

Electrical properties and phase transitions of ammonium sulphate ferroelectrics

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Abstract: Electrical conductivities, dielectric constant and dielectric loss factor in crystal of ammonium sulphate have been measured as a function of temperature. But at the temperature indicates 480K indicates of a phase transition in the above material at this temperature. The existence of such a phase transition electrical conductivity evaluated from the $\log \sigma$ vs. $10^3/T^{-1}$ plot. The all possible mechanism of electrical conductivity and dielectric constant are discussed with temperature variation by using capacity measuring unit zenith M92A.

Keywords: Dielectric Loss, Conductivity, Curie Temperature, Phase Transition

1. Introduction

Ferroelectric is a phenomenon which was first discovered by scientist J. Valasek in 1921. The name refers to certain magnetic analogies through it is somewhat misleading as it has no connection with iron (ferrum) at all. Ferro electricity has also been called Siegnete electricity as siegnette or Rocble salt RS Wasst material found to show ferroelectric properties such as a spontaneous polarization on cooling below the curie point ferroelectric hysteresis loop. A huge rap in the research on ferroelectric materials came in the 1950's, leading to the wide spread use of Barium titanate based ceramics in capacitor and piezoelectric transducer devices.

All ferroelectric materials have a transition temperature called the curie point. At a temperature $T > T_C$ the crystal does not exhibit Ferro electricity while $T < T_C$ it is ferroelectric on decreasing the temperature through the curie point a ferroelectric crystal undergoes a phase transition from a non-ferroelectric phase to a ferroelectric phase. If there are more than one ferroelectric phase, the temperature at which crystal transforms from one ferroelectric phase to another called the transition temperature. Early research work on ferroelectric transition has been summarized by Nettleton (1922).

Pyro electric crystal shows a spontaneous polarization P_s in a certain temperature range. If the magnitude and direction of P_s can be reversed by an external dielectric

field. Then sugc crystal are said to show ferroelectric behaviour hence all single crystals and successfully pooled ceramics which show ferroelectric behaviour are pyroelectric but not vice versa. Ferroelectric crystal

$$\epsilon = \frac{C}{T - \theta} + \epsilon_0$$

possesses regions with uniform polarization called ferroelectric domains. Within a domain all the electric dipoles are aligned in the same direction. There may be many domains in a crystal separated by interfaces called domain walls. The dielectric constant of a ferroelectric is, of courses not a constant but depends on the field strength at which it is measured this is a consequence of the non linear relationship P and E . The dielectric constant so defined is very large in the vicinity of the transition temperature of the order 10-10 above the transition temperature and the obeys the Curie Weiss laws

Where C is constant, θ is characteristic temperature. ϵ_0 is constant which occurs from electronic polarization .In the vicinity of the transition temperature may be neglected since it is of the order of unity $\epsilon \gg \epsilon_0$ and likewise the susceptibility.

$$\chi = \frac{C}{T - \theta}$$

In this paper we report the results of measurements of a.c and d.c electrical conductivity, dielectric constant and dielectric loss of single crystals of this material. These measurements clearly reveal the occurrence of a high temperature phase transition which is also confirmed by differential scanning calorimetry.

2. Experimental Details

The compound was purchased from Fluka AG Switzerland. The compound sample was prepared by grinding mechanically into fine powder and it has been procured from Fluka Switzerland, about 99.9% puris form sample is avoiding from direct sun light and preferably the most of the sample preparation work was done at night. The powder was heated up to 1073K for 16 hours then cooled to room temperature and grinded mechanically again for an hour to achieve fine powder the pellets were prepared with the compression machine (flexural testing machine CAT No. AIM -313 S. No 9170 AIMIL Associated India limited have pressure range 0-10 tonn wt/cm². A suitable die was used to have rectangular cross section of the position about 2.33cm². the pellet so obtained were covered with the remaining powder and heated again at 1073 K for 16 hours.

The pellets were polished to obtain smooth parallel surface and colloidal silver paint was used to form the electrodes the sample was annealed at 2/3 of its melting point in a furnace for about 8-10 hours. The capacitance of the sample was measured by the MOTWANE capacity measuring Unit DM-3750-B.

The dielectric constant and bulk ac conductivity of the sample were calculated by using following expressions:

$$\text{Dielectric constant (real)} \ \varepsilon = \frac{1 + \Delta C}{C_0}$$

$$\text{Where } \Delta C = C'' - C'$$

C'' is the capacitance of sample holder with specimen.

C' is the capacitance of sample holder without specimen.

C_0 is the geometrical capacitance

$$C_0 = \frac{0.68854A}{d}$$

Where A is area of pellet and d is the thickness of pellet in cm.

