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Characterisation of glue behaviour under thermal and mechanical stress conditions

Gianluca Caposciutti[1], Bernardo Tellini[1], Alfredo Cigada[2], Stefano Manzoni[2]

[1] *Dip. di Ingegneria dell'Energia, dei Sistemi, del Territorio e delle Costruzioni, Università di Pisa, Largo Lucio Lazzarino, 56125 Pisa, Italy*
[2] *Dip. di Meccanica., Politecnico di Milano, Via La Masa 1, 20156 Milano, Italy*

ABSTRACT
New low-cost measuring devices require that the box housing and electronics have the cost aligned with the sensing system. Nowadays, metallic clips and/or glue are commonly used to fix the electronics to the box, thus providing the same motion of the structure to the sensing element. However, these systems may undergo daily or seasonal thermal cycles, and the combined effect of thermal and mechanical stress can determine significant uncertainties in the measurand evaluation. To study these effects, we prepared some parallel plates capacitors by using glue as a dielectric material. We used different types of fixing and sample assembly to separate the effects of glue softening on the capacitor active area and plates distance. Therefore, we assessed the sample modification by measuring the capacitance variation during controlled temperature cycles. We explored possible non-linear behaviour of the capacitance vs. temperature, and possible effects of thermal cycles on the glue geometry. Further work is still needed to properly assess the nature of this phenomenon and to study the effect of mechanical stress on the sample's capacitance.

Section: RESEARCH PAPER

Keywords: Temperature cycles; glue bonding; mechanical stress; thermal stabilization

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Corresponding author: Gianluca Caposciutti, e-mail: gianluca.caposciutti@ing.unipi.it

1. INTRODUCTION

Attention about Structural Health Monitoring (SHM) systems, to monitor any possible infrastructure damage, is growing a lot these days [1]: unfortunately, the same attention is not being gained by metrology issues, dealing with the quality of measurements and their reliability. New low-cost measurement nodes, monitoring the structure motion, also require that the box housing the electronics and the elements fixing the boxes to the structure have costs aligned with those sensing nodes.

The most common solution today is the use of plastic boxes, granting the required IP protection grade; these are fixed to the concrete structure by means of dowels and/or glue. Also, the boards hosting sensors, microcontrollers and the needed electronics, today designed with a flat bottom to grant the same motion of the box and the sensor, preventing from the dynamic behaviour of the board to affect measurements, are connected to the enclosing box by means of metallic clips or glue. Thus, there is a chain between the structure surface and the sensing element with many interfaces, influencing the metrological performances of the whole system.

The presence of these interfaces can generate significant consequences on the measurements, especially when MEMS clinometers and accelerometers are taken into consideration. Indeed, their working principle is based on the estimation of the angle between the sensing axis and the gravity vector. Being the sensor glued to the enclosing box, when temperature changes (e.g., due to usual environmental thermal shifts), the glue can change its behaviour and geometrical layout. Regarding the latter aspect, this implies that a significant systematic error can affect the readout e.g., by introducing a temperature dependant offset, while this effect must be avoided at best to preserve the quality of the measurement. Such a goal can be achieved by properly choosing the type of glue through a study of its thermo-mechanical behaviour. In this paper, this is accomplished by capacitive measurements on tailored set-ups, as outlined below.

Sensors fixed to a structure for monitoring purposes, for instance on a bridge, can undergo important temperature changes, very high during summer and very low during winter. In order to better define the glue behaviour, a reference to capacitive displacement sensors has been adopted for a thorough understanding of the temperature related phenomena. Capacitive displacement sensors are those exhibiting the highest sensitivity (up to hundreds of Volt per millimetre), so it has been decided to use the glue as a dielectric interposed between two metallic plates: even the smallest change in the plate distance due to temperature can therefore be easily detected.

Capacitance measurements have therefore been carried out during varying temperature cycles, trying to split any change in the glue/dielectric features out of a real change of the plate distance: the latter is the main concern for SHM issues.

According to the theory, the capacitance between two conductors is defined as the ratio between the charge on one conductor and the potential difference between them. Such a parameter depends on the geometry of the conductors, on their relative distances, and on the characteristic of the interposed dielectric medium. Apart from simple geometries and ideal dielectric medium, an accurate calculation and measurement of the capacitance is a difficult task.

For slowly varying fields, the electric permittivity ε is a macroscopic constitutive parameter which relates the macroscopic fields electric flux density $\vec{D}$ and electric field $\vec{E}$ [2], [3]. A comprehensive discussion of the physics of frequency dispersion and of the effective time constants in dielectric media remains a complex issue, as well as a clear operative meaning of the measurement of ε in static conditions [4]. More in general, the properties of the dielectric material can vary as a function of several other parameters.

