Proceedings of the 17th International Conference on Force, Mass, Torque and Pressure Measurements,
IMEKO TC3, 17-21 Sept. 2001, Istanbul, Turkey

A Novel Hydraulic Pressure (Capacitive) Gauge

Vinay Kumar*, Kamlesh K. Jain* and Subhash C. Kashyap**
** Force Standards, National Physical Laboratory, New Delhi (India)*
*** Department of Physics, Indian Institute of Technology Delhi, New Delhi (India)*

Abstract

The dielectric behaviour of a composite of lead magnesium niobate, lead titanate and lead magnesium tungstate ferroelectric, having different compositions, has been investigated as a function of pressure and temperature. This study reveals that this material can be used as a capacitive pressure gauge in the range 0.1 to 415 MPa to measure the pressure within full scale accuracy of ±0.1% or better.

1. Introduction

The secondary/transfer pressure gauges are those instruments, which have to be calibrated against a primary standard. In these pressure gauges, the variation of some physical property of any material with the applied hydrostatic pressure is a measure of pressure. A number of physical properties such as resistance, capacitance, refractive index and frequency [1] are employed for pressure gauging. Only manganin wire gauges (pressure coefficient and temperature coefficients of resistance are 2×10^{-11} /Pa and 5×10^{-4} /K respectively) [2] and strain gauges (having 350 Ω bridge resistance with an output of 1.4 mV/V at pressure value of 700 MPa)[3] based on variation of resistance, are commercially available. Besides their limited accuracy of ± 0.1 % in the measured pressure, zero shift, hysteresis, temperature sensitiveness, and low-pressure cutoff are some of the other disadvantages which restrict their use as a useful transfer pressure standard (for high pressures). In view of this a need was felt for a device with improved measurement capabilities.

Attempts made in the past to use some of ionic crystals [4] as a capacitive pressure gauge, revealed that these ionic crystals served more like a temperature sensor than the pressure sensor due to its low pressure- (3.8×10^{-11} /Pa) and high temperature- (2.5×10^{-4} /K) coefficients of capacitance. Further, detailed studies carried out at NIST, USA on a number of crystalline and poly-crystalline materials [5-6] led to the identification of bismuth germanium oxide (BGO) and arsenic trisulphide (As_2S_3), to be used as a capacitive

pressure gauge. Though these studies have demonstrated the possibility of using this combination as a practical pressure gauge, long relaxation time and stringent temperature controlled condition were the main drawbacks. Recently, dielectric studies carried out on BGO single crystals [7] gave significantly improved value of pressure coefficient (1×10^{-10} /Pa) and temperature coefficient (6×10^{-5} /K) of capacitance. Even though these studies did not lead to a satisfactory material, the possibility of a capacitive pressure gauge, in principle, was demonstrated.

Due to the limitations of tailor making the materials in single crystal form, the interest has been shifted to polycrystalline materials, mainly composite ferroelectric relaxors, wherein one can obtain desired dielectric properties by changing either the constituents or their concentration. Further, ferroelectric relaxors have an added advantage of having high dielectric constant, relatively low firing temperature with almost negligible thermal expansion and low aging effect. It is only recently [8-9] that the present authors demonstrated the feasibility of using ferroelectric materials for pressure gauging. A few reports [10-12] are available in the literature describing the effect of pressure on the relative dielectric constant of PMN (lead magnesium niobate), PMW (lead magnesium tungstate) and PFN (lead iron niobate) etc., however, no information is available regarding their use for pressure gauging. Keeping in view that PMN has high dielectric constant (≈ 10000 at the Curie temperature), PT (lead titanate) can shift the Curie temperature of PMN from room- to a higher- temperature [13] and the addition of the PMW helps in lowering the temperature coefficient of capacitance, a systematic study of variation of relative dielectric constant of several specimens of PMN-PT- PMW composite both with pressure and temperature, is carried out and the results are reported here.

2. Experimental

Specimens with varying composition in the system - (0.7– X)PMN – (X)PT – (0.3)PMW – where X = 0.1, 0.27 and 0.35 were prepared with excess WO_3 using high purity oxides (PbO-99.9%, Fe_2O_3-99.9%, Nb_2O_5-99.9%, TiO_2-99.9%, WO_3-99.99% and MgO-97%) by a two-step calcination process described elsewhere [8]. The diameter of the pellets was 18 mm and thickness was approximately 1.6 mm. All of the sintered pellets were polished, and guarded silver electrodes were deposited by vacuum evaporation, using a special mask. Specimens of different compositions were prepared in different batches to see the effect of processing parameters and to investigate their reproducibility.

