Characterization of geometric imperfections and determination of the thermal expansion of a spherical acoustic resonator

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ABSTRACT. Important metrological applications in the field of primary thermometry and in the determination of the universal gas constant R from accurate measurements of speed of sound in spherical resonators have been demonstrated in recent years. A promising tool for the determination of the volume and the thermal expansion of a spherical cavity is the measurement of its microwave resonances in vacuum. This work reports results obtained at IEN in the characterization of a 1-liter spherical resonator. The thermal expansion of the resonator was determined in the range between 270 and 330 K. Deviations from ideal spherical shape were measured and tested by a comparison of non-radial acoustic modes and microwave multiplets.

INTRODUCTION. Recent developments of experimental practice and associated theoretical models make possible to measure the acoustic resonance frequencies f_N of the purely radial modes excited in a low-pressure gas contained within a spherical resonator with a precision in the order of 10^{-7} and an accuracy of $1\cdot10^{-6}$. In order to obtain from the acoustic frequencies a measure of the speed of sound u, an independent method to determine the resonator's volume V at a comparable level of accuracy is needed. Depending on the application of interest the volume must be determined at only one temperature, like in the determination of the gas constant R at the triple point of water T_t, or over an extended temperature range. For primary thermometry applications only the ratio of the speed of sound at the temperature of interest and at T_t is needed and only the volumetric thermal expansion of the resonator must be determined.

Different possible methods for determining the resonator's volume and thermal expansion include direct mechanical metrology using coordinate measuring machines on the two hemispheres of the resonator prior to their assembling; weighing the cavity filled with a reference fluid of well-characterized density and thermal expansion (e.g. mercury) [1], measurement of the resonance frequencies of the cavity filled with a reference gas whose speed of sound can be calculated, as a function of pressure and temperature, from independent measurements (at present only argon and nitrogen have been acoustically characterized [2, 3] with a sufficient degree of accuracy).

A promising alternative to these methods exploits the *exact* definition of the value of the speed of light in vacuum, through the determination of the microwave resonances of the cavity. This method was first developed and demonstrated by Moldover and co-workers who measured the thermal expansion of a spherical cavity in the range from 217 to 303 K [4, 5] and by Trusler [6] who extended the range between 90 and 300 K in primary acoustic thermometry applications.

The method exploits the observation demonstrated by Mehl and Moldover [7] that the average frequency of a non-radial microwave multiplet is independent of volume–preserving deformations of a spherical cavity, in the first order of perturbation theory.

In this work we report preliminary results obtained in determining the thermal expansion of the spherical resonator used at IEN[1] in the range between the triple point of water T_t and 330 K. Acoustic and microwave triply degenerate non-radial modes were measured and compared in the same temperature range in order to characterize the deviations of the resonator from perfect spherical shape. This work is part of a cooperative project between the Acoustics Department of IEN and the Thermometry Department of IMGC[2] which is currently under development with the aim to determine the differences (T-T_{90}) between the Kelvin thermodynamic temperature T and the International Temperature Scale of 1990 (ITS-90) in the range from the triple point of mercury up to 400 K.

APPARATUS AND PROCEDURE. A detailed description of the spherical resonator and the associated experimental apparatus used at IEN for speed of sound measurements can be found elsewhere [8]; only its main features which are of interest for the results presented in this work will be summarized hereafter.
The resonator has a nominal inner diameter of 12 cm and a nominal wall thickness of 1.28 cm. It is fabricated in two hemispheres from an austenitic stainless steel (316L). Conventional mechanical metrology with a co-ordinate measuring machine showed that the resonator had an average inside diameter of 11.9982 ± 0.0005 cm. The deviations from sphericity of each hemisphere were about ±1µm, a tolerance much finer than required for accurate measurements. The two hemispheres differed in average diameter by 0.0009 cm. Two identical transducers are used to excite and detect the acoustical resonances; they are commercially manufactured 1/4 inch condenser microphone, constructed with materials compatible with clean gases (nickel, quartz and gold). Transducer housings are used to support the microphones with their membrane in precisely defined positions and flush with the inner surface of the resonator. They are both located at 90° from each other on the same great circle through the north pole. This relative position is chosen in order to minimise the influence of the (l, n) non-radial modes having a node at the detector position (l =1,3,5,...) on the nearby (0, n) purely radial modes. The temperature of the gas within the resonator is inferred from reading two 100 Ω capsule-type platinum resistance thermometers which are embedded in metal blocks attached to the bosses at the top and the bottom ends of the resonator. Thermometers were calibrated by IMGC at the triple points of mercury, water and gallium. Accuracy achievable in the measurement of temperature, according to the results of the calibration and the performance and characteristics of the resistance bridge and the thermometers was estimated to be within ± 5 mK. Vertical temperature gradients between the two poles of the resonator during acoustic and microwave measurements were maintained within this limit. No effort is made to make the resonator gas-tight. Instead it is enclosed in a cylindrical steel pressure vessel which also acts as the innermost stage of a multi-stage thermostat. Gas flow from the vessel to the resonator interior volume is permitted by a vent-hole machined through the resonator shell. The diameter and length of this hole have been chosen to minimise the effects of perturbation on the radial modes of the cavity. Temperature is maintained constant throughout the acoustic and microwave measurements by immersion of the pressure vessel containing the resonator in a liquid stirred bath with a long-term stability of ±1 mK.

