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► **To cite this version:**

Athanasios Laliotis, Santiago Villalba, Horacio Failache. Simple fabrication of miniaturized glass cells for atomic vapor spectroscopy. Applied physics. A, Materials science & processing, 2022. hal-03668740

HAL Id: hal-03668740

<https://hal.science/hal-03668740>

Submitted on 16 May 2022

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Simple fabrication of miniaturized glass cells for atomic vapor spectroscopy

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Abstract

Recent technological applications like atomic clocks, magnetometers or gyroscopes require the miniaturization of alkali vapor cells. We propose a procedure to manufacture miniaturized glass cells by cutting them from a larger one, previously filled with the required vapor. The proof of concept was demonstrated by fabricating millimeter-size cells with pure Rb vapor. We perform measurements of the mean lifetime of the $^{85}\text{Rb}(5D_{5/2})$ excited state in order to sensitively probe the presence of impurities inside the miniaturized cells. We find that the proposed cutting procedure seals the cells without compromising the Rb vapor purity. This demonstrates that the proposed method can be successfully used for vapor cell miniaturization.

Keywords: glass cell, miniaturized cell, atomic spectroscopy, photonics devices

1 Introduction

Spectroscopic studies on diluted atomic vapors were essential for the development of atomic physics. Most of this research was done, over the last hundred years, on low density (highly diluted) and high purity alkali vapors in glass cells. Glass is today the preferred material for vapor cell fabrication as it is inert enough when exposed to alkali atoms (this was verified by testing very old cells). Moreover, glass has a very low permeability, with a probable exception for the lightest alkalies as H and Li, and a good transmission for most light field wavelengths used in spectroscopy research. Most macroscopic glass cells are fabricated using traditional glass blowing techniques. However, technological applications of atomic physics like atomic frequency references and atomic clocks[1], magnetometers [2][3] and gyroscopes [4], which require miniaturization and mass production of spectroscopy cells need a different approach.

Several technological improvements were recently made towards fabricating miniaturized, mass produced cells [5–9]. Anodic bonding [10] of glass-silicon pieces is the most used technique, as described in [9]. This technique allows the fabrication of miniaturized cells where the body of the cell is etched in a wafer of Si to which the cell windows are bonded. It allows mass production, as several identical cells can be obtained from the same wafer of Si, with a cell geometry that is easily defined by the etching pattern. Anodic bonding requires, however, to heat the entire cell to around 300 °C. This high temperature is a drawback because the thermal expansion coefficients of materials must be matched to avoid cracks and hinder the use of anti-relaxation polymer coatings in the internal surface of the cell because these polymers have low melting point temperatures. Moreover, another drawback of this technique is that alkali atoms, or their precursors, must be placed inside the cell before sealing it. At these elevated temperatures, alkali atoms may diffuse in the glass, reducing the number of available alkali atoms and eventually darkening the glass. Several methods have been developed to fill in the cells [7][9][11–15]. Some involve the use of alkali chlorides, chromates or azides, that are placed inside the cell, together with other components, before sealing the cell. Pure alkali atoms are then produced in a chemical reaction that takes place at relatively high temperatures. These reactions have some by-products that in some cases must be pumped before sealing the cells, that must be therefore placed inside a vacuum chamber during the manufacturing procedure. Most applications require a buffer gas, or a mixture of them, together with alkali atoms. More recently a low-temperature bonding was tested as an alternative to anodic-bonding [16]. Cell production using these above elaborate techniques is nevertheless complex and relatively expensive.

In this work, we present a procedure to fabricate miniature spectroscopy glass cells based on the idea of cutting a tiny cell from a larger one, that was previously filled with the required molecular or atomic vapor. This strategy preserves therefore all the flexibility of the filling procedure of macroscopic cells. This miniaturization process is based on a cutting element that cuts

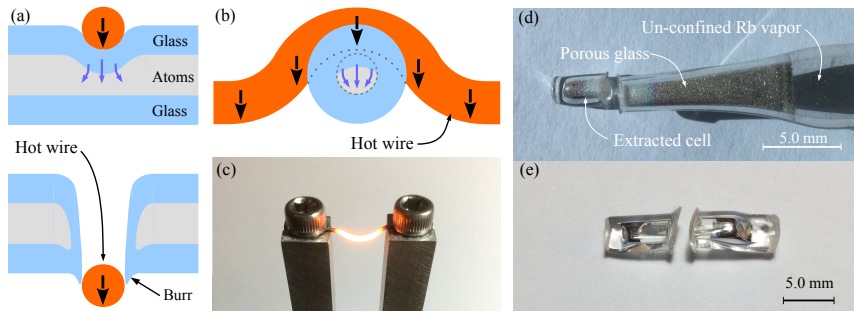


Fig. 1 (Color online) (a),(b) Diagram describing the proposed method of cutting and sealing miniature spectroscopy cells. In (a) are represented two stages of the cutting process, showing a longitudinal section of a portion of a cylindrical sealed cell, and in (b) the corresponding transversal section. (c) A photograph of the hot wire. (d) Photograph of a porous cell and the tiny cell extracted from it. We also show the volume of unconfined Rb vapor where spectroscopy measurement can be done (the porous glass is colored by Rb clusters, that migrated away from the cutting point when its temperature increased [17]). (e) A standard cell was divided to make two smaller cells.