For instance, temperature, and mechanical stress generally play an important role and should be properly taken into account for a valid description [5], [6]. Regarding the temperature, the number of polarizable ions per unit of volume is a direct consequence of volume expansion, the ions polarizability depends itself on their thermal energy, which also influences the number of defects and disorder in the material, and the effective relaxation time. The total stress on dielectric material also affects its physical properties [2], [7], for which three different contributions are identified: mechanical stress, Maxwell stress, and electrostriction component. In more detail, in absence of electric field, the material (assumed in an elastic regime) finally obeys to Hooke's law [8]. Moreover, it is shown that the Maxwell stress in solid dielectrics, such as many polymers, directly affects the material stress status, and causes molecular deformation, thus modifying the dielectric and electrical properties of the material [9].

The main purpose of the current work is to characterize the effect of the glue bonding between a sensor or its housing, and the target surface. Particular attention is given to the glue thermal behaviour, which may significantly affect the sensor readout due to bonding deformation, e.g., in the case of a tilt or strain-sensitive element. Indeed, the temperature variation can lead to the deformation of the glue, possibly altering the relative distance between the plates and affecting the reference position of a sensor with respect to the base surface. To this aim, the mechanical and thermal variation of the glue is studied by using an electrical model of the system. In a first approximation, the bonding between the sensor or its housing and the target surface can be modelled as a flat plates capacitor, where the glue plays the role of the dielectric medium.

The paper is structured as follows. Section 2 shows the model used in this study, and Section 3 reports the experimental analysis performed. Moreover, Section 4 shows the results obtained with the devices described in Section 3, and finally, some conclusions are drawn in Section 5.

2. MODELLING

Let us assume a capacitor having parallel plane electrodes with surface *S*, separation distance *d*, and dielectric medium with constant permittivity *ε*. According to the theory, for slowly-varying and uniform electric field distribution between the two electrodes, the capacitance *C* between them reads as:

$$C = \varepsilon \frac{S}{d} \tag{1}$$

It is worth to point out that for finite electrodes, the surface charge distribution is indeed not uniform [10], and an accurate estimation of the field distribution is not straightforward due to the fringing field effect and possible divergent field values at the electrode edges. Metrological institutes commonly adopt guard rings as a method for the mitigation of such an effect [11], [12]. An accurate characterization of the dielectric material bases on a high control of the electric field distribution, and, in analogy with the characterization of magnetic properties of a material, we can more properly refer to characterizing the capacitance of the sample [13].

As a consequence, we assume for our analysis the following capacitance model:

$$C(\varepsilon, S, d, f, T) = \varepsilon(f, T) \frac{S(T)}{d(T)}, \tag{2}$$

for which we refer to capacitors made of two parallel metal disks, filled by a glue substance as interposed dielectric material and capacitance *C*. In equation (2), we made explicit for the capacitance parameters the dependence on the frequency *f* and the temperature *T*.

Thus, as mentioned in the Introduction, *ε* generally presents frequency dispersion and depends on the temperature. The frequency dispersion represents a variation of *ε* vs. frequency and, following a classical approach, it is described by spring-mass models. For a review on such a topic, Clausius-Mossotti's, Debye's expressions, and their novel modified versions are recognized models of such a phenomenon. The temperature dependence of the dielectric properties finds a general description for relatively rarefied media, such as the description of the orientation polarization based on Maxwell-Boltzmann statistics. On the other hand, such a relationship can significantly vary depending on the temperature range, structure, and physical status of the material.

A conceptual scheme of the modelled configuration is shown in Figure 1.

The values of *d* and *S* are constant in the ideal configuration. However, these parameters vary with possible material contraction and dilatation because of the temperature variation. Furthermore, glue softening can modify the material structure, influencing its dielectric and mechanical properties (e.g., viscosity reduction when temperature increase). Thus, as made explicit in equation (2), the geometry of the system and the capacitance *C* are expected to vary with the temperature. As a consequence of such behaviours, the capacitor plates can get closer one each other under the effect of their weight and the electrostatic forces, thus reducing their relative distance. Moreover, a viscosity reduction of the glue can let it slide over the plates, thus varying the geometry of the sample. These two main phenomena, together with the proper dielectric material temperature characteristic, generate a complex behaviour of the sample capacitance as a function of the temperature.