To measure the variation of capacitance both with pressure and temperature, a specimen was placed in a specially designed specimen holder [7] made of invar. The specimen holder was placed in a high-pressure vessel of margering steel. A conventional high hydrostatic pressure system was used to generate the pressure using ethyl hexyl sebacate as pressure transmitting fluid and it

does not react with the specimen as the reported observations taken on the same day and on different days on the same specimen are within the experimental deviations. Pressure was measured by an online piston gauge pressure standard (Desgranges and Haut, France) with an accuracy of ± 0.01%. During measurements, the pressure was stable (within ± 0.03 MPa). Temperature of the specimen was controlled using a temperature bath (Model RTD 8dd Nes Lab, USA) capable of maintaining the temperature within ± 0.05 K. The temperature of the specimen was measured with a Chromel – Alumel thermocouple and a digital temperature indicator of resolution 0.01K. The capacitance was measured at 1 kHz with an auto digital capacitance bridge (Andeen- Hagerling, Model 2500A, USA) with an accuracy of 0.1ppm in the capacitance range used here. For estimating the hysteresis, observations were made over a complete pressure cycle, i.e. by increasing the pressure in steps from atmospheric pressure to 415 MPa and then bringing the pressure down to atmospheric pressure in similar steps.

3. Results and Discussions

The dielectric constant of PMN-PT-PMW composite of three different compositions has been calculated from the observations made over the entire pressure cycle and at a fixed temperature. The Ko is the dielectric constant at atmospheric pressure and K is the dielectric constant at any pressure. Figure 1 shows the variation of relative dielectric constant (K/Ko) as a function of pressure at 301 K of all the three specimens. Fig 2 shows the variation of the relative dielectric constant as a function of the temperature at constant applied pressure (0.1 MPa). At each pressure point at least three observations were made and it is the mean of the three observations presented in these figures. Consequently, fifteen observations were made in one complete test.

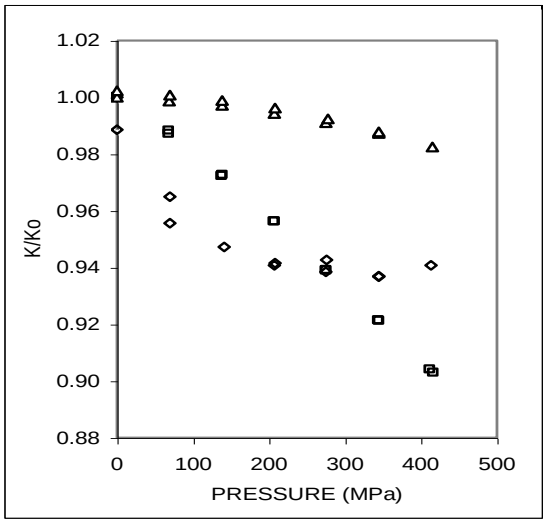

Fig 1. Variation of Relative dielectric constant (K/Ko) with pressure of (0.7-X)PMN-(X)PT-(0.3)PMW specimens(◇) X=0.1 (□) X= 0.27 (Δ) X=0 .35

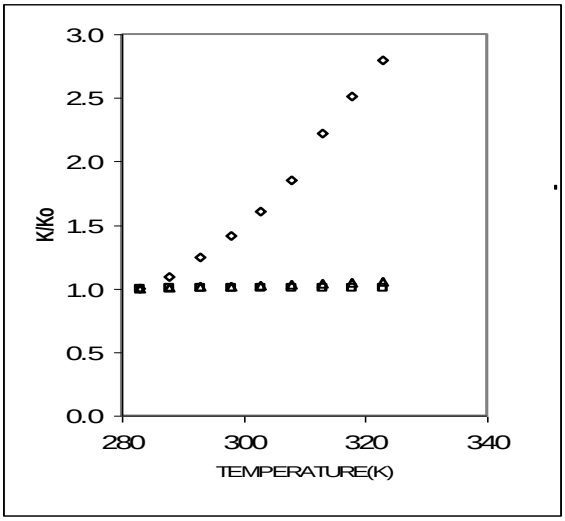

Fig 2. Variation of Relative dielectric constant (K/Ko) with temperature of (0.7-X)PMN-(X)PT-(0.3)PMW specimens (◇) X=0.1 (□) X= 0.27 (Δ) X= 0.35

It is observed that the relative dielectric constant for the specimen of composition X = 0.27 decreases almost linearly and most rapidly with the increase of applied pressure (Fig1) whereas its value remains nearly unchanged with the increase of temperature (Fig2). The values of the pressure- and temperature-coefficients of dielectric constant, as calculated from these curves, are nearly -2.36×10^{-10} /Pa and 1.5×10^{-4} /K, respectively. Figure 3 shows the variation of relative dielectric constant for specimens of X= 0.27 (of batch A) as a function of applied pressure.

