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Dielectric hysteresis of undoped and dysprosium doped ferroelectric potassium vanadate

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Abstract. Dielectric hysteresis properties of undoped and dysprosium-doped potassium vanadate have been studied in the temperature range covering their transition points. The coercive field of these materials was measured by the hysteresis loop method. It is observed that the coercive field of KVO_3 doped with Dy_2O_3 at different concentrations (0 to 3 mol%) is remarkably dependent on doping concentration. It is also seen that undoped KVO_3 shows ferroelectric behaviour up to 320°C while Dy_2O_3-doped KVO_3 samples show ferroelectric behaviour up to 380°C for all concentrations.

Keywords. Ferroelectricity; hysteresis doping; coercive field; phase transition.

1. Introduction

Dielectric hysteresis is an essential feature of ferroelectric materials, and one of the important parameters defining hysteresis is the coercive field, which is the most sensitive property of ferroelectric materials. Wieder (1955) has defined coercivity as the magnitude of the field necessary for switching of the polarization along the major hysteresis loop. The theory of the coercive field of $BaTiO_3$ was reported by Janovec (1958). Sawada *et al* (1961) discussed the temperature and amplitude dependence of the coercive field for KNO_3 crystals. Bertaut and Lissalde (1967) measured the hysteresis loops in ferroelectrics by X-ray intensities and true coercive fields in yttrium and rare earth manganates. A new family of oxide ferroelectrics, RVO_4 [R = Nd, Eu, Gd, Tb, Dy, Ho, Er, Yb, Lu and Sc], was discovered by Ismailzade *et al* (1981). Glazer *et al* (1984) described a new type of ferroelectric loop tracer which allows continuous compensation for stray capacitances and resistive losses of the sample. The pyroelectric and dielectric properties of ferroelectric $NaVO_3$, KVO_3 and their solid solutions were investigated by Patil *et al* (1988).

The effect of Dy-doping on the properties of $BaTiO_3$ ceramics were investigated by Yamaji *et al* (1977). The dependence of permittivity of Gd-doped $BaTiO_3$ on doping concentration was reported by Issa *et al* (1984). In recent years considerable advances have been made in investigations on various properties of ferroelectric $NaVO_3$ and KVO_3, but the effect of impurities on these properties have practically not been studied thus far.

The objective of the present paper is to report the behaviour of the dielectric hysteresis of undoped KVO_3 and that doped with different concentrations of Dy_2O_3 (0·025 to 3 mol%) and to study the variation of the coercive field with temperature and doping concentration for these ceramic materials.

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2. Experimental

The polycrystalline solid of potassium vanadate was prepared from a stoichiometric mixture of K_2CO_3 of purity 99% (K Chem. Chemicals) and V_2O_5 of purity >99% [Fluka A G, Switzerland], according to the reaction

$$K_2CO_3 + V_2O_5 = 2KVO_3 + CO_2\uparrow.$$

The mixture was slowly heated in a platinum crucible inside a globar furnace up to 750°C for 5 h and then allowed to cool up to room temperature. The Dy additive used was Dy_2O_3 of 99·9% purity (John Baker Inc., Colorado, USA). The samples were prepared by weighing Dy_2O_3 in different quantities ranging from 0·025 to 3 mol% Dy_2O_3 in KVO_3. Every batch was dry-mixed and then mixed wet with ethyl alcohol in an agate mortar. After complete evaporation of alcohol, the batches were calcined in a platinum crucible at 750°C for 5 h inside a globar furnace and then allowed to cool to room temperature. The samples formed were confirmed by scanning them on an X-ray diffractometer. Pellets of the samples in the form of a disc (1 cm dia and about 0·1 cm thick) were pressed at 6 t pressure. These pellets possessed about 90% of the theoretical density and were further sintered on platinum foil at 500°C for 3 h in a furnace. The two faces of the pellet were well-polished and coated with a very thin layer of silver paste in order to achieve good electrical contact with the electrodes.

