CHAPTER - III

DIELECTRIC HYSTERESIS AND COERCIVE STUDIES

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3.1 Introduction :

The basic criterion for the identification of the ferroelectric material is that it must show hysteresis loop under the action of an alternating voltage on CRO screen, connected in the Sawyer and Tower circuit (1930). The hysteresis loop observations enable to measure the spontaneous polarization and coercive field of ferroelectrics. Today large number of materials are known which exhibit ferroelectric properties. Sodium vanadate is one of the ferroelectric crystals. It does not exhibit ferroelectric properties at room temperature but at higher temperature its properties are well observed. Recently, it has been pointed out that several kinds of crystals of XVO_3 type exhibit the ferroelectric properties similar to those of barium titanate.

Ferroelectric properties in sodium vanadate below $380^{0}C$ was first reported by Sawada et al (1951). Cross et al (1955) and Cross (1956) reported that sodium Niobate ($NaNbO_3$) exhibits paraelectric, antiferroelectric and a ferroelectric phase. Wieder (1955) reported the dependence of coercive field upon thickness of single crystal of $BaTiO_3$. Landauer et al (1956) studied the variation of coercive field on frequency and amplitude of applied field for $BaTiO_3$. Abe (1960) gave a theoretical treatment based on the assumption that the coercive field was determined by the velocity of forward growth of the domains.

The ferroelectric behaviour of mixed crystal of sodium vanadate niobate $[Na(V_X Nb_{1-X})O_3]$, was reported by Pulvari (1960). Structural aspects of ferroelectric sodium vanadate ($NaVO_3$), below and above the transition temperature was studied by Ramani et al (1975). Kuroda and Kubota (1980) studied the diffuse phase transition of $(Ba_X - Sr_{1-X})Nb_2O_6$ doped with Pr^{3+} and Nd^{3+} by measuring the spontaneous polarization and dielectric constant. Ferroelectric properties of sodium vanadate and potassium vanadate was studied by Chavan et al (1986) and Patil et al (1988).

3.2 Hysteresis loop :

The properties of ferroelectric materials can be studied by using hysteresis loop. The shape of hysteresis loop depends strongly upon the material as well as upon the rate of change of externally applied electric field. The parameters describing the hysteresis loop are the spontaneous polarization (P_S), the coercive field (E_C) and the susceptibility. When the temperature is lowered from well above the curie point, then spontaneous polarization usually increases rapidly on crossing the curie temperature and reaches a saturation value at low temperature. The coercive field depends not only on temperature but also on the measuring frequency and on the waveform of the applied voltage. The hysteresis loop enables to measure the spontaneous polarization and the coercive field.

3.3 Experimental :

The experimental set-up used to observe the hysteresis loop is shown in fig (3.1). The experimental set-up consisted of an electrically heated furnace, a digital microvoltmeter (Vasavi Electronics Make) and modified form of Sawyer and Tower circuit (1930). The pellets of $NaVO_3$ doped with Nd_2O_3 (0.025 to 1 mol%) were heated slowly inside a furnace and hysteresis loop was observed on the screen of an oscilloscope very easily. The field amplitude across the pellet was 1 KV/cm and frequency 50 Hz. The half-width hysteresis loop enables to measure the coercive field at various temperatures. Modified form of Sawyer and Tower circuit for the display of the hysteresis loop is shown in fig (3.2).

3.4 Hysteresis Loop of Sodium Vanadate :

The hysteresis loop of sodium vanadate was observed on the CRO screen by using modified form of the Sawyer and Tower circuit. The hysteresis loop was observed at different temperatures by heating the sample in the pellet form inside

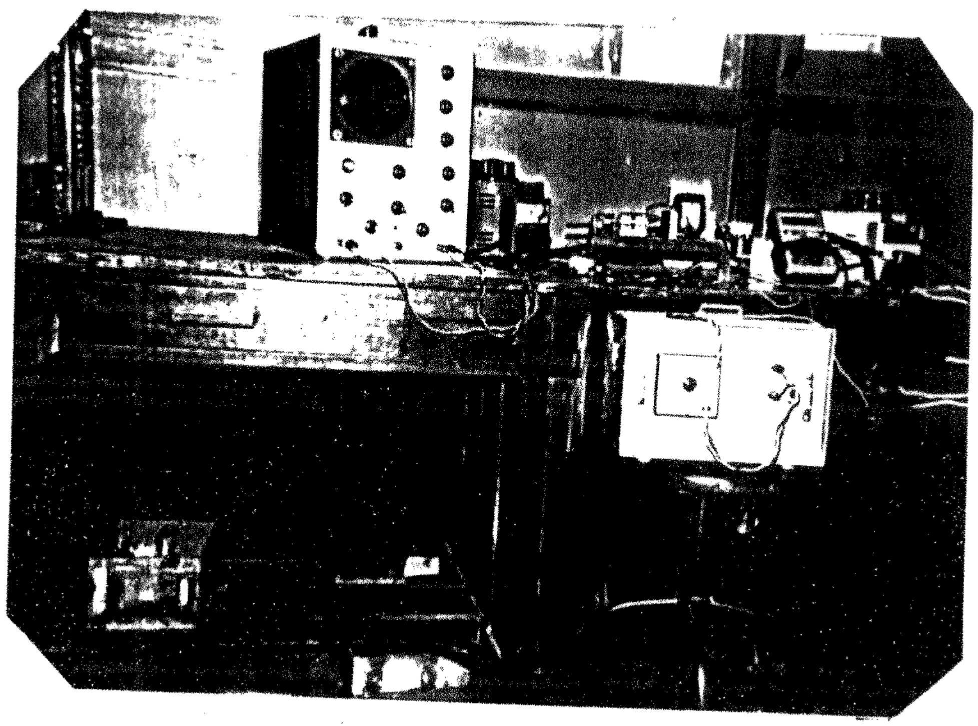


Fig.3.1 – Experimental set-up for studies of the hysteresis loop and coercive field.