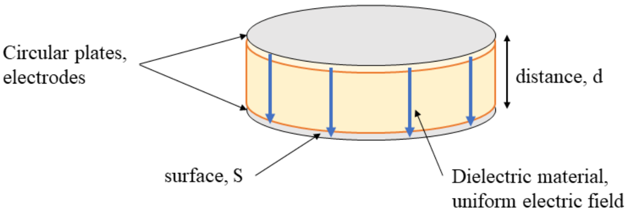


Figure 1. Scheme of the modelled configuration.

As an example for raising the temperature, the glue softening might reduce *d*, determining an increase of *C* according to equation (2). Moreover, the possibility of glue sliding also contributes to the variation of the *C* at high temperatures. After the heating process, the values of *d* and *S* can be eventually stabilized and, in a cooling stage, the behaviour of $C(T)$ might follow a different curve, thus showing a hysteresis behaviour with the temperature. This complex phenomenon is expected to be more evident at high temperatures and it may exhibit some relaxation toward a stable condition after a few thermal cycles.

To carry out a consistent characterization of *C*, we built several capacitor samples with specific features to distinguish the above-mentioned effects. Firstly, the overall characteristic $C(T)$ is evaluated by using a parallel face capacitor with circular electrodes. The geometry of the device is left free to evolve as a function of the temperature. The dielectric glue is positioned in the centre of the plates, far from the edges of electrodes, thus reducing the fringing field effect at the electrode's edge. A second sample is built as the first one, but keeping fixed *d* to a minimum value by using ad-hoc glass spacers. The use of glass spacers with a low thermal dilatation coefficient allows for neglecting the electrode distance variation as a function of the temperature. However, for such a configuration, the glue can still possibly slide through the inter-electrode region, and finally affect the geometry and the active volume of the dielectric medium of the capacitor. A third sample is made with glass spacers, and by covering the border of the electrodes with dielectric glue. In this last case, the glue geometrical variation is expected not to significantly affect the material within the plates, thus further reducing the possible effective variation of both $S(T)$ and $d(T)$. Therefore, temperature cycles are applied to the different samples and the behaviour of their capacitance is assessed at different operating frequencies.

3. EXPERIMENTAL SETUP

Different capacitor samples are built with plain faces configuration, by adopting circular plates with 60 mm diameter and 3 mm thickness made by aluminium. A layer of glue is placed at the centre of the capacitor as the dielectric. Particularly, the glue is placed in a 40 mm internal disk using proper PLA spacers, which also kept the plates at the desired distance d of 1 mm, 1.4 mm, and 2.8 mm, respectively. The central glue positioning allows reduction as much as possible of the edge effect due to the electrode's borders. The distance d of the obtained samples can be further fixed to a minimum extent utilizing some 0.2 mm thickness glass spacers, placed at about 120° angular distance around the capacitor. The spacer's material is chosen to present a negligible thermal dilatation coefficient value with respect to the aluminium of the electrodes. The capacitor forming process and a final sample are shown in Figure 2.

The capacitors made as described, with d = 1 mm, d = 1.4 mm, and d = 2.8 mm, are namely C_1, C_2, and C_3, respectively.

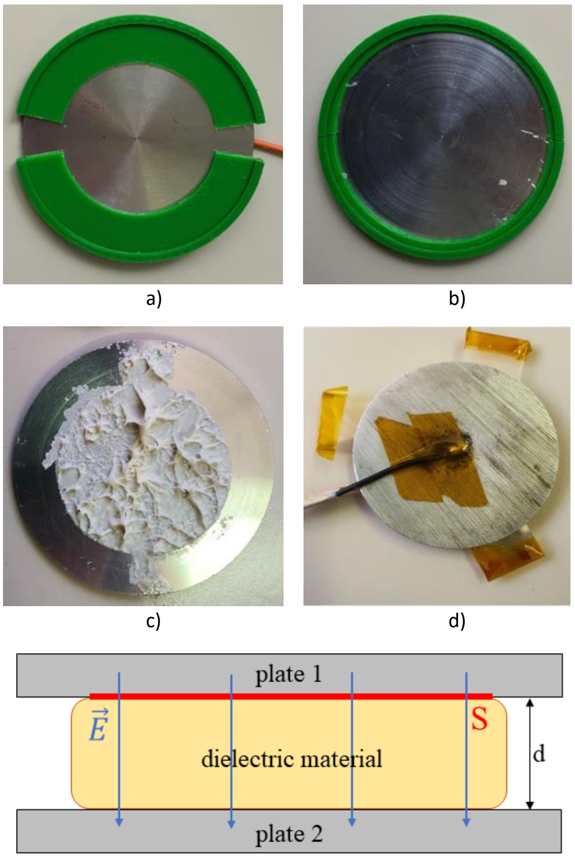


Figure 2. Spacers used for glue deposition (a) and final spacers for glue hardening (b). Internal glue disk area (c), final capacitor assembly (d) and its main scheme (e).