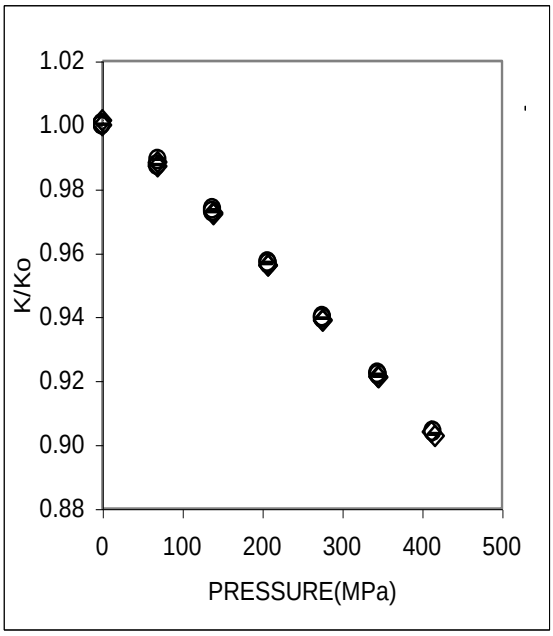

Figure 3. Variation of relative dielectric constant (K/Ko) with pressure for specimen of batch A in three different cycle. (◇) Ist cycle, (-) IInd cycle and (O) IIIrd cycle

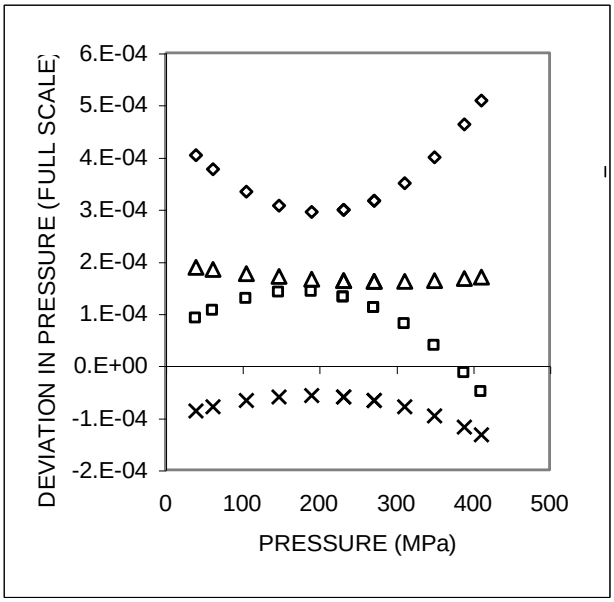

Figure 4. Deviation (full scale) in the pressure calculated from the master equation and calibration equations for specimens of different batch (◇) batch A, (□) batch B, (Δ) batch C and (×) batch D

The best least square fit using all the observations of Figure 3 was obtained as a second order interpolation equation (pressure p is expressed in Megapascal),

$$p = 438.9029 + 3651.87 \times X - 4073.827 \times X^2$$
$$\sigma = 0.278 \text{ MPa} \quad (1)$$

The best least square fit of all the observations made on the four specimens from four different batches is obtained as the master equation,

$$p = 467.919 + 3591.489 \times X - 4042.353 \times X^2$$
$$\sigma = 0.18 \text{ MPa} \quad (2)$$

where, $X = K / K_o$, is the output of capacitive pressure gauge, and p is in the Megapascal.