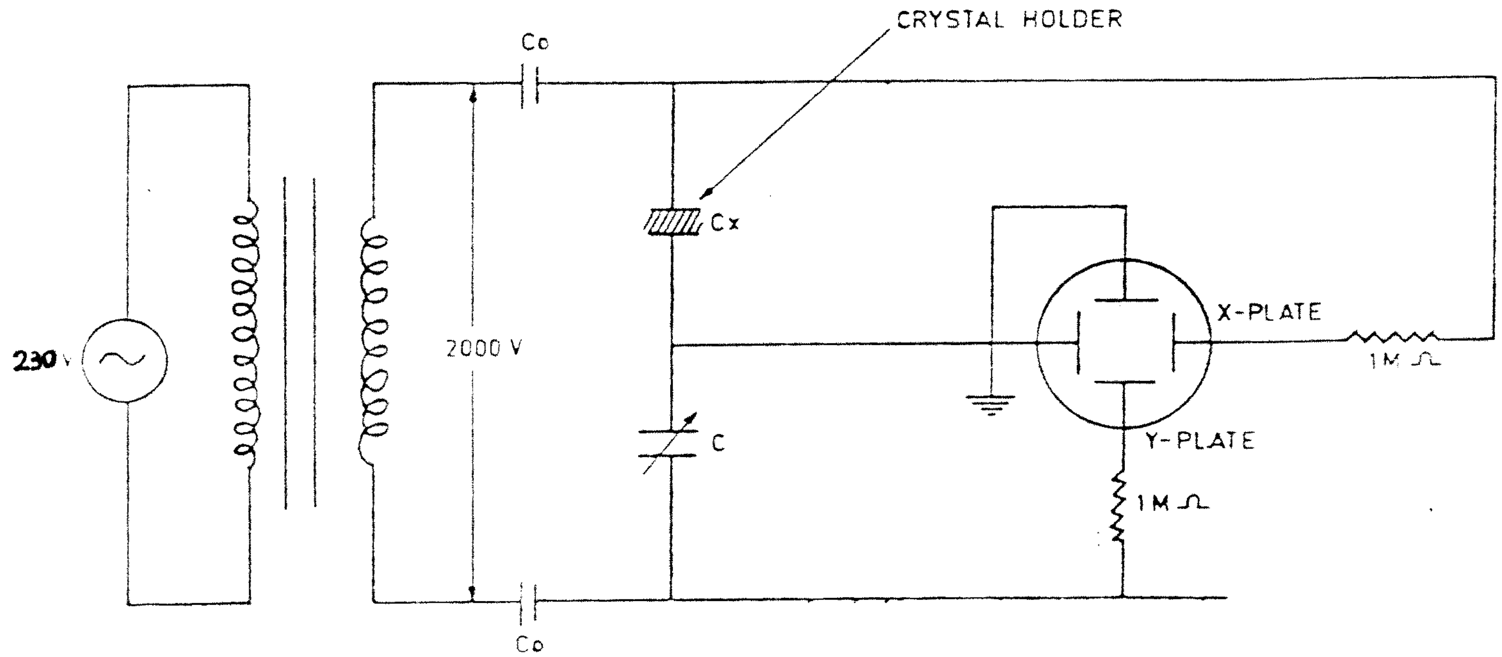


FIG.3.2 SAWYER - TOWER CIRCUIT FOR THE DISPLAY OF THE HYSTERESIS LOOP (MODIFIED FORM).

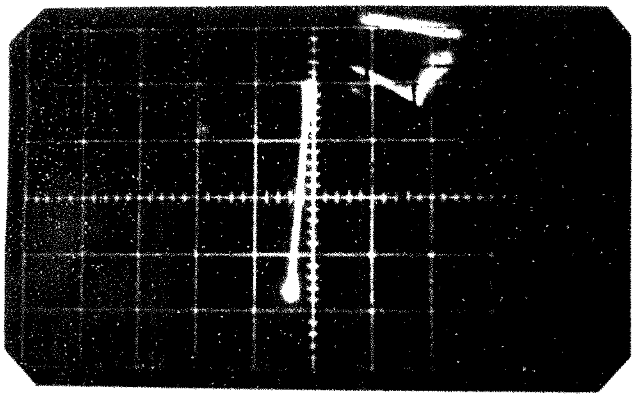
the furnace. The hysteresis loops for $NaVO_3$ at different temperatures are shown in fig (3.3). It is observed that broadens the hysteresis loss is small at room temperature and it broadnens at a certain temperature. The loop width gradually decreases and finally vanishes at curie temperature of the material. The curie temperature of $NaVO_3$ is found to be 380°C.

3.5 Hysteresis loops of $NaVO_3$ doped with Nd_2O_3 (0.025 to 1 mol%) :

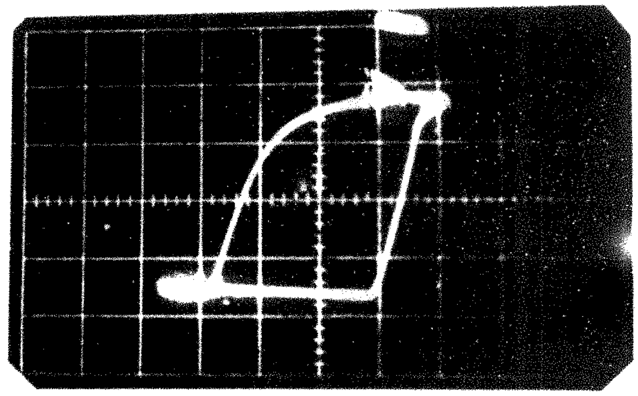
Ferroelectric hysteresis loops of $NaVO_3$ doped with different concentrations (0.025, 0.05, 0.1, 0.5 amd 1 mol%) of Nd_2O_3 are shown in figs (3.4 to 3.8) respectively. For these mixtures, it is observed that the loop width (loss) is small at room temperature and it becomes pronounced at a certain temperature. The loop width gradually decreases and vanishes at the ferroelectric curie temperature of the respective mixture. The ferroelectric curie temperatures investigated for $NaVO_3$ doped with different concentrations (0.025, 0.05, 0.1, 0.5 and 1 mol%) of Nd_2O_3 are 330°C, 320°C, 310°C, 300°C and 290°C respectively.