The second type of capacitor, namely C_4, is built with a distance d of 1.4 mm, kept by a 7 glass spacer with 0.2 mm thickness each, placed as the previous device. However, in this case, the glue is placed to fill all the plates and their borders. Moreover, the upper circular plate has 60 mm diameter and

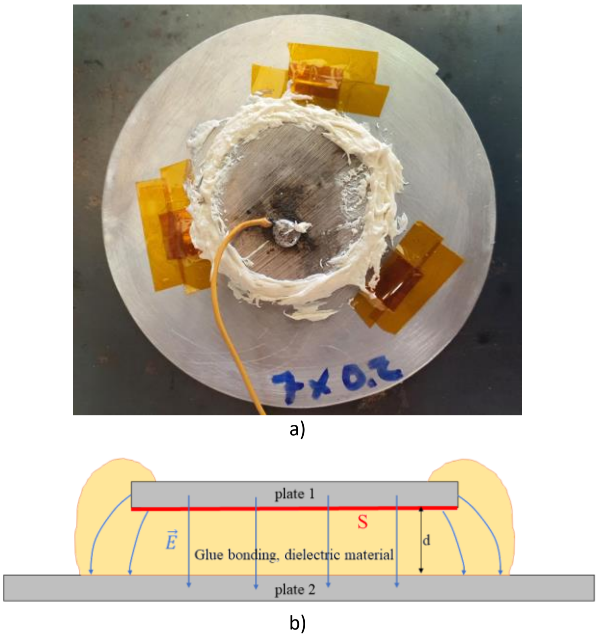


Figure 3. Capacitor sample C_4 with glue covering the plate border (a) and its scheme (b).

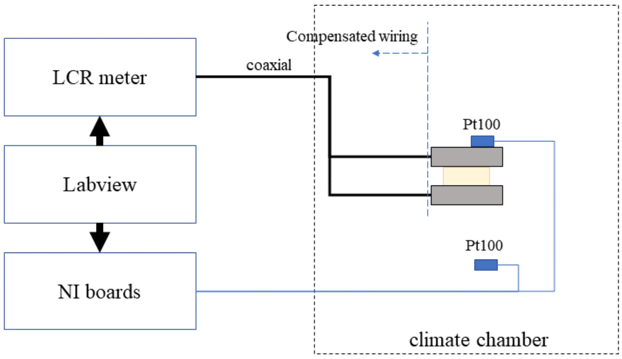


Figure 4. Scheme of the experimental setup.

3 mm thickness, while the bottom plate is 180 mm diameter and 1 mm thickness. The sample C_4 is shown in Figure 3.

To evaluate the sample's capacitance variation as a function of the temperature, the devices undergo to thermal cycling between -70 °C to +70 °C. The samples are placed in a Genviro 060LC climate chamber and cycled with a maximum heating rate of 7 °C/h. Such a low value is necessary to ensure thermal homogeneity of the sample, and to provide symmetric cycles during heating and cooling, thus limiting the influence of the thermal dynamic on the capacitance of the sample. The temperature is monitored on the top plate and on the climate chamber environment by using 2 RTD 1/10 DIN Pt100 sensors, which signals are acquired through a NI9219 board mounted on a NI9178 chassis. The capacitance C and the conductance G of the samples are measured using an LRC meter E4980A in the 20 Hz – 2 MHz range. Proper compensation of the connecting wire is made before each test. The model used for calculating C and G is a parallel between the capacitance C and a resistance, which value is equal to the reciprocal of G. Instruments management and data acquisition are performed through a Labview software made on purpose.

The scheme of the experimental setup is shown in Figure 4.