For microwave measurements the acoustic transducer assemblies were removed and replaced with plugs from which the microwave coupling probes extended. These were straight gold-plated conductors, with a diameter of 1 mm and protruding 3 mm into the spherical cavity, forming antennae which were able to couple efficiently to the TM modes, but not to TE modes. Microwave resonances were recorded while the resonator was maintained under vacuum below 1 mPa by the action of a mechanical and a turbo-molecular pump. The microwave signals were generated and detected by a Hewlett-Packard 8753E network analyzer which was connected to the probes through standard SMA connectors and semi-rigid copper conductors. This instrument permitted measurement up to 6 GHz. The analyzer was configured to measure $\mathbf{S}_{12}$,

[1] IEN-Istituto Elettrotecnico Nazionale Galileo Ferraris
[2] IMGC-Istituto di Metrologia Gustavo Colonnetti of CNR.

which is defined (for properly terminated lines) as the complex ratio of transmitted voltage to incident voltage.

MICROWAVE MEASUREMENTS. The microwave resonances f_{ln}^{s} of an evacuated cavity with non-magnetic wall are related to the radius *a* by:

$$f_{ln}^{s} = \left[(c/2ða)\right]í_{ln} + \Delta f_{ln}^{s} \qquad (1)$$

where *c* is the speed of light, $í_{ln}$ for the TM case is the *n*th zero of the *l*th order spherical Bessel function, Δf_{ln}^{s} is the sum of small correction terms. Allowed values of the indices are n = 1,2,3,…; l = 1,2,3,..; and s = - l, ,-1,0,1,.., l. In an idealized model defined by $\Delta f_{ln}^{s} = 0$ the normal component of the electric field vanishes at the boundary. As with the acoustic modes, the idealized resonance frequencies depend only on *l* and *n* and each frequency is therefore (2*l*+1) fold degenerate. In the present paper we are interested in TM modes for which *l*=1 and *n*=1,2, which are triply degenerate. We shall refer to them using the notation TM1n.

The most important contribution to the correction term Δf_{ln}^{s} arises from electrical dissipation in the wall, which for TM modes is :

$$\Delta f_{\mathrm{e}} = -\frac{fä}{2a}\left(1 - \frac{l(l+1)}{(í_{ln})^2}\right)^{-1} \qquad (2)$$

where $d = (ðfi_0\, ó)^{-1/2}$ is the skin depth, s is the electric conductivity of the cavity walls and m_0 the magnetic permeability of free space. The same dissipation mechanism is also responsible for the theoretical half-width of the microwave resonances which is given by $g_{ln} = -\Delta f_{\mathrm{e}}$.

<u>Data acquisition.</u> Preliminary measurements have been performed in the vicinity of the TM11 and TM12 triplets, at temperatures of 273, 303 and 333K. The TM11 and TM12 modes of the resonator used in this experiment occur at approximately 2.18 GHz and 4.86 GHz respectively. The network analyzer was used to scan 101 equally spaced frequencies spanning each of the two multiplets, with a width of 2.5 MHz and 3.5MHz respectively. Tipically 10 scans with an IF bandwidth of 10 Hz were averaged.

