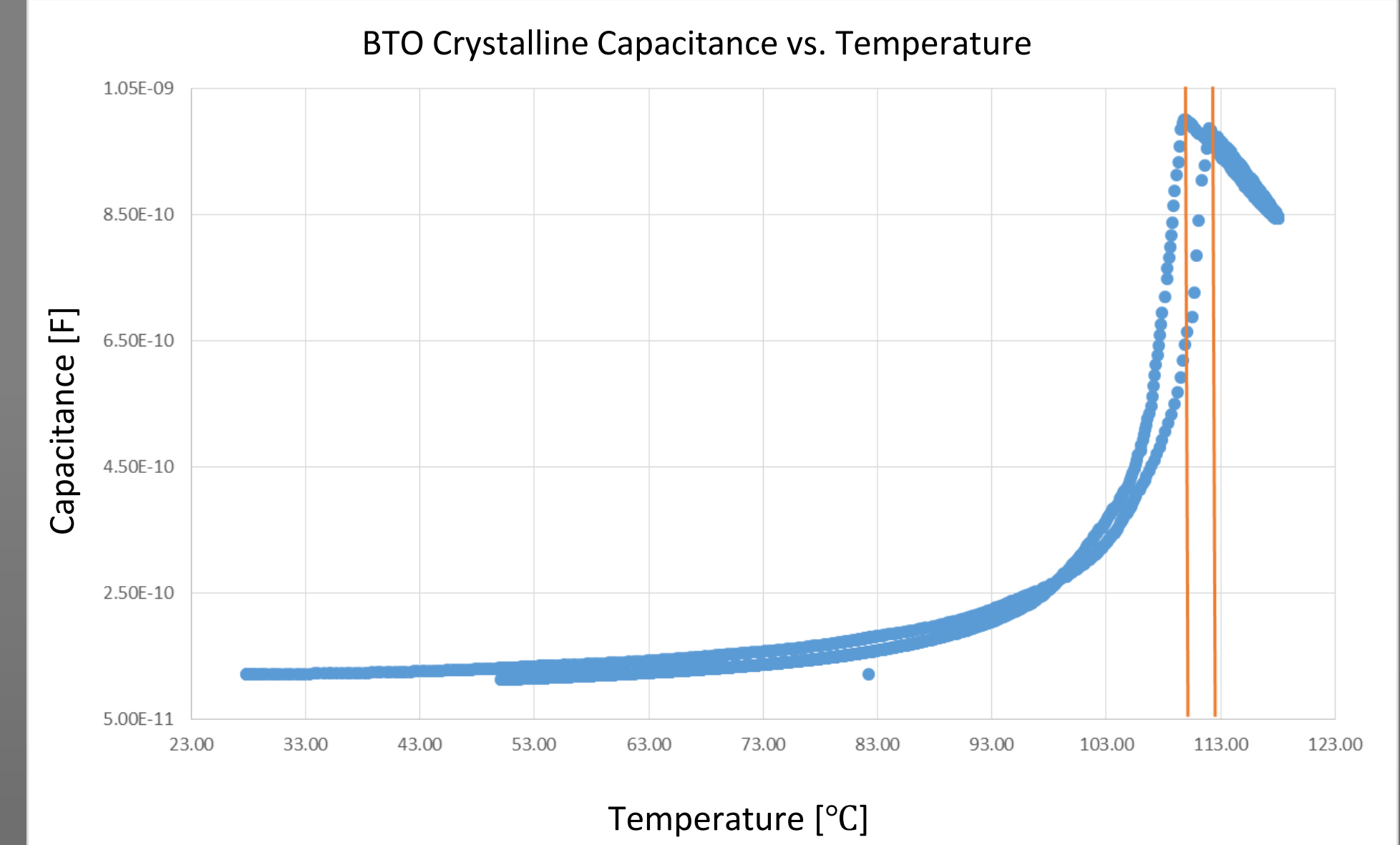


Figure 5: Sample of Crystalline BTO (See figure 1) prepared and tested in closed system. Data of CT conquer with Results from literature. [1,6]

TGS was used in calibration of the closed system due to its low Curie point and ease of replication as compared with literature findings [2,3]. Known samples of crystalline BTO, see figure 1, also produced promising data, see figure 5. Our experimental values were found to be similar to that of published values [1,6]. We concluded our closed temperature system was operational and ready to test unknown piezoelectrics.

Future Work

Due to time constraints, further data collection was limited. Further adjustments are as follows: create an enclosed system around furnace to limit air flow, adjust the programming to restrict user error, test various unknown and known samples for comparison, and create a CT system that lowers in temperature.

References

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