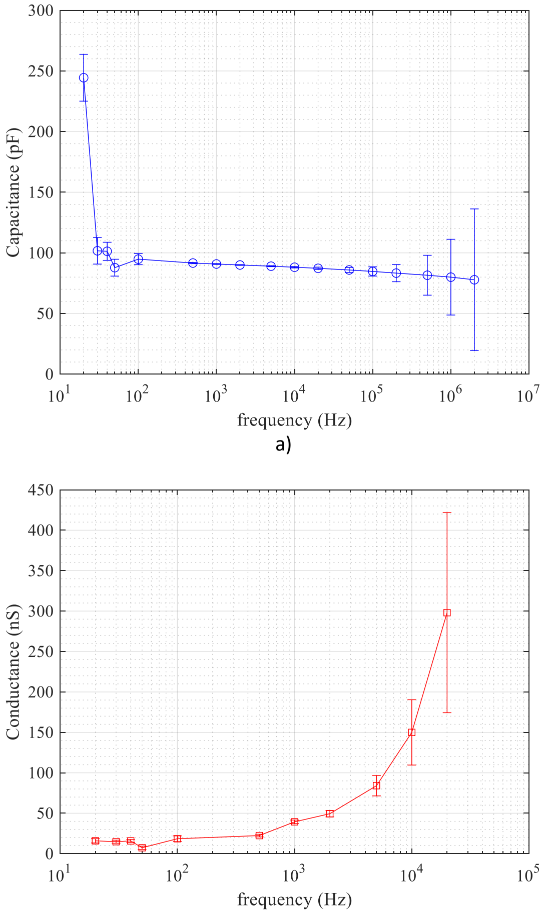
4. RESULTS AND DISCUSSION

The frequency response of the sample is reported at the temperature of (24.0 ± 0.9) °C in Figure 5 for the sample C_1.

Figure 5 shows that the capacitance C decreases with the frequency, while G increases with it. C and G are in the order of 100 pF and 100 to 300 nS, respectively. Data of G above 20 kHz are not reported due to high uncertainty in their value. In particular, a significant reduction of the capacitance occurs at around 20 Hz. In liquids, low excitation frequencies, typically in the order of some tenth of hertz, can cause ionic transport and layering phenomena, thus producing a double layer capacitance which largely increases the value of C. Besides, the C and G isothermal curves are consistent with the recent literature regarding polymers and amorphous materials for the studied frequency range [14].

The capacitance values obtained by C_1, C_2 and C_3 at (27.2 ± 0.6) °C are reported with respect to the value of C_3 in Figure 6.

Therefore, Figure 6 shows that the capacitance values vary as a function of $1/d$. Indeed, C_1/C_3 and C_2/C_3 agree with d_3/d_1 and d_3/d_2 according to the model shown in equations (1) and (2). However, for frequencies in the order of a tenth of hertz, the capacitance results in a different behaviour as discussed in relation to Figure 5a.



a)

b)

Figure 5. Capacitance C (a) and Conductance G (b) as a function of the analysis frequency for C_1. Uncertainties are given with 95% confidence interval.

In Figure 7, we show C_1 vs. T at 10 kHz frequency, with no glass spacers used.

The C_1 device (d = 1 mm) undergoes a quick cooling to -70 °C, where for 10 hours a steady-state temperature is maintained.

The initial thermal settlement is used to make uniform the temperature between the environment and the sample, and to reduce thermal gradients within the dielectric material. The first cooling branch in Figure 7b shows that, when the temperature is

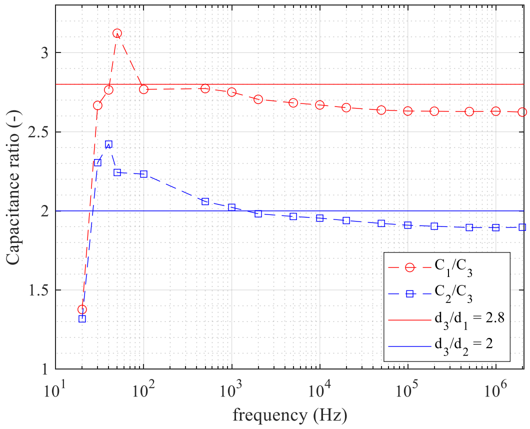
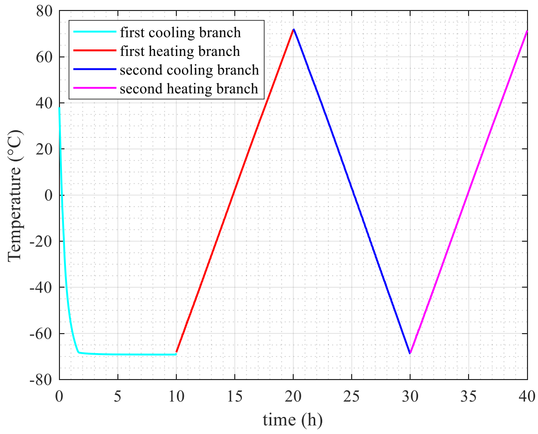
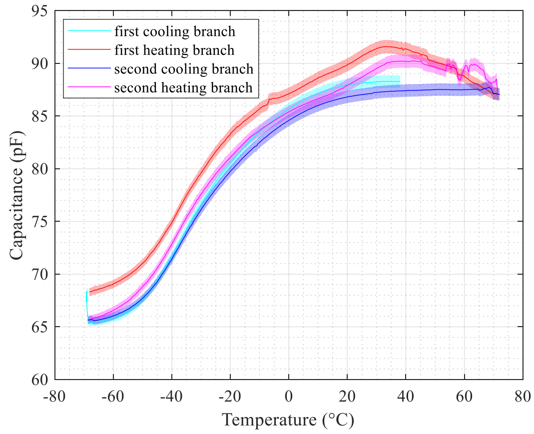