3.6 Coercive Field :

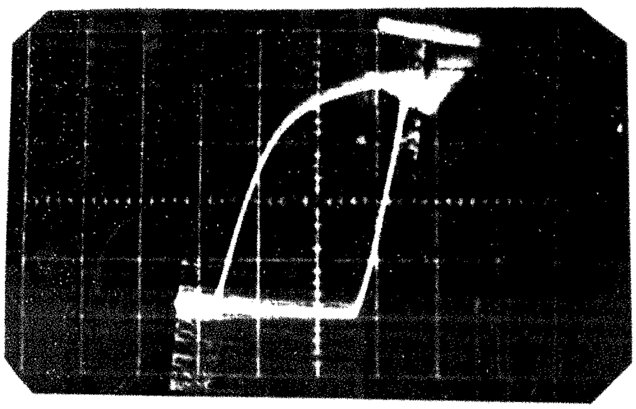
The coercive field for ferroelectric substances is obtained from the figure of hysteresis loop [fig (1.1),



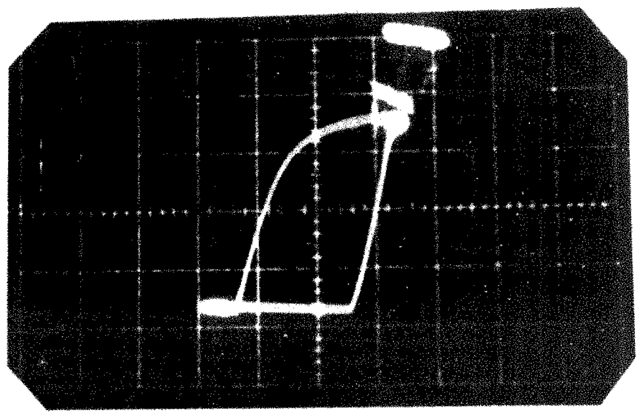
(a) ROOM TEMP



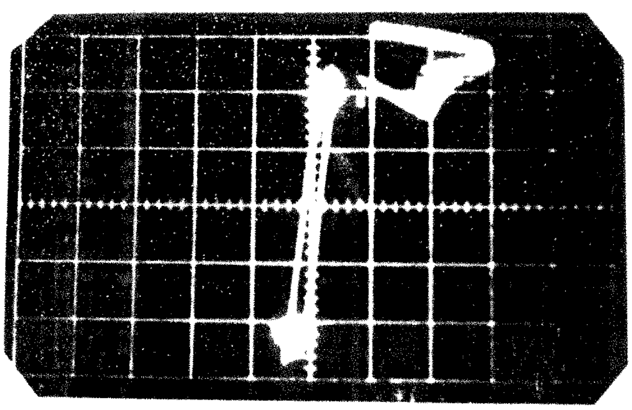
(b) 170 °C



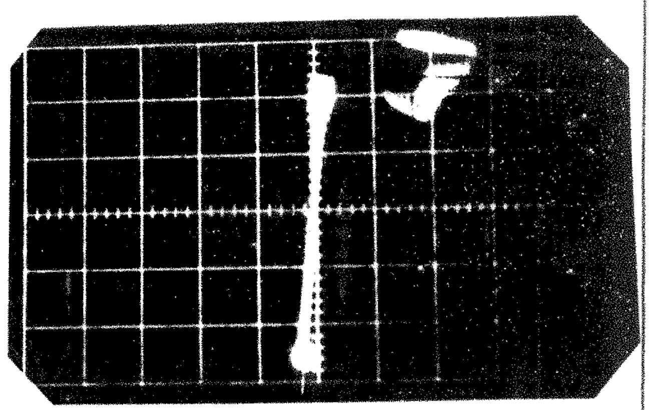
(c) 220 °C



(d) 250 °C

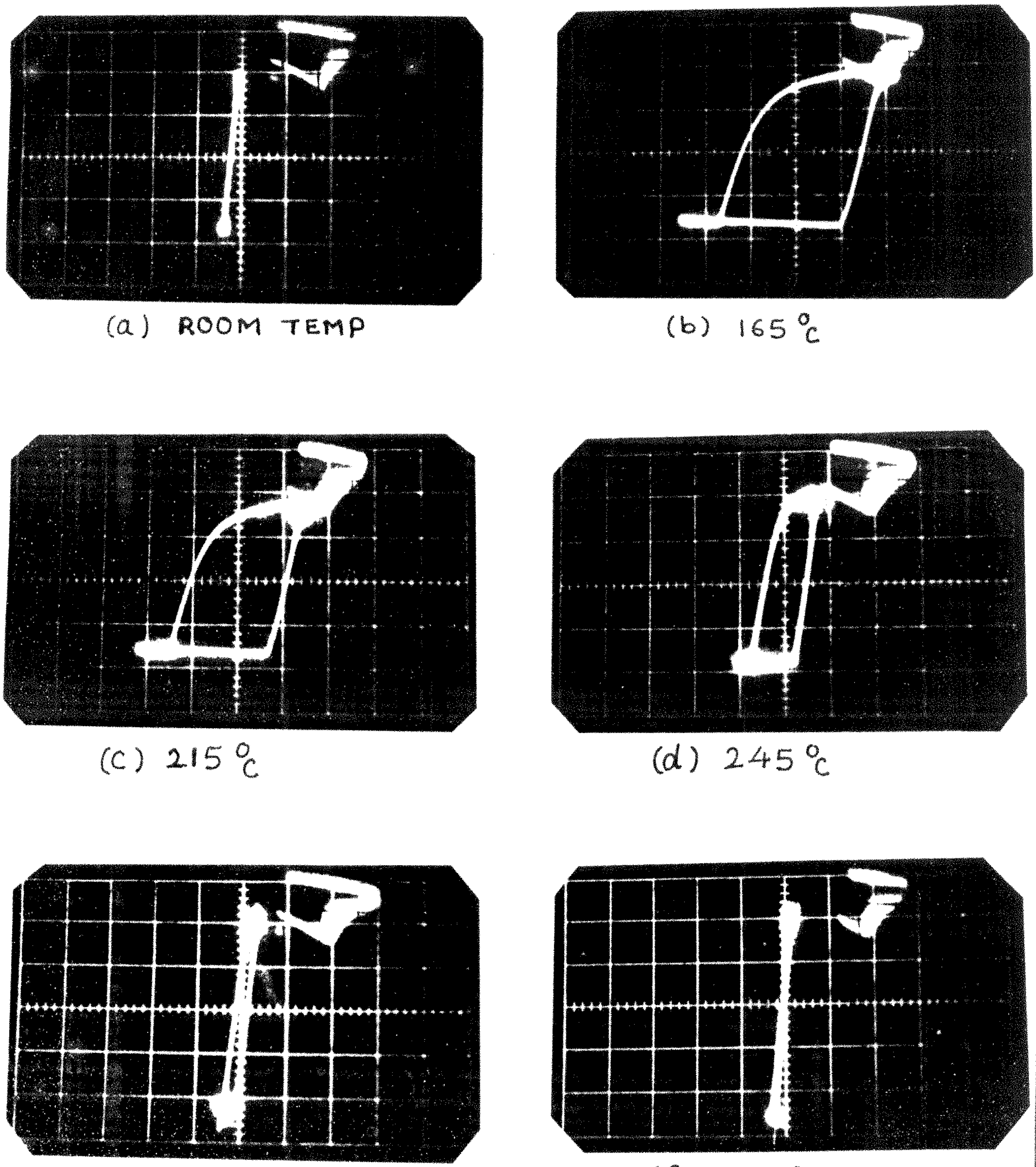