To investigate the consistency between the pressures calculated from the calibration

equations obtained for the specimens of different (A,B,C&D) batches (similar to equations 1, that is for specimen of batch A) and master equation (equation 2), the deviation in the pressure values is plotted as a function of the nominal pressure, in Figure 4. It is seen that the maximum deviation at each measured pressure is 6×10^{-4}. Considering the contribution due to its hysteresis, zero shift and reproducibility in individual and in combined pressure cycles of any specimen belonging to the same batch or to different batches, the agreement is excellent

In order to see short term reproducibility, three pressure cycles were taken on the same day with a specimen of same batch or with three specimens of different batch. The short-term reproducibility in all the pressure values calculated either for the same specimen or for the specimens of different batches ranges from 0.69×10^{-6} to 9.08×10^{-6} during increasing pressure and from 1.73×10^{-6} to 5.34×10^{-6} while decreasing the pressure. However, an improved reproducibility is obtained in any one individual pressure cycle, which is always less than 0.47×10^{-6} irrespective of whether the pressure is increased or decreased. Though the hysteresis has been observed, its magnitude is very small, vary from 1.28×10^{-6} to 6.36×10^{-6} in all three combined pressure cycles and even smaller 0.75×10^{-6} to 1.2×10^{-6} in any individual pressure cycle.

In different pressure cycle the shift of zero, was always less than ± 0.05 pF, corresponding to ±0.23 MPa. It may be noted that this value (±0.23 MPa) is well below 0.06% of the full scale of the capacitive gauge and as well as the expected uncertainty of the capacitive gauge. The present study shows that the ternary system 0.43PMW-0.27PT-0.3PMN with excess WO_3 posseses, high pressure- and low temperature- coefficients of dielectric constant along with a negligible hysteresis.

4. Conclusions

A reliable, reproducible and accurate capacitive pressure gauge to measure pressures with accuracy better than ±0.1% (full scale) is demonstrated. It is encouraging to note that the over all level of disagreement between the different specimen of the same batch or specimens prepared in different batches is significantly lower than the estimated total uncertainty of the capacitive gauge.

Acknowledgement

The authors would like to sincerely thank Dr. Krishan Lal, Director, NPL, New Delhi for permission to publish the work. We are also thankful to Dr. R.N. Pal for his interest in this work.

5. References

[1]. Peggs G.N., *Secondary- standard gauges for the accurate pressure measurement up to 1 GPa*, High Temp - High Press., 1980,**12**, p. 1-10

[2]. Molinar G. F., Bianchi L., *Metrological characterization of manganin gauge calibrated against the mercury melting*

line, IX AIRPART Conference , Albany, NY, July 1983

[3]. Birks A.W., *Strain gauge pressure transducer*, Internal Report No. 1566 Queen's University of Belfast, 1981

[4]. Andeen C., Fontanella J., Schuele D., *A capacitive gauge for the accurate measurement of high pressure*, Rev. Sci. Instru., 1971, **42**, p. 495-496.

[5]. Bean V. E., *Transducer for very high pressure*, Proceeding of the 2nd International conference on high pressure engineering,UK, 1975 p. 29-31.

[6]. Colwell J., Tilford C. R., *Capacitive Pressure Gauge*, NBS Internal Report, (1981).

[7]. Jain K.K., Kashyap S.C., *Dielectric properties of BGO single crystal under high hydrostatic pressure*, High Temperatures –High pressures, 1995/1996**, 27/28**, p. 371-376.

[8]. Radhika Rao M.V., Umerji A.M., Kumar Y, Jain K. K., Gopal E.S. R., *Effect of high pressure on the dielectric properties of relaxor compositions PFN-PZN-BT*, J. Mater. Sci. Letter, 1997, **16**, p.122-125.

[9]. Kumar Y., Kumar V., Jain K. K., Kashyap S. C., *A capacitive pressure gauge reliable transfer pressure standard*, Sensors and Actuators **B 55**, 1999, 217-221.

[10]. Fristberg V.Ya., Fristberg P. A., *Dielectric properties lead magnesium niobate under high pressure near phase transition*, Sov. Phys. Crystallogrpy, 1979, **24(4)**, p. 492-493

[11]. Yasuda N., Fujimoto S., Yoshimura T., *Pressure and temperature dependences of dielectric properties of PMW*, J. Phys. C: Solid State Physics, 1986, **19**, p. 1055-1063.

[12]. Yasuda N., and Ueda Y., *Temperature and pressure dependences of dielectric properties of PFN with diffuse phase transition*, J. Phys.: Condens. Matter, 1989, **1**, p. 5179-5185.

[13]. Kuwabara M., Takahashi S., Goda K., Oshima K., Watanabe K., *Continuing in phase transition behavior between normal and diffuse phase transition in complex perovskite composition*, Jpn. J. Appl. Phys., 1992, **31(9B)**, p. 3241-3244.

Contact Person for Paper:

Kamlesh K. Jain

Force Standards, National Physical Laboratory, New Delhi-110012 (India)

E-mail:kkjain4@yahoo.com