Figure 6. Capacity ratio at around 30 °C for C_1, C_2 and C_3 capacitors as a function of the frequency.



a) Temperature profile



b) Capacitance behaviour

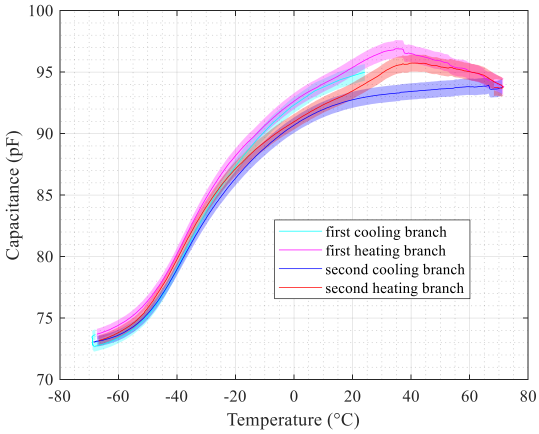
Figure 7. Measured temperature profile (a) and behaviour of C_1 sample (d = 1 mm) (b) with no fixing to the geometric sample parameters. Uncertainties are given with 95% confidence interval through the coloured strips.

constant, the capacitance slightly varies by increasing its value by around 3 % (i.e., 66 pF to 68 pF). This variation can be attributed to some stabilization phenomena.

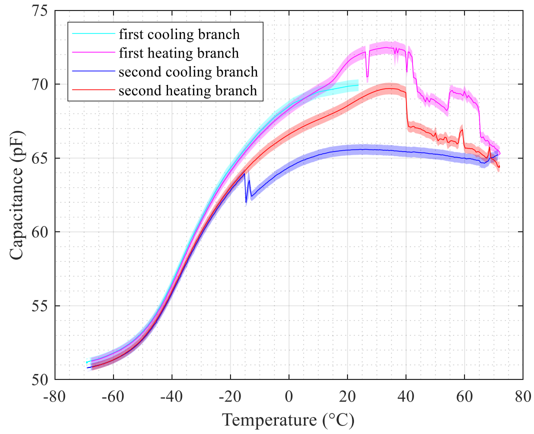
The next cycles clearly show a hysteresis on the C-T plot on the high-temperature side. In the present case, the hysteresis could be generated by the effect of the mechanical instability of the sample, as the combination of the effect of the glue softening, collapsing, and sliding, together with the characteristic permittivity evolution as a function of the temperature, proper of the dielectric material. However, at this study stage, hysteresis can also derive from more complex phenomena involving the physical characteristics of the dielectric material and depending on the permittivity behaviour vs. T [15], [16].

Figure 8 shows the capacitance behaviour of C_1, C_2 and C_3 samples with fixed minimum distance d under the thermal cycles shown by Figure 7a. Results are provided for 10 kHz frequency value.

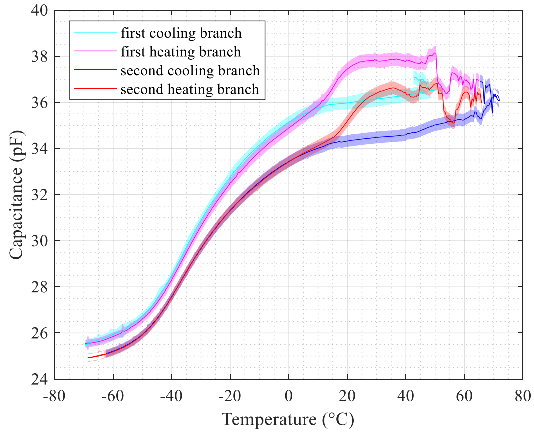
Figure 8 shows that hysteretic behaviour occurs in all the different samples with no significant difference concerning the case without spacers, shown in Figure 7. Furthermore, the samples C_2 and C_3 present a significant instability in the measure of C above 40 °C. This effect can be attributed to a major impact of the glue softening on the capacitor effective geometry. Moreover, in the cases shown by Figures 8b and 8c, second cycles (i.e. the second cooling and heating branches) present a shifted and lower value of C, indicating possible glue settlements.



