Retraction

A miniature capacitance dilatometer for magnetostriction and thermal expansion measurements

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Abstract. In the present article we want to report about our miniature capacitance dilatometer developed for magnetostriction and thermal expansion measurements. The dilatometer is foreseen to be used on millimeter-sized single crystalline samples in the temperature range 2 - 320K. The dilatometer main body is produced of brass and is mounted in low vacuum. The capacitance was measured with a digital, self-balancing, three terminal, commercial capacitance bridge operating in the frequency range 50 Hz - 20 kHz. The temperature was determined using commercial resistive thermometers Cernox BG 1030 and Cryo-con Model 44 Temperature controller.

1. Introduction
To understand the lattice dynamics of the solids, where the motion of atoms always responds to cooling or heating in a way that is consistent with the symmetry of the space group to which they belong, is one of the basic aims of solid state physics. When the atoms move, the electronic structure of the solid changes, leading to changes in different physical properties, such as elastic coupling, thermal expansion (TE), specific heat. The coupling between the magnetic and elastic regimes in a magnetostrictive material is known as Joule magnetostriction (MS). The MS is a property of the ferromagnetic materials and causes them to change shape when subjected to a magnetic field. Knowing the TE of the solids we can obtain useful information about their electronic and photonic properties, their specific heat (Gruniesen formalism), propose a thermodynamic treatment of their phase transitions (Ehrenfest relations) etc. The idea for using capacitor as a detection device was developed in 1920 by R. Whiddington [1]. His ultramicro-meter was the first capacitance device used to measure very small distances changes. In 1961 White [2] has developed his capacitance dilatometer. White’s design principles of absolute and relative dilatometers were adopted and improved by number of different authors. For MS measurements, initially the strain gauges method [3] was predominately used. Green, Chandrasekhar [4], Fawcett [10] were among the first, that used capacitance dilatometer for MS measurements. Based on this we decided to concentrate our efforts to develop CD for both TE and MS measurements.
2. Apparatus

A schematic draw of our capacitance dilatometer is shown on figure 1. The measuring cell is made from brass. In order to reduce the internal strains from the machining process all its parts were cleaned with 10\% dilute nitric acid and annealed at 200°C for about 6 h in nitrogen atmosphere.

2.1. Thermometry and temperature controller

The CD is foreseen for usage with MagLab Exa system fitted with a 9T SC magnet and is capable of measurements in the range 2-320 K. In order to ensure highest thermal stability of the measuring cell, the CD is placed in a closed stainless steel tube, evacuated to rotary pressure of about 10⁻³ Torr and then flooded with ⁴He exchange gas to a pressure of about 1 Torr. So far the thermal equilibrium and not the stability of the capacitance measuring circuit limits the resolution of the method, the most important issue is to ensure the thermal equilibrium of the whole dilatometer. For example at room temperature a temperature difference of 0.01 K causes a length change of several nm of the cell, assuming the effective length of the cell ≈ 40 mm and typical samples length of 3-5 mm we have an inaccuracy of α of about 10\%. To obtain an accuracy of 1\% we had to stabilize the temperature of the dilatometer to within 1 mK at 300 K. To measure the sample temperature we use a Cernox BG 1030 thin film resistance sensor. The sensor tightly fitted with Apiezon N grease on the upper surface of the fixed capacitor plate (3). For temperature reading and heater control we use a Cryo-con Model 44 Temperature controller.

2.2. Capacitance system

The CD represents cylindrical cell containing a flat capacitor, build by two parallel plates surrounded by three frame rings (FR), used as electrical shielding and construction frame. The upper capacitor plate (3) is fixed to the upper FR (2) with Stycast. The gluing process includes 24 h holding at room temperature followed by 24 h at about 80°C. To ensure that ring and plate are coaxial 0.2 mm thick fishing line is slipped into the gap between them. To ensure that their lower surfaces remain also coplanar both parts are placed on a flat glass plate. Three stainless steel screws pass through the connecting flange and upper FR before threading into the middle FR. The second capacitor plate (5) is bolted on the spring (6) using the nut (7). The spring is made of 0.125 mm thick Be-Cu foil and is insulated between two 0.5 mm thick Teflon washers. The spring is positioned between the middle (4) and the lower (8) FRs as shown in figure 1: the lower FR and the spring are bolted with three stainless steel screws into the middle FR. To hold the spring and lower capacitor plate concentric during the mounting we use again fishing line. For the purpose of capacitor gap evacuation and exchange gas circulation the spring has...
six small holes. The distance between the capacitor plates can be adjusted by use of separator washers placed between the upper and middle FRs or varying the thickness of the Teflon washers used with the Be-Cu spring. In order to ensure better sensitivity the initial capacitance has to be ca. 10±15 pF. The capacitance C is measured with a digital, self-balancing, three terminal, commercial Andeen-Hagerling capacitance bridge AH2700A. According to our test results for best performance the bridge has to be operated with frequency between 16 and 2.5 kHz (typical 1 kHz), excitation voltage 15 V rms and average over 5±8 seconds. The connection between the capacitor plates of the dilatometer is performed first through an Unimicrowave Coaxial Cable - Type SS (Lakeshore). Before fixing the capacitor plates to the FRs we have soldered to them short wires (from the coaxial cable center conductor), these wires are used as electrical contact points for the coaxial cables. To ground the coaxial cables shield at the dilatometer body, the shields are soldered to a small copper foil tabs, fixed on top on the axis. The connection to the bridge, outside the cryostat, is performed via double-shielded BNC cables. By careful construction of the capacitance measuring circuit (shielding, avoiding of ground loops etc) the maximum sensitivity could be equal to that of the bridge 1 x 10^-6 pF, which corresponds with C≈ 15 pF to a length change of 0.2 nm.

