



Volume relaxation of quenched silica at room temperature monitored by whispering gallery mode resonance wavelength



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ABSTRACT

We used a resonance wavelength shift of a whispering gallery mode to monitor volume relaxation at room temperature in cylindrical and spherical resonators fabricated by quenching the molten tip of a pure silica optical fiber. The high sensitivity of the method allowed measurement of the linear dimension at sub-ppm resolution. All the resonators registered a volume increase in the first few hours, in agreement with the volume-temperature equilibrium curve of type IV silica below the glass transition temperature. When heating of the fiber was insufficient, the volume increase was greater, ascribed to a larger volume of strained silica. We also formulated thermodynamics to estimate the temperature coefficients of polarizability at constant pressure and at constant density from resonance shift data obtained in experiments performed on relaxed silica resonators.

1. Introduction

In a transparent dielectric medium of a circular cross section, light can travel near the surface via total internal reflection to complete a cycle [1]. This mode of wave propagation is called a whispering gallery mode (WGM). If the cycle contains an integral number of waves, the light superimposes constructively onto itself, and repeating this every cycle leads to a large amplitude. Since the intensity is proportional to the square of the amplitude, a strong wave can develop. The latter requires a strict condition for the wavelength, resulting in an extremely narrow resonance. In air, the Q factor can exceed 10^8 .

Different applications of WGM resonance have been proposed [2]. Among others, applications to sensing have been most intensively pursued, [3–7] as the resonance wavelength is sensitive to a small, localized refractive index (RI) change near the resonator surface in the surrounding medium.

Since most of the wave travels within the resonator, the resonance wavelength is sensitive to a change of the resonator itself. Suppose the radius of a spherical resonator increases without changing its RI. Then, detecting a radius change of 0.1 ppm is not difficult with WGM. In practice, however, the radius change and the RI change occur at the same time, and it is often the case that they cancel each other to some extent. Nevertheless, the resultant sensitivity is among the highest, and WGM has been applied to detecting pressure, temperature, and stress changes [8–11].

The present report focuses on monitoring the changes in a silica resonator after it is fabricated by heating the tip of a pure silica optical fiber and then quenched. Silica has been extensively studied, [12–16] and its thermo-mechanical properties are well understood. The properties depend on the type of silica, distinguished by the starting material and processing [13]. There are four types of silica. Their fabrication methods and concentrations of OH are summarized in Table 1. The silica core of the fiber used in our experiment belongs to type IV, containing OH at about 0.2 ppm according to the supplier.

Since silica is a preferred material for WGM resonators, their applications require that we have a good understanding of silica's properties and how the small variations in the starting material and processing affect the properties. Fortunately, monitoring the resonance wavelength shift can be a right tool to address this concern. Typically, a silica resonator is fabricated by exposing the tip of an optical fiber to a localized heat source to melt it. The surface tension results in a spherical resonator. When the heat source is turned off, the cooling takes just a second [17]. It means that the silica resonator is quenched. Therefore, the resonator is not at thermal equilibrium at least immediately after it is fabricated. Since the mobility of supercooled silica is low, it may take time to approach the equilibrium. This study finds how long it will take to get close to the equilibrium to the extent to stabilize the resonance wavelength.

In the present report, we first develop a thermodynamic theory to express the resonator's RI and the resonance wavelength as a function of

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Table 1
Fabrication methods and concentrations of OH for four types of silica.

Silica type	Method of preparation	OH concentration/ppm
I	Electrical fusion of natural quartz in vacuum or inert gas	Several
II	Melting quartz crystal with oxy-hydrogen flame	150–400
III	Hydrolyzing SiCl ₄ when sprayed into oxy-hydrogen flame	~1000
IV	Hydrolyzing SiCl ₄ with water-free plasma flame	< 1

temperature and density. In the theory, we will find a molecular-level expression of the resonance shift that will occur in volume relaxation post quenching and in a temperature change at around room temperature. We conducted these two lines of experiments on each of resonators of different diameters prepared by two different heating methods. After describing the experimental details, we will show the results and interpret them in the light of the theory.

2. Theory of WGM resonance shift in air

We employ here thermodynamic analysis to express the resonance wavelength shift in terms of a change in the state of the glass that constitutes the resonator. To describe the state of glass including non-equilibrium states, we choose the temperature T and the density ρ as two independent variables, rather than T and pressure p . This provision allows describing the state when the glass relaxes toward the equilibrium to change its volume V at constant temperature and pressure. Since the resonance wavelength in air is proportional to the product of the refractive index (RI) n and radius a of the spherical resonator, we will first look at how n and a change with T and ρ , assuming that the changes are isotropic. Since most thermodynamic quantities are measured at constant T and p , we need to find formulas to convert these quantities into those at a given temperature and density.

We model the glass as consisting of independent molecules with polarizability α at density ρ . When the temperature changes by ΔT and the density by $\Delta\rho$, the polarizability changes according to

$$\Delta\alpha = \left(\frac{\partial\alpha}{\partial T}\right)_\rho \Delta T + \left(\frac{\partial\alpha}{\partial\rho}\right)_T \Delta\rho \quad (1)$$

At constant pressure,

$$\left(\frac{\partial\alpha}{\partial T}\right)_p = \left(\frac{\partial\alpha}{\partial T}\right)_\rho + \left(\frac{\partial\alpha}{\partial\rho}\right)_T \left(\frac{\partial\rho}{\partial T}\right)_p \quad (2)$$