a) C_1 (d = 1 mm)



b) C_2 (d = 1.4 mm)

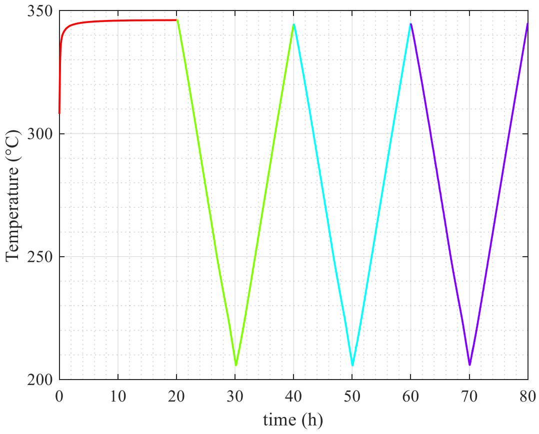


c) C_3 (d = 2.8 mm)

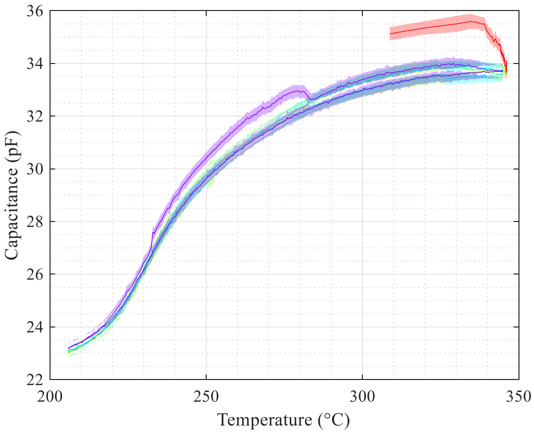
Figure 8. Behaviour of C_1 (a), C_2 (b) and C_3 (c) capacity as a function of the temperature during thermal cycling with fixed minimum plate distance and stabilization at -70 °C. Uncertainties are given with 95% confidence interval through the coloured strips.

According to these observations, the test is repeated on the case C_3 by stabilizing the sample at + 70 °C for 20 hours. The temperature profile over the time and the results in terms of capacitance at 10 kHz vs. temperature are shown in Figure 9.

In Figure 9, the hysteretic behaviour is significantly reduced and barely observable, while the measurement instability disappeared. This evidence highlights that the observed phenomena are determined by thermo-mechanical effects on the sample geometry. Moreover, the high-temperature treatment

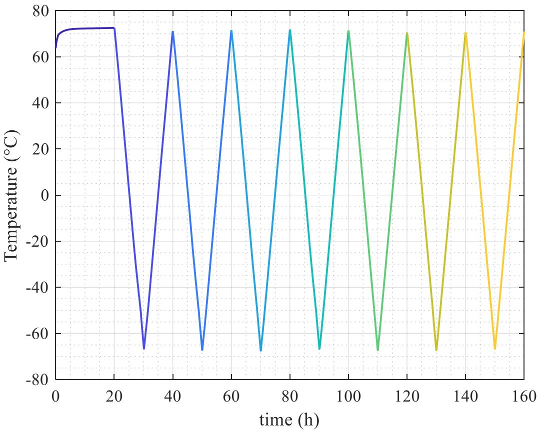


a) Temperature profile

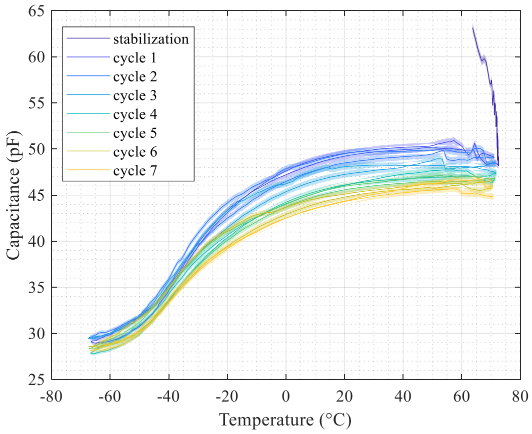


b) Capacitance behaviour

Figure 9. Measured temperature profile (a) and behaviour of C_3 sample (d = 2.8 mm) (b) with fixed minimum plate distance and stabilization at +70 °C. Uncertainties are given with 95 % confidence interval through the coloured strips.