2.3. Sample adjustment system
The sample adjustment system contains a sample holder screw (11), screwed into lower flange (10). The lower flange (10) is attached to the lower FR (8) using Stycast and fishing line as described above. After the sample and sample holder screw (11) are positioned appropriately they have to be fixed in place with the lock-nut (12). If necessary different sample holder screws with different lengths can be used to accommodate different sample sizes.

3. Test and results
The first test we have performed on our dilatometer was to check its sensitivity and linearity of the response. These tests were performed at room temperature, outside the cryostat. To avoid any kind of external disturbances the capacitance cell was protected by grounded aluminum cover. As test sample we use a cylindrical single polycrystalline Ni 3 mm length and 2 mm diameter. In order to measure the exact angle to capacitance dependence an azimuth disk was mounted on the lower flange and a pointer on the sample holder screw. Rotating in small steps the sample holder screw we have received the capacitance dependence for our dilatometer. The result plotted as ∆C vs θ is shown in figure 4. The capacitance dilatometer the dilution ∆L of a sample of length L manifests as a change in the gap between a pair of capacitor plates. For an ideal parallel-plate capacitor the relationship between the measured capacitance C and d is simply C = ε_0 S/d, where ε_0 = 8.854 19 x 10^-6 F/m is the permittivity of free space and S is the area of the capacitor plates. The dependence between the capacitor gap d and the angle θ is d = d₀ − kθ, where d₀ is the length of unloaded dilatometer and the constant k is related to the thread pitch on the sample holder screw. We can rewrite the equation as C = ε_0 S/d₀ - kθ, thus, a plot of C vs θ should be a straight line whose slope yields the effective area of the capacitor plates. More detailed discussion about the capacitive edge and tilting effects is given in Pott and Schefzyk [7]. In general to determine the temperature dependent coefficient of linear TE or the field dependent MS of a sample we need to know not only the dependence with the sample, but also the dependence with the reference material in use the dilatometer. As discussed above the capacitance of the dilatometer is sensitive to thermal gradients, therefore it is recommended to use reference sample from the same material as the capacitance cell. Then the coefficient of linear TE is \( \lambda = \lambda_{\text{cell+unknown sample}} - \lambda_{\text{cell+ref sample}} + \lambda_{\text{ref sample}} \). As described above our capacitance dilatometer can be used for MS and TE measurements. In general MS measurements have to be done in slow changing magnetic (recommended sweep rate 0.1±0.2 T/min) and at measurements the temperature sweep rate strongly depends from the temperature...
range, starting with 0.1 K/min or less for the range 2-10 K and reaching 0.5 K/min or less for the range 180-330 K. Using our dilatometer with the MagLab Exa system we have to consider that its temperature stabilization is better by warming and as a generally data collected during warming are less noisy than during cooling. In case the temperature/ magnetic field was changed to fast we can observe "tails" (the capacitance continues change after the final temperature/ magnetic field is reached). Sometimes a small "step" can appear in the temperature/ magnetic field dependence. We suppose these peculiarities can be caused by sudden relaxation of small strains in the CD or in the sample. In generally there are no special requirements to the samples shape. Special attention is needed in case of measuring monocrystalline samples with high magnetic anisotropy. Varying the sample orientation with respect to the applied magnetic field can cause a torque if its magnetic moment is not parallel to the field. Any motion of the sample in response to this torque will contribute to the measured capacitance change. According Birss [6] the specimen should have cylindrical symmetry, and measurements must be made as the magnetic field is rotated about the axis of symmetry. Furthermore, the specimen shape should ideally be an ellipsoid. In practice, thin discs are assumed to approximate sufficiently well to the shape of an oblate spheroid. Let us briefly describe the TE and MS results of our high-purity sample of polycrystalline nickel. The TE and MS results are shown in figures 3 and 4 respectively. On figure 3 the data received with our dilatometer is represented by open circles and dashed line which represents the TE values for Ni published in the literature [8, 9]. The average absolute value of the fractional deviation of our nickel measurements from those in the literature is about 1.5% for the whole temperature range 2-330 K. The longitudinal MS of a cylindrical sample of high purity, polycrystalline nickel is shown in figure 4, a pronounced negative Joule MS and a slightly positive forward MS are observed. Both of these features are consistent with published results [10, 11].

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