(e) 330 °C



(f) 380 °C

Fig 3.3 – Ferroelectric hysteresis loop of $NaVO_3$.



(a) ROOM TEMP

(b) 165 °C

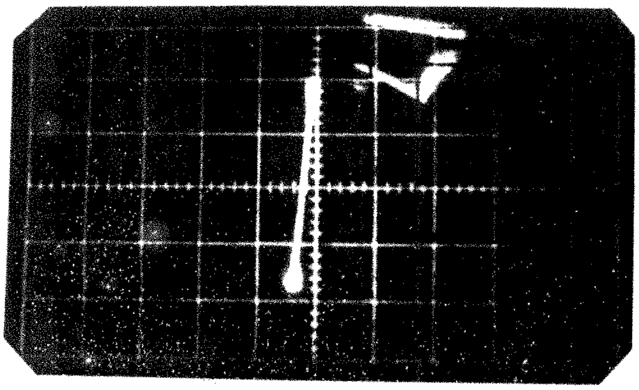
(c) 215 °C

(d) 245 °C

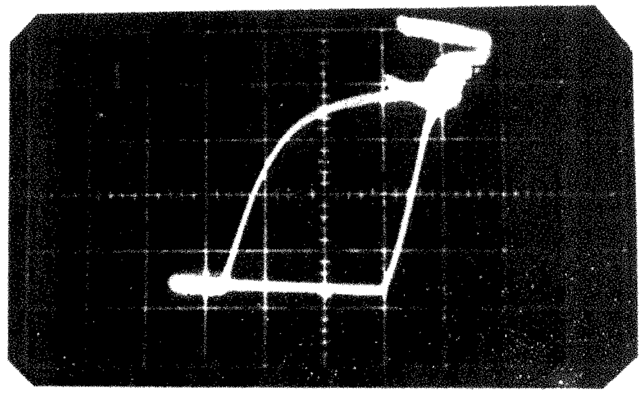
(e) 265 °C

(f) 330 °C

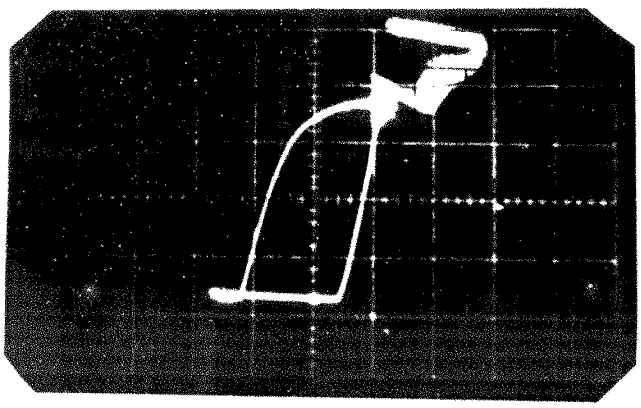
Fig 3.4 – Ferroelectric hysteresis loop of $NaVO_3$ + 0.025 mol % Nd_2O_3.