For each frequency the values of the real (*v*) and imaginary (*w*) part of the signal were stored on a computer and fit to a sum of three Lorentzian functions of the frequency:

$$\mathbf{S}_{12}(f) = v(f) + \mathrm{i}w(f) = \sum_{s=0,\pm 1} \frac{2\mathrm{i}\cdot f \cdot g_{ln}^{s} \cdot \mathbf{A}_{ln}^{s}}{(\mathbf{F}_{ln}^{s\,2} - f^2)} + \mathbf{B} + \mathbf{C}(\mathbf{F}_{ln}^{s} - f) \qquad (3)$$

Here ***A***, ***B*** and ***C*** are complex constants and ***F*** are the complex, nearly degenerate resonance frequencies of the investigated triplet.

<u>Partial splitting of the triplets and comparison with acoustic results.</u> At a casual inspection the mode TM11, which is shown in fig.1, appeared as a resolved doublet, which suggests an axysimmetric deformation of the sphere [5]. Evidence of the presence of a very weak excitation of the unresolved lower frequency component was obtained by comparing the results of a fit to the experimental data when including two or three terms in the sum of eq.(3). Summing over just two complex resonance frequencies (12 adjustable parameters) gave unsatisfactory systematic deviations and a standard deviation of the fit, expressed as a fraction of the maximum measured amplitude, of $1.25 \cdot 10^{-3}$. Including a third resonance in eq.(3), which takes up to 16 the number of adjustable parameters, the deviations were reduced by a factor 5, with a standard deviation of $2.5 \cdot 10^{-4}$ and became random (see right side of fig. 1). For the TM12 triplet the deviations from the fitting function were systematic and the standard deviation one order of magnitude larger than that typical for TM11. These observations are likely to be a consequence

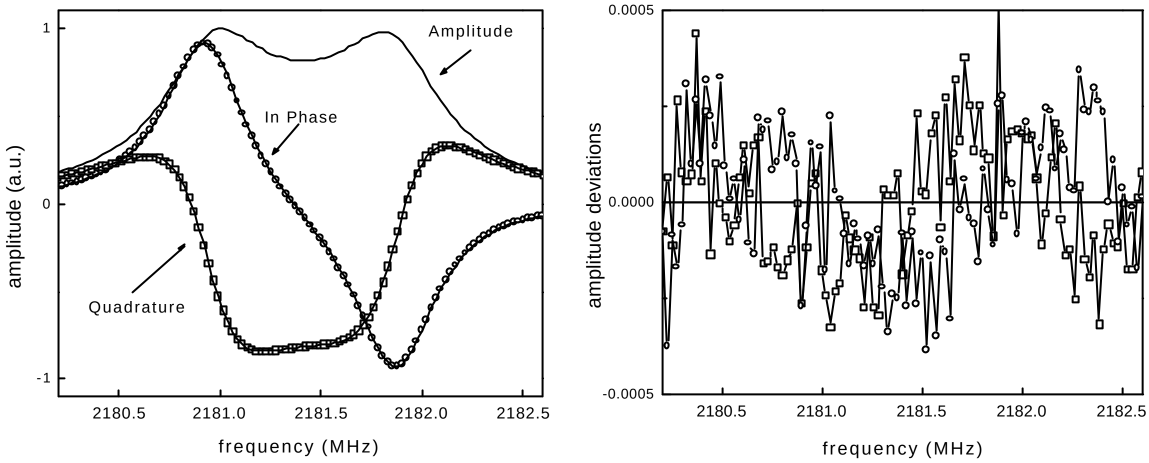

Fig. 1. Left: in phase and quadrature signals in proximity of TM11 mode. Right: deviations from a fit of eq. (3).

of the presence, close to the mode TM12, of the neighbouring mode TM41 and of its much more efficient coupling to the antennae.

For TM11 the fit gave evidence of a splitting of 500 kHz between the lowest two components and of 680 kHz between the highest two components. We obtained further evidence supporting the presence of a lower frequency double component by soldering a curve copper wire to one of the antennae and tuning by hand on the cavity filled with air at ambient temperature; the recorded amplitude of mode TM11 is shown in fig. 2.

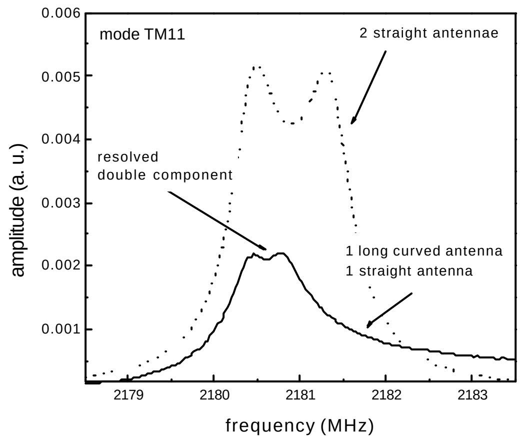

Fig. 2. Comparison of measurements of TM11 mode with antennae of different length.