Photo room temperature sample holder high temperature sample holder the loss tangent ($\tan\delta$) and parallel capacitance (cp) of the samples were directly measured as function of temperature and frequency by the HP 4194 Impedance analyser.

$$\text{The conductance } G = \omega C_p \tan \delta$$

$$G = 2\pi f C_p \tan \delta$$

$$\text{A.C conductivity } \sigma_{a.c} = \frac{Gd}{A}$$

Where f is the frequency of a.c signal.

The a.c conductance G, capacitor C and dielectric loss $\tan\delta$ were measured as a function of frequency using

Hewlett Packard LF impedance Analyzer (Model 4192A), in the temperature range 100-495K and $\tan\delta$ for single crystals were found to be below the detection limit of instrument except in the high temperature region near the phase transition. The value of a.c conductivity was calculated using the known dimensions of the sample. The dielectric constant was derived from the measured capacitance after eliminating the lead and fringe capacitance using standard methods. Differential scanning calorimetry traces were recorded using a Perkin Elmer DSC-7 instrument in a nitrogen atmosphere at a scanning speed of 10K/min.

3. Results and Discussion

The variation of d.c electrical conductivity with temperature in pure and phosphate doped single crystal is shown in fig. the increase of with temperature is extremely small up to about 340K. A careful analysis of $\log \sigma_{d.c}$ vs. $10^3/T$ plot above this experiment reveals that it is linear up to about 408K. Thereafter it shows a continuous curvature up to 480K where a prominent conductivity anomaly indicative of a phase transition occurs. The conductivity of the doped sample is found to be slightly higher than that of pure sample in the entire temperature range. Again the conductivity along the c-axis is found to be higher than that of temperature dependence of the a.c conductivity and dielectric loss single crystal are shown in fig. the change in $\tan\delta$ and a Δ shaped anomaly in σ_{ac} indicate again a phase transition at 480K in conformity with the d.c conductivity results.

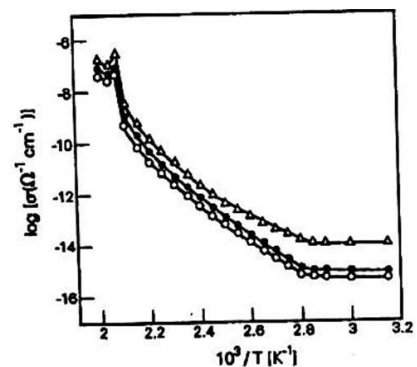


Fig 1. D.C conductivity vs Temperature plots

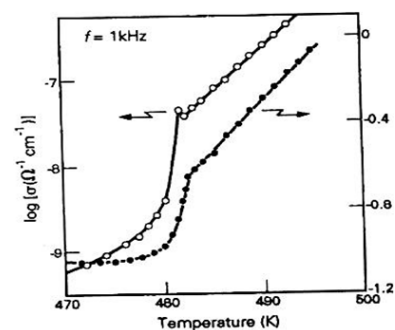


Fig 2. Variation of $\tan\delta$ with temperature

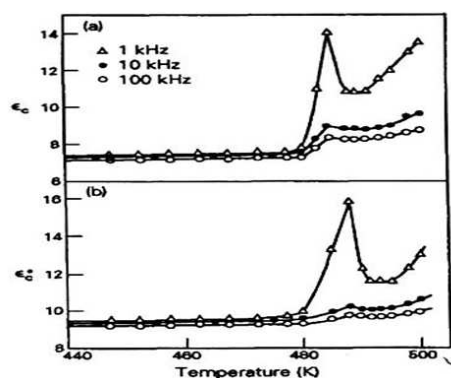


Fig 3. Dielectric constant of EDS as a function of temperature

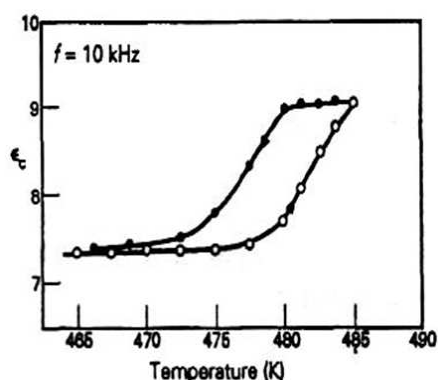


Fig 4. Dielectric constant vs temperature

In fig.3 The variation of dielectric constant along the different frequencies. The value of dielectric constant at 300K is 7.29. ϵ_o vs T plots exhibits Δ shaped peaks exactly at the same temperature where the conductivity anomalies occur viz.480K. The height of the peak is notably suppressed at higher frequencies. Figure 4 shows the temperature recorded on heating and cooling cycles, the measuring frequency being 10KHz. The anomaly shows a thermal hysteresis of about 5K, which is indicative of first order

transition. This transition shows thermal hysteresis characteristics of a first order phase transition. The compound does not exhibit any transformation in the temperature range 300-400K, confirming that it is not a hydrate. The measurement of d.c conductivity and dielectric constant do not show anomalous variation in the temperature region 100-300K ruling out the possibility of a phase transition in this region.

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