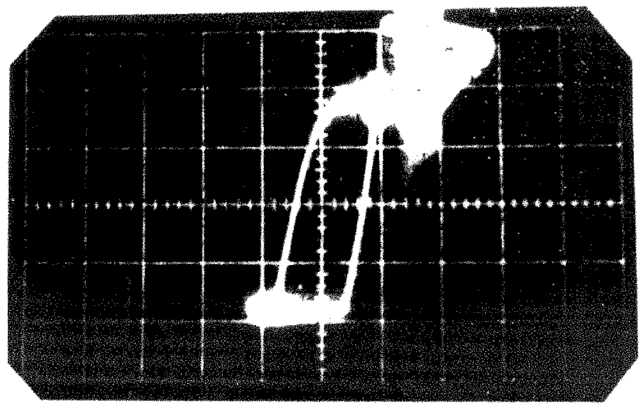
(a) ROOM TEMP



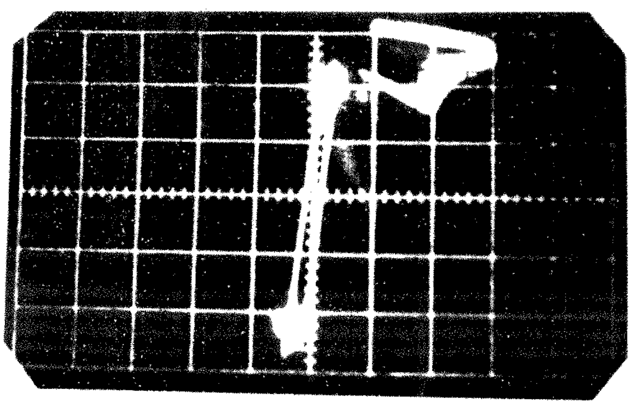
(b) 215 °C



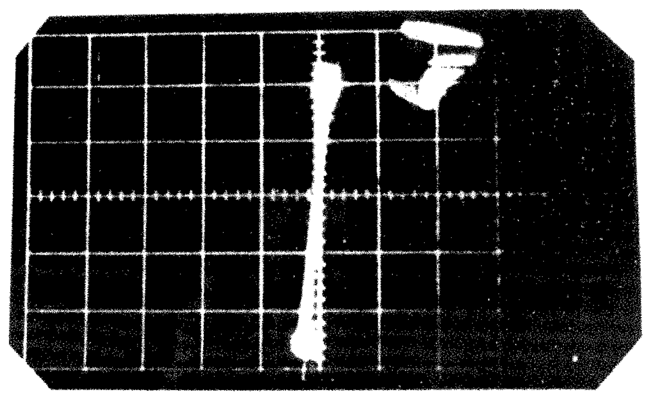
(c) 245 °C



(d) 255 °C

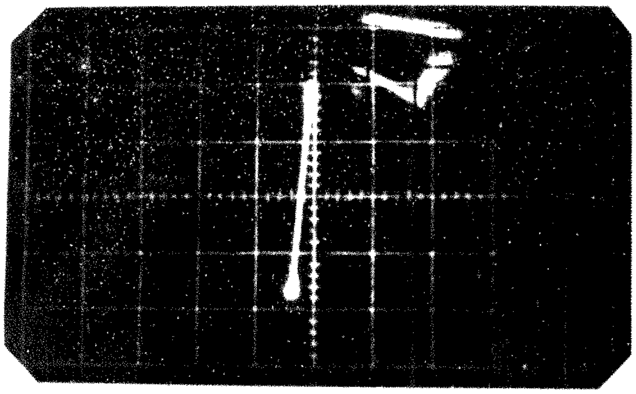


(e) 285 °C

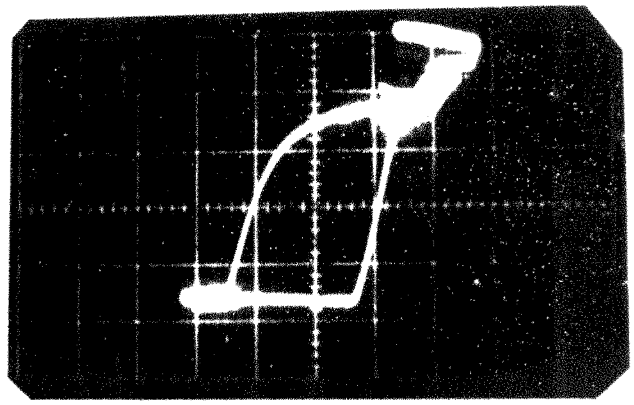


(f) 320 °C

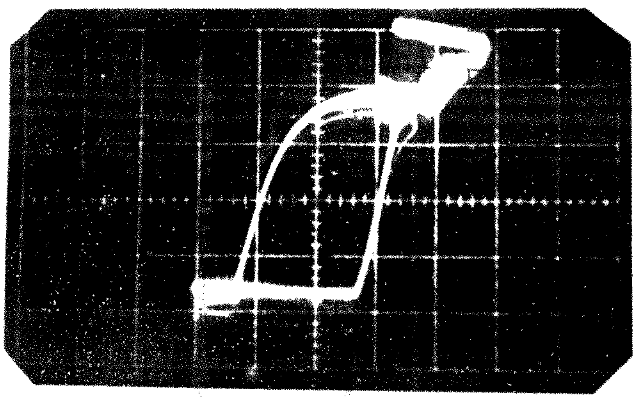
Fig 3.5 – Ferroelectric hysteresis loop of $NaVO_3$ + 0.05 mol % Nd_2O_3.