Since $\rho \sim 1/V$,

$$\frac{1}{\rho} \left(\frac{\partial\rho}{\partial T}\right)_p = -\frac{1}{V} \left(\frac{\partial V}{\partial T}\right)_p = -3 \left(\frac{\partial \ln a}{\partial T}\right)_p \quad (3)$$

where $(\partial \ln a / \partial T)_p$ represents the linear thermal expansion coefficient. For types I, II and IV silica, the linear thermal expansion coefficients are similar [18] and they are typically 0.52 ppm/K [19]. Therefore, Eq. (1) is now

$$\Delta\alpha = \left[\left(\frac{\partial\alpha}{\partial T}\right)_p + 3\rho \left(\frac{\partial \ln a}{\partial T}\right)_p \right] \left(\frac{\partial\alpha}{\partial\rho}\right)_T \Delta\rho + \left(\frac{\partial\alpha}{\partial\rho}\right)_T \Delta\rho \quad (4)$$

Lorentz-Lorenz equation relates the relative permittivity n^2 to $\rho\alpha$ as

$$\frac{n^2 - 1}{n^2 + 2} = \frac{4\pi}{3} \rho\alpha \quad (5)$$

For small changes,

$$\frac{6n\Delta n}{(n^2 - 1)(n^2 + 2)} = \frac{\Delta\rho}{\rho} + \frac{\Delta\alpha}{\alpha} \quad (6)$$

With Eq. (4), Eq. (6) is rewritten to

$$\frac{6n\Delta n}{(n^2 - 1)(n^2 + 2)} = \left[\frac{1}{\alpha} \left(\frac{\partial\alpha}{\partial T}\right)_p + 3 \left(\frac{\partial \ln a}{\partial T}\right)_p \right] \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \Delta T + \left[1 + \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \right] \frac{\Delta\rho}{\rho} \quad (7)$$

In Eq. (7), the RI change by pressure at a constant temperature is expressed as

$$\frac{6n}{(n^2 - 1)(n^2 + 2)} \left(\frac{\partial n}{\partial p}\right)_T = \frac{1}{K} \left[1 + \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \right] \quad (8)$$

where K is the bulk modulus (reciprocal of the isothermal compressibility):

$$\rho \left(\frac{\partial p}{\partial\rho}\right)_T = -V \left(\frac{\partial p}{\partial V}\right)_T = K \quad (9)$$

For typical silica, $n = 1.447$ (at wavelength 1.3 μm), $K_T = 35.9$ GPa, [20] and $(\partial n / \partial p)_T = 9.2 \times 10^{-12} \text{ Pa}^{-1}$ [21]. With these numbers, we can estimate, from Eq. (8), $(\rho/\alpha)(\partial\alpha/\partial\rho)_T$ as -0.36 . The negative coefficient indicates that silica decreases its polarizability (per unit SiO₂) with an increasing density.

Now, we consider the WGM resonance shift:

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta n}{n} + \frac{\Delta a}{a} = \frac{\Delta n}{n} + \frac{1}{3} \frac{\Delta V}{V} = \frac{\Delta n}{n} - \frac{1}{3} \frac{\Delta\rho}{\rho} \quad (10)$$

With Eq. (7),

$$\frac{\Delta\lambda}{\lambda} = \frac{(n^2 - 1)(n^2 + 2)}{6n^2} \left[\frac{1}{\alpha} \left(\frac{\partial\alpha}{\partial T}\right)_p + 3 \left(\frac{\partial \ln a}{\partial T}\right)_p \right] \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \Delta T + \left\{ \frac{(n^2 - 1)(n^2 + 2)}{6n^2} \left[1 + \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \right] - \frac{1}{3} \right\} \frac{\Delta\rho}{\rho} \quad (11)$$

For a density change at constant temperature,

$$\frac{\Delta\lambda}{\lambda} = \left(\frac{\partial \ln \lambda}{\partial \ln \rho}\right)_T \frac{\Delta\rho}{\rho} \quad (12)$$

where

$$\left(\frac{\partial \ln \lambda}{\partial \ln \rho}\right)_T = \frac{(n^2 - 1)(n^2 + 2)}{6n^2} \left[1 + \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \right] - \frac{1}{3} \quad (13)$$

With $n = 1.447$ and $1 + (\rho/\alpha)(\partial\alpha/\partial\rho)_T = 0.64$, we obtain $(\partial \ln \lambda / \partial \ln \rho)_T = -0.105$. This coefficient indicates that a density decrease of 1 ppm causes a 0.105 ppm red shift of the resonance wavelength. Alternatively, 1 ppm red shift of resonance wavelength amounts to a 9.51 ppm decrease in density, and a 9.51 ppm increase in volume.

Different experiments can measure Δn due to a volume change at a constant temperature. In Eq. (7), setting ΔT to zero leads to

$$\frac{\Delta n}{n} = -\frac{(n^2 - 1)(n^2 + 2)}{6n^2} \left[1 + \frac{\rho}{\alpha} \left(\frac{\partial\alpha}{\partial\rho}\right)_T \right] \frac{\Delta V}{V} \quad (14)$$

With $(\rho/\alpha)(\partial\alpha/\partial\rho)_T = -0.36$, $\Delta n/n = -0.23 \times \Delta V/V$. A 1 ppm red shift of resonance wavelength amounts to a 2.17 ppm decrease in RI. This coefficient compares favorably with the one in $\Delta n/n = -(n^2 p_1/2)(\Delta V/V)$, [16] where $p_1 \approx 0.22$ is the orientationally averaged elasto-optic coefficient.

For an experiment in which the temperature changes at a constant pressure, an expression of the WGM resonance shift can be derived from Eq. (10) as

$$\frac{\Delta\lambda}{\lambda} = \left(\frac{\partial \ln \lambda}{\partial T}\right)_p \Delta T \quad (15)$$

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