a) Temperature profile



b) Capacitance behaviour

Figure 10. Measured temperature profile (a) and behaviour of C_4 sample (d = 1.4 mm) (b) with fixed minimum plate distance and stabilization at +70 °C. Uncertainties are given with 95 % confidence interval through the coloured strips.

process (red path in Figure 9) contributes to significantly mitigate these effects.

The last test is carried out on sample C_4, where the dielectric glue is placed over the electrodes and their borders, thus reducing the glue softening on the active region in between the electrodes. Thus, a 20 hours stabilization is performed at +70 °C and several cycles are provided between + 70 °C and -70 °C to further observe the capacitance behaviour. The results are shown in Figure 10, together with the performed temperature cycles. Moreover, Figure 11 shows the capacitance behaviour at 10 kHz as a function of the temperature for the 7th cycle (i.e. the last performed cycle) and different operating frequencies.

Figure 10 shows that the first 3 cycles exhibit an accommodation behaviour. However, the capacitance-temperature curve does not present any significant difference among the cycles after the 4th thermal cycle. Moreover, the capacitance hysteresis practically disappeared at this level of approximation, as also supported by Figure 11, for all the frequencies studied, and the C-T curve appears mainly monotone in the studied temperature range. The growth of C with the temperature is consistent with the recent literature regarding polymers and amorphous materials [14].

According to the presented results, the hysteresis shown in Figure 7, Figure 8, and Figure 9 is significantly reduced adopting thermal treatment and the C_4 configuration. Possible thermal-dynamic and thermo-mechanic phenomena still could affect the hysteretic behaviour of the capacitance. Further experiments are in progress aimed at investigating such behaviour. As discussed in the previous Section, it is worth highlighting that Figure 11 reports the behaviour of $C(T)$ of the C_4 sample for which the geometry variation can be practically neglected as a function of the temperature. Therefore, the results in Figure 11 can represent the behaviour of the permittivity $\varepsilon(T)$ according to equation (2).

5. CONCLUSIONS

The accurate positioning and referencing of sensors, such as strain or tilt sensors, is crucial for the proper measurand evaluation. In many cases, the sensor and the measuring body are glue bonded together, ensuring a solid and cheap fixing. On the other hand, the time stability of the bonding may be an issue especially when temperature cycles and the forces into play can affect the glue's physical and geometrical features. Therefore, thermal and mechanical analysis of a glue bonding is made via electrical measurement technique, by studying the behaviour of the capacitance in a device using glue as the dielectric material. Several types of fixing and pre-processing are used to separate the effects of the temperature cycles over the glue geometry variation. To assess such a behaviour, sample capacitance was analysed as a function of the electric field frequency and the environment temperature. It turned out that when no geometrical fixing is used, temperature cycles cause hysteretic

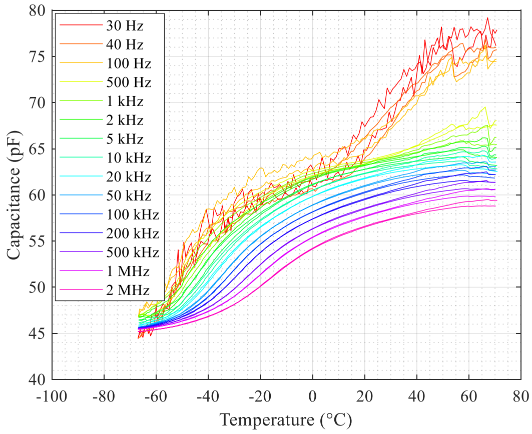


Figure 11. Behaviour of C_4 sample (d = 1.4 mm) at different temperature and excitation frequencies

behaviour of measured capacitance. When the capacitor geometry is fixed independently of the temperature, and after thermal stabilization of the dielectric, the hysteresis disappeared, and only a monotone behaviour *C-T* remains for all the tested frequencies. Therefore, we shown that the glue deformation is possibly responsible for the capacitance hysteretic behaviour. Despite this could be kept under control by e.g. proper thermal stabilization of the glue, an underestimation of this occurrence can lead to significant systematic errors in the evaluation of measurands through glue-fixed sensors.

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