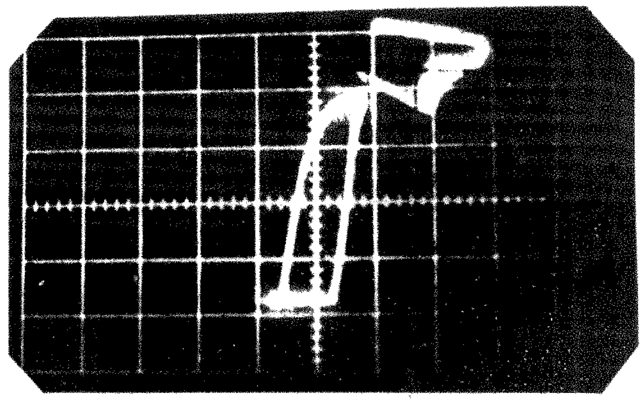
(a) ROOM TEMP



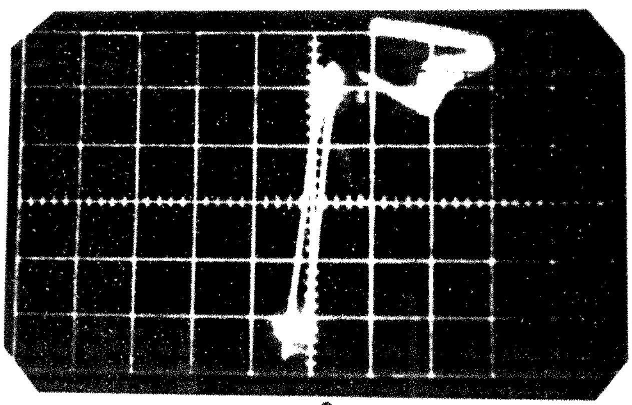
(b) 180 °C



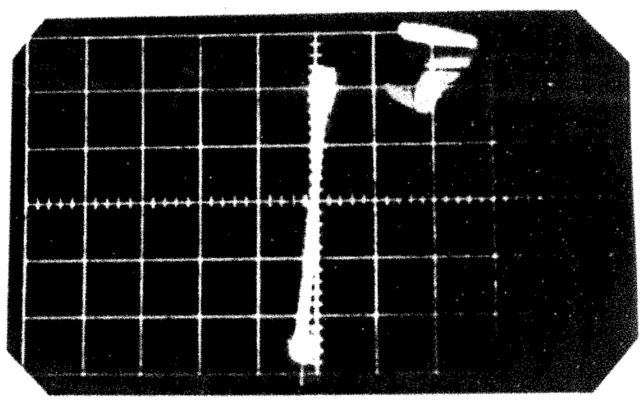
(C) 220 °C



(d) 270 °C

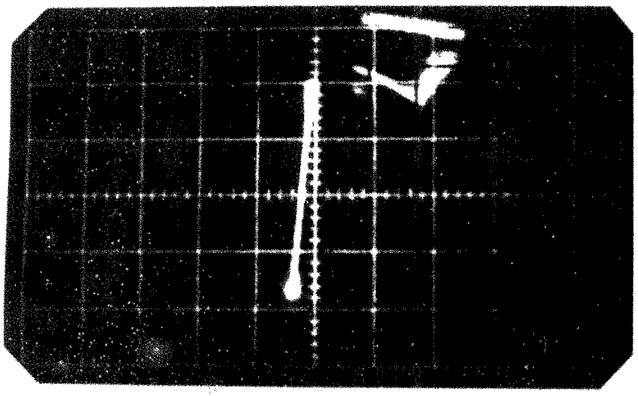


(e) 295 °C

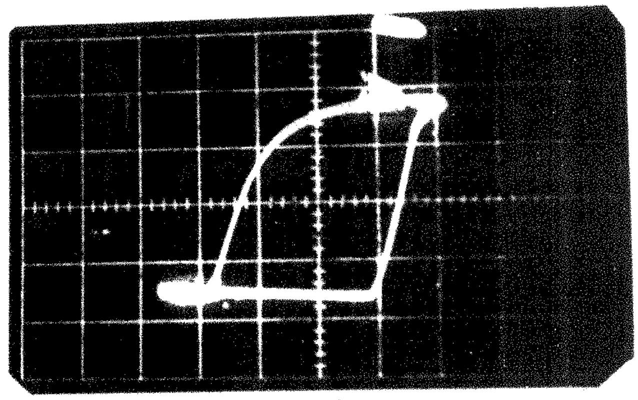


(f) 310 °C

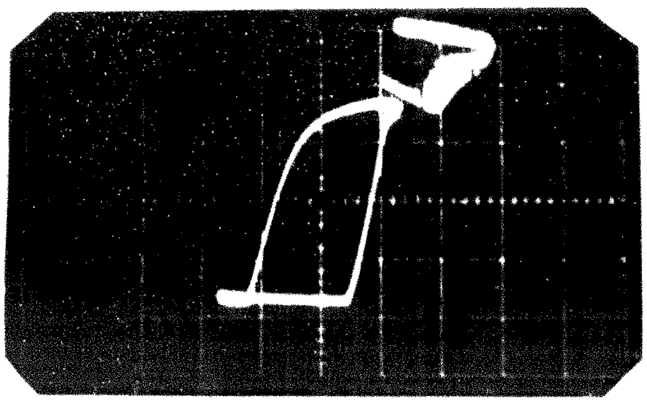
Fig 3·6 — Ferroelectric hysteresis loop of $NaVO_3$ + 0.1 mol % Nd_2O_3.



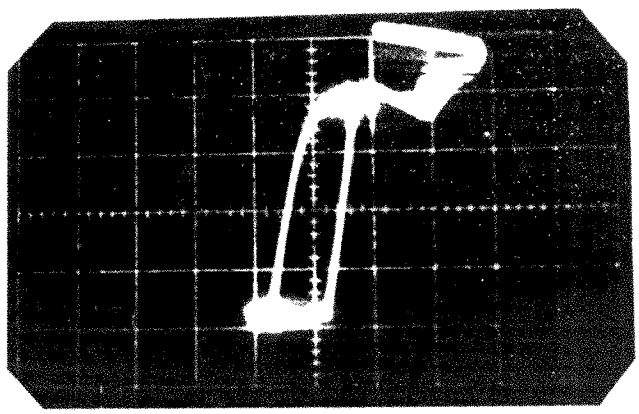
(a) ROOM TEMP



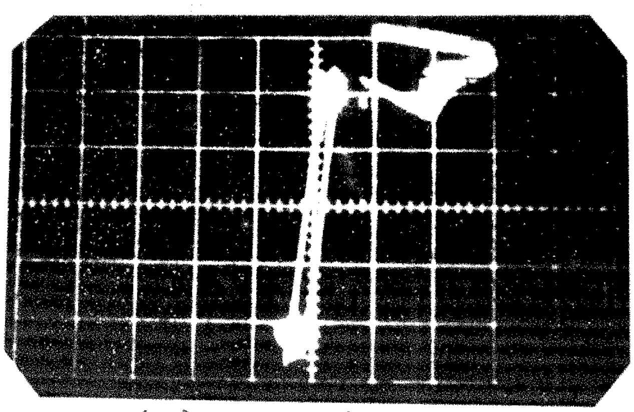
(b) 150 °C



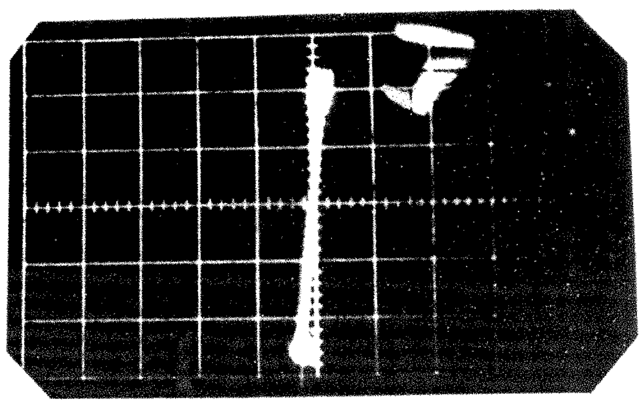
(c) 200 °C



(d) 210 °C



(e) 230 °C



(f) 300 °C

Fig 3·7 — Ferroelectric hysteresis loop of $NaVO_3$ + 0·5 mol % Nd_2O_3.