As a further check of the correct interpretation of the detected splitting of the triply degenerate modes as arising only from geometric deformations from a perfect spherical shape, we tried to find evidence of the same kind and amount of splitting on the triply degenerate non-radial acoustic modes (1, *n*). According to the analysis of Mehl [9] of the effect of geometric imperfections of a spherical cavity on the resonance frequency of non-radial acoustic modes, we expected to identify the higher frequency component as a doublet (the sign of the frequency shift for TM1n multiplets is opposite to that of the acoustic (1,n) multiplets). Analysis of the acoustic multiplets (1,2), (1,3) and (1,4) with eq. (3) confirmed this hypothesis. For instance the two lower components of mode (1,4) which are shown in fig. 3 were found to be separated by 2.7 Hz while the two higher components were separated by 1.8 Hz.

Considering only a major axisymmetric deformation as responsible for the splitting of the microwave TM1,n and the acoustic (1,n) modes, we fitted again the experimental data with the function indicated in eq. (3) summing over just two complex resonance frequencies. For such a deformation, proportional to a small parameter e, the radial coordinate r describing the inner surface of the sphere would be given by an expansion in spherical harmonics:

$$r = a\left[1 - e\sum_{l=0}^{\infty} c_{l0} Y_{l0}(q, j)\right] \qquad (4)$$

where a is the cavity's radius, q and j are angular coordinates and c_{l0} are the coefficients of the expansion. The theory [7, 9] shows that in this case, the microwave and acoustic triplets would be split into a singlet and a degenerate doublet whose observed splitting determine the magnitude of εc_{20}. For the mode TM11 and TM12 at 273 K we found values of εc_{20} of $-1090 \cdot 10^{-6}$ and $-1087 \cdot 10^{-6}$ in good agreement with the values observed for the (1,2), (1,3) and (1,4) acoustic modes at the same temperature which were respectively equal to $-1090 \cdot 10^{-6}$, $-1055 \cdot 10^{-6}$ and $-1045 \cdot 10^{-6}$.

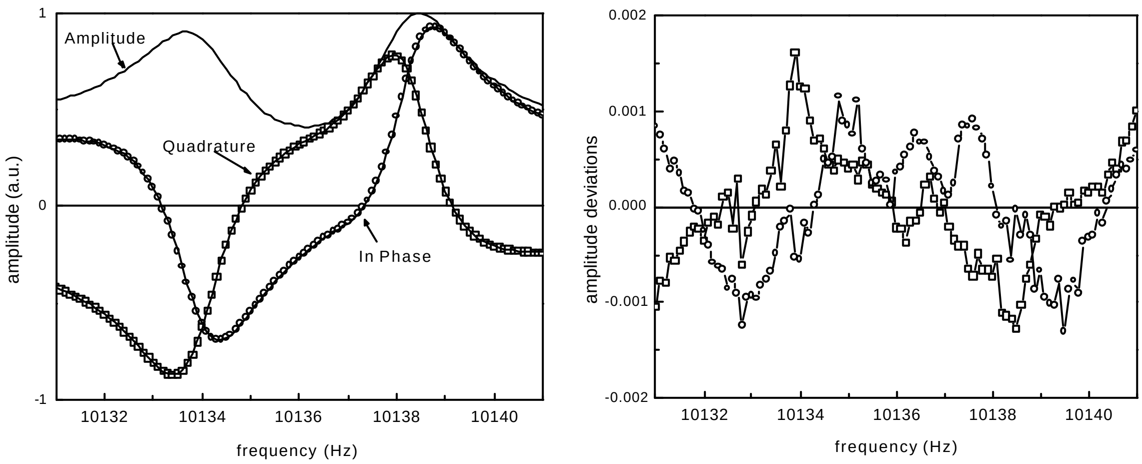

Fig. 3. Left: in phase and quadrature signals in proximity of the acoustic mode (1,4). Right: deviations from a fit to eq. (3).

<u>Widths of microwave resonances.</u> The differences Δg_{ln} between the experimental half-widths and the theoretical half-widths, calculated according to eq. (3) may be used to check the validity of the microwave model. For this purpose the microwave permeability of the cavity was assumed equal to the permeability of free space and the electrical conductivity of the cavity's walls equal to the dc conductivity [5]. Fig. 4 shows a comparison between experimental and calculated values of g_{ln}. As expected, the values of Δg_{ln} are positive and nearly temperature independent. Remarkably, differences in Δg_{ln} for the three components of each triplet are within less than 5 ppm for TM11 and 10 ppm for TM12.