The experimental set-up consists of a digital micro-voltmeter and a furnace. The well-known circuit of Sawyer and Tower (1930) was used for obtaining the dynamic hysteresis loop. Pellets of KVO_3 doped with different concentrations of Dy_2O_3 (0, 0·025, 0·05, 0·1, 0·5, 1 and 3 mol%) were placed in a sample holder fabricated in our laboratory and were heated slowly inside a furnace. A dielectric hysteresis loop was observed on the screen of an oscilloscope by using the 50 Hz a.c. electric field for a field strength of 1 Kv/cm. The half-width of the hysteresis loop is taken as the coercive field at various temperatures, by calibrating the screen of the oscilloscope.

3. Results and discussion

The ferroelectric coercive field (Ec) as a function of temperature for KVO_3 doped with different concentrations of Dy_2O_3 is shown in figure 1. As seen from figure 1, the coercive field (Ec) strongly depends upon the temperature as well as on the doping concentration of the material. Initially the coercive field remains constant in a wide range of temperature and then decreases with further increase of temperature and vanishes at a certain temperature indicating the Curie temperature Tc of the material. Figure 1 also shows that the Curie temperature of undoped-KVO_3 as observed by the hysteresis loop method was 320°C which is in good agreement with the Curie temperature reported by Chavan and Suryavanshi (1985) and Patil *et al* (1988b), while the Curie temperature of the Dy_2O_3-doped KVO_3 ceramic material for all doping concentrations (0·025 to 3 mol%) was 380°C. Peak values of coercive field investigated for undoped KVO_3 and that doped with Dy_2O_3 are summarized in table 1.

Table 1 reveals that the peak value of the coercive field is maximum for 0·025 mol% doping concentration of Dy_2O_3. As doping concentration increases from 0·025 to 3 mol%, the peak value of coercive field decreases.

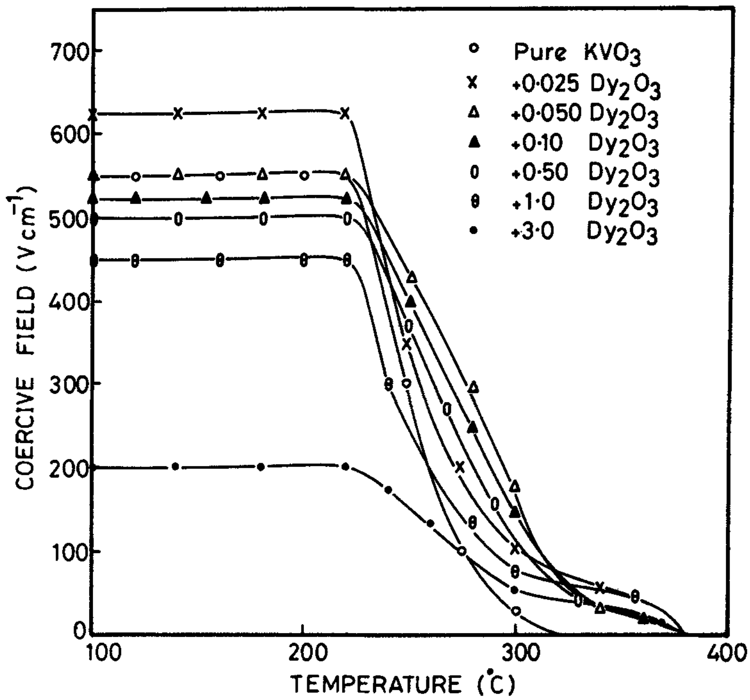

Figure 1. Variation of coercive field with temperature measured at a field of 1 Kv/cm at 50 Hz.

Table 1. Peak values of coercive field of KVO_3 with Dy_2O_3 concentration.

Dy_2O_3 content (mol%)	Peak value of coercive field (V/cm)
0	550
0·025	625
0·050	550
0·10	525
0·50	500
1·0	450
3·0	200

4. Conclusions

(i) Undoped KVO_3 ceramic material shows ferroelectric behaviour up to 320°C, while Dy-doped KVO_3 samples show ferroelectric behaviour up to 380°C for all concentrations (0·025 to 3 mol%).

(ii) The coercive field of undoped KVO_3 and that doped with Dy_2O_3 strongly depends upon temperature.

(iii) Peak value of the coercive field varies with doping concentrations of Dy_2O_3 in KVO_3. It is maximum for 0·025 mol% doping concentration and then decreases with further increase of doping concentration up to 3 mol%.

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