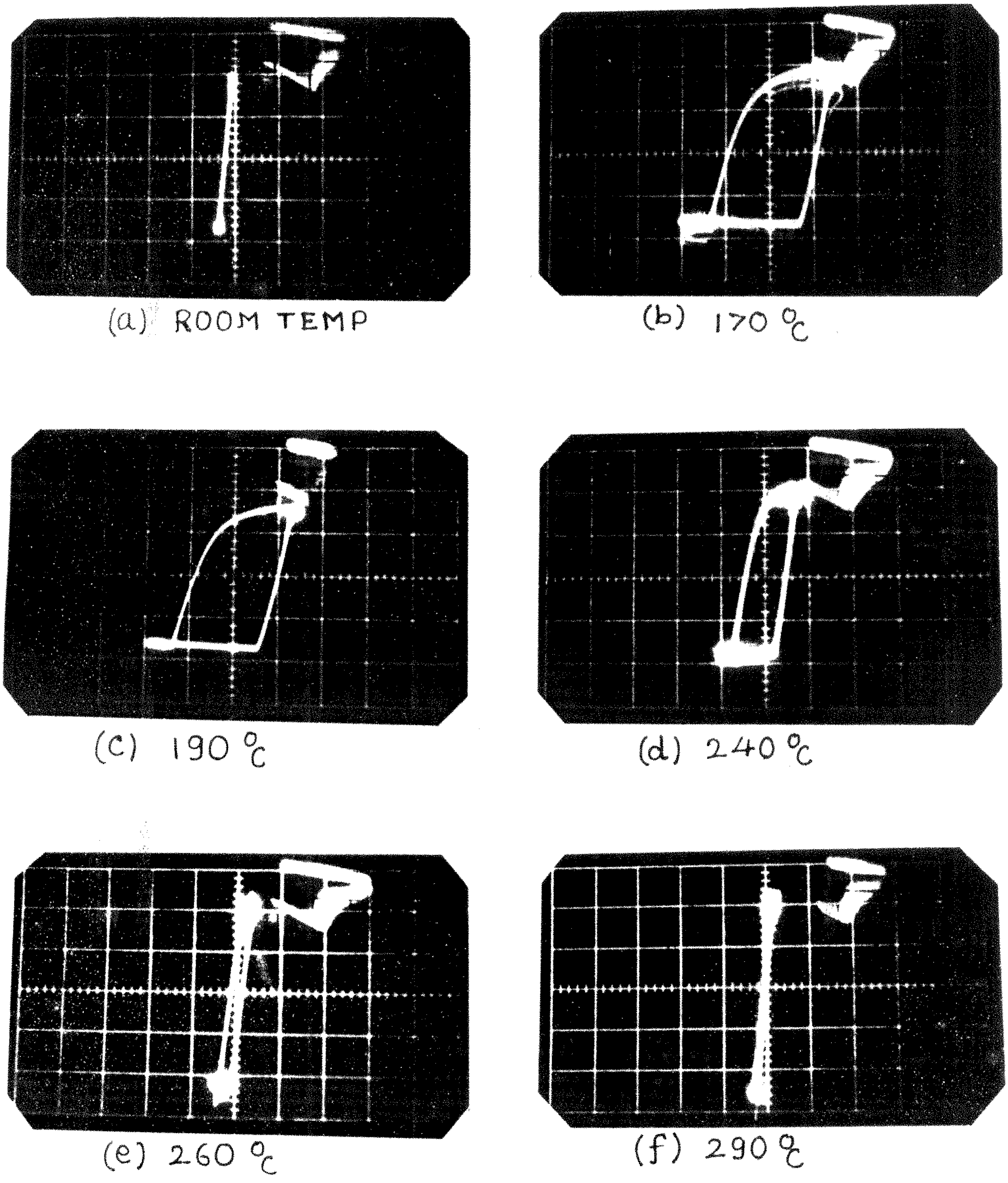


Fig 3·8 – Ferroelectric hysteresis loop of $NaVO_3$ + 1 mol % Nd_2O_3.

Chapter - I]. In order to annihilate the overall polarization of the crystal, it is necessary to apply an electric field in the opposite (negative) direction. The value of the field required to reduce P to zero (OD) is called the coercive field E_C. It is identical with threshold field at which almost all the domain walls can begin to move. For ferroelectric substances, on the contrary the threshold field is fairly lower than the coercive field. Therefore, for ferroelectric substances the coercive field is not determined by threshold field, but will be determined by the characteristics of the movement of domain wall under external field.

The coercive field can be represented in terms of the movement of domain walls. An example of $BaTiO_3$ can explain the above statements. $BaTiO_3$ single crystal has only 180° domains. The coercive field depends upon applied field, thickness of crystal and temperature. Furthermore, it has been found that the imperfections affect the hysteresis loop, and double loop may be observed, if the crystal is heavily stressed. These effects have been used in interpreting the various features of the damage in crystals such as "Rochelle Salt" and triglycine sulfate caused γ-ray irradiation as shown by Chynoweth (1959).

3.7 <u>Theoretical Expression For Coercive Field</u> :

Theoretical expression for coercive field can be obtained by considering the following assumptions :

(1) The growth of the 180^{0} domain is mainly caused by the forward growth of domain i.e. the growth in the direction of applied field. The direct observation of the forward growth by Merz (1954) leads to this assumption.

(2) The switching time of the domain is determined mainly by velocity of the forward growth.

(3) All domains have nearly the same magnitude of polarization and behave similarly in the applied external field.

By considering the above assumptions, the following relation can be obtained,

$$d_o = \int_0^{t_s} V_c \, dt = \int_0^{E_c} V_c \frac{dt}{dE} \cdot dE \qquad (3.1)$$

where d_o --- the thickness of the sample

t_s --- the switching time

E_c --- the coercive field

V_c --- Velocity of the forward growth of the domain.

For the sinusoidal wave E is related with t by the relation

$$E = E_o \ \sin wt \qquad (3.2)$$

where E_o is the amplitude of the applied field and W is the angular frequency. Then using the relation

$$\frac{dE}{dt} = E_o\, W \sqrt{1-(E/E_o)^2} \qquad (3.3)$$

equation (3.1) becomes

$$d_o\, E_o\, W = \int_0^{E_c} V_c \frac{dE}{\sqrt{1-(E/E_o)^2}} \qquad (3.4)$$

If the dependence of V_c on E is known in the above equation then coercive field can be calculated.

3.8 Coercive Field For Undoped $NaVO_3$ and Doped With Different Concentrations (0.025 to 1 mol%) of Nd_2O_3 :

The temperature dependence of coercive field for undoped $NaVO_3$ and doped with different concentrations (0.025, 0.05, 0.1, 0.5 and 1 mol%) of Nd_2O_3 is shown in figure (3.9). From this figure, it is clear that the coercive field strongly depends upon the temperature. It decreases with increasing temperature and vanishes at certain temperature, indicating the transition temperature of ferroelectric material. It is also observed from fig (3.9) that the coercive field depends upon doping concentrations. The maximum value of coercive field for sample containing 0.5 mol% Nd_2O_3 is 700 volt/cm. Table (3.1) shows maximum values of coercive field of $NaVO_3$.