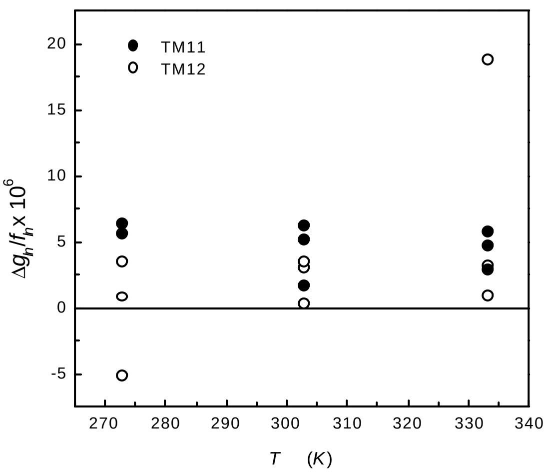

Fig. 4 Temperature dependence of the fractional excess half-widths of the three components of two microwave triplets TM11 and TM12. $\Delta g_{ln} = g_{ln}$(exp.)-g_{ln}(calc.).

Volumetric thermal expansion of the resonator. In order to determine the thermal expansion of the resonator, we computed a "corrected" average radius on the basis of the relation:

$$\langle a \rangle = \frac{c\nu_{ln}}{2\pi \cdot \left[\dfrac{1}{3} \sum_{\sigma=0,\pm1} f_{ln}^{s}(\exp.) + g_{ln}(\text{calc.}) \right]} \qquad (5)$$

Left part of fig. 4 reports the relative differences $\Delta a = \left(\langle a_{\mathrm{TM11}}\rangle - \langle a_{\mathrm{TM12}}\rangle\right) / \langle a_{\mathrm{TM11}}\rangle$ between the values of radius obtained from modes TM11 and TM12 as a function of temperature; the agreement is within 15 ppm. In order to determine the thermal expansion of the resonator, the results for the two modes were fitted to a linear function of temperature. The deviations between experimental values and the fit are within ten parts per million (see fig. 4 right). It is worth noticing that, of the two modes considered, TM11 shows a higher degree of confidence. The choice of a simple functional form of the fitting function is not critical for primary thermometry measurements [6]; more realistic forms of the variations of the ratio $a(T)/a(T_t)$ will be tested in the course of future work, when a higher number of modes will be available over a wider temperature range. We expect that this will allow to diminish the standard deviations of the fit to a few parts per million.

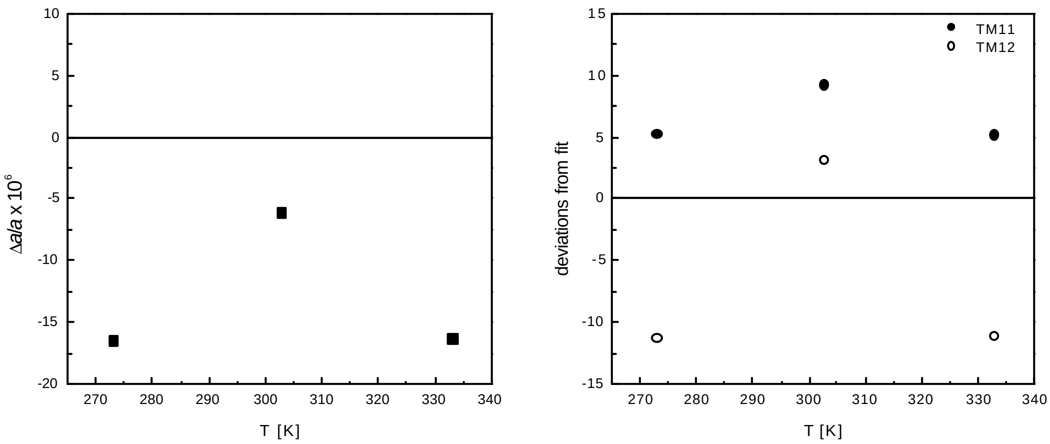

Fig. 4. Measurement of the resonator radius with microwaves. Left: comparison of results obtained from modes TM11 and TM12. Right. Deviations of the values from a linear fit as a function of temperature.

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