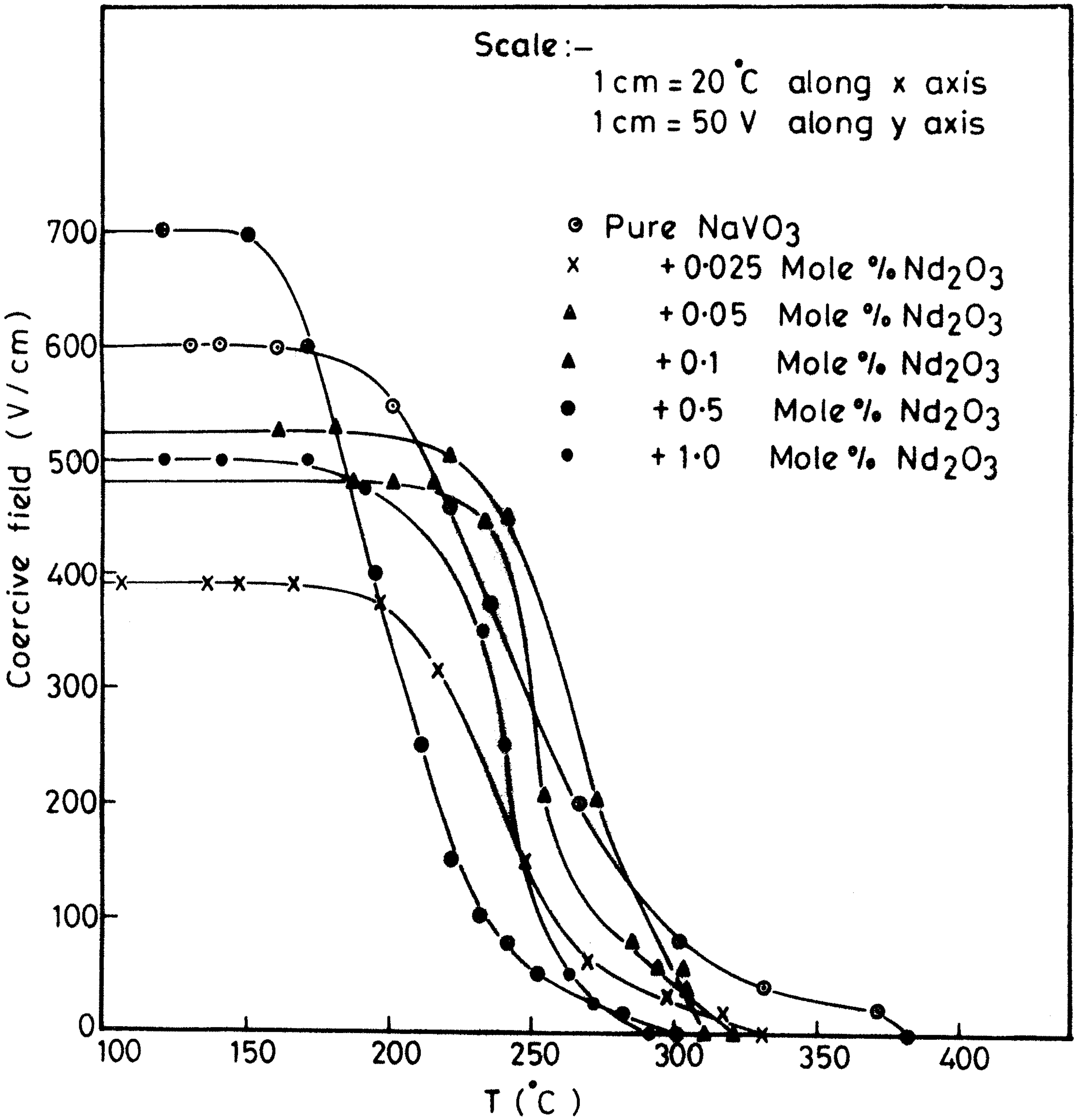


Figure 3·9 – Variation of coercive field with temperature for different concentrations.

Table (3.1) Maximum values of coercive field and densities of $NaVO_3$:

Nd_2O_3 content (mol %)	Coercive field (V/cm)	Density (g/cm^3)
0	600	2.78
0.025	390	2.61
0.05	481	2.65
0.1	525	2.69
0.5	700	2.75
1	500	2.68

3.9 Results and discussion :

The fig (3.9) reveals that the coercive field depends upon the temperature. It decreases with increasing temperature and vanishes at certain temperature, indicating the curie temperature of ferroelectric material. The curie temperature investigated by hysteresis loop method of $NaVO_3$ is found to be 380°C, in good agreement with those reported by Sawada and Nomura (1951), Chavan and Suryavanshi (1985) and Patil et al (1988). The addition of Nd_2O_3 to $NaVO_3$ shows change in the curie temperature.

It is also observed from fig (3.9) that the coercive field depends upon doping concentrations. The peak values of coercive field for samples containing 0.025, 0.05, 0.1, 0.5 mol% Nd_2O_3 increase with respect to undoped ceramics $NaVO_3$.

It decreases for the sample containing 1 mol% of Nd_2O_3. The maximum values of coercive field of $NaVO_3$ doped with Nd_2O_3 are summarized in table (3.1). Table (3.1) reveals that the coercive field is maximum at 0.5 mol% . The effect of Nd_2O_3 doping on the dielectric hysteresis and coercive field of $NaVO_3$ ceramic indicates that the high coercive field of $NaVO_3$ containing 0.5 mol% Nd_2O_3 was attributed to a rather more solid state interaction that takes place in the materials. This can be studied from the pronounced increase of density with addition of Nd_2O_3. The maximum densification is observed at 0.5 mol% doping of Nd_2O_3. Thus in this investigation the dielectric saturation states are attained at 0.5 mol% Nd_2O_3 addition, which may represent the solubility limit of Nd_2O_3 in $NaVO_3$ lattices.

From the experimental observations the following conclusions could be noted (i) the curie temperature of $NaVO_3$ changes for various doping concentrations of Nd_2O_3. (ii) the coercive field depends upon the temperature, it vanishes at temperature 380°C for $NaVO_3$, indicating its transition temperature. (iii) the coercive field increases with increasing doping concentrations upto 0.5 mol% and then decreases for higher doping concentration (1 mol%).

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