while simultaneously seals the otherwise exposed internal volume of the cell. This technique has the advantage of being simple, fast, and inexpensive. The procedure was tested on glass spectroscopic cells filled with pure Rb vapor, on which we have verified that the purity of the alkali vapor was preserved during the cutting process.

2 Miniature cell fabrication

To extract small spectroscopic cells from a larger one, we have used a cutting element that is hot enough to melt glass. While this element moves it drags melted glass creating sealing walls that separate the larger cell in two smaller ones, as sketched in the inset of Fig.1a,b. This cutting element is an electrically heated NiCr (Nichrome alloy) wire with a circular cross-section. This alloy is one of the most commonly used electric heating elements, because of its relatively high electrical resistivity, if compared to other metallic conductors, and for its stability at high temperatures. This alloy shows high resistance to oxidation which preserves the smoothness of the wire surface at high temperature. The shape and dimensions of the cross-section, as well as the thickness of the cell's glass wall, are relevant parameters that determine the amount of melted glass that is dragged in and become available to build the seal between the walls of the cell. The amount of this melted glass determines therefore the maximum extension of the sealing walls (or the maximum cell vacuum gap that could be sealed).

The wire temperature is also an important parameter, as it determines the temperature and therefore fluidity of the melted glass. We have performed the cutting process in the open air, therefore, if the melted glass is too fluid, the

sealing wall could collapse under the pressure difference between the external atmospheric pressure and the vacuum in the cell. On the other hand, a less fluid glass hinders the wire movement.

All these parameters, together with the velocity at which the wire moves, should be chosen for a given particular cell geometry. We have started roughly optimizing these parameters testing the cutting procedure with several dummy cells, evacuated but not containing any vapor.

Our initial tests on Rb-filled cells were done on porous glass cells [17][18]. These home-made cells were created by sintering pyrex glass powder inside a glass tube to create a porous medium. The tube was then sealed after filling it with pure Rb vapor [17] (Fig.1d). These porous glass cells are for example useful for the development of robust atomic frequency references [18]. We have used porous glass cells to test our cutting procedure because they have the advantage that the vacuum gap to be sealed is determined by the size of the pores (typically 50 μm) in our cells. Additionally, there is ample glass available to build these seals. We have used a hot wire with a diameter of 700 μm , and a temperature of approximately $T_w \sim 1100$ °C. Despite our initial tests on dummy cells, the cutting procedure on two porous cells failed, compromising the Rb purity. The analysis of these two cells using an optical microscope revealed a failed seal that shows bubbles. Two other porous cells were later successfully cut using a colder wire ($T_w \sim 1050$ °C) to increase the viscosity of the melted glass.

After successfully cutting few porous cells, we proceed with a standard cell. We have specially made a cell using a pyrex-glass cylindrical tube, with an external diameter of 5 mm and a wall thickness of 1.0 mm, that was partially flattened to define an internal gap of approximately 1 mm. This cell was connected to a vacuum system composed of a turbo-molecular pump and an ion-pump and was evacuated down to a pressure of 10^{-9} Torr. During this pumping process, the cell was baked at 200 °C for 24 hrs. A small dot of high purity Rb was then distilled into the cell before sealing and extracting it from the vacuum system. To extract the cell we followed the standard procedure that uses the flame of a torch to soften the glass tube to make it collapse under atmospheric pressure. The cell can be then sealed and separated from the vacuum system by melting the glass at the point where the tube collapsed.

In order to reduce the risk of failure of the seal during the cutting process of this cell, we have used a wire temperature slightly lower than the one used on porous cells to have a less fluid glass. The cutting process lasted approximately 2 minutes. Moreover, we have used in this case a curved wire, like the one shown in Fig.1(b,c), to drag and feed the seal with more glass and reducing burrs. The result of this cutting process is shown in Fig.1d.

During the cutting procedure, it is important to reduce the number of Rb atoms in contact with hot or melted glass, because alkali atoms diffuse into the glass at high temperature reducing the amount of pure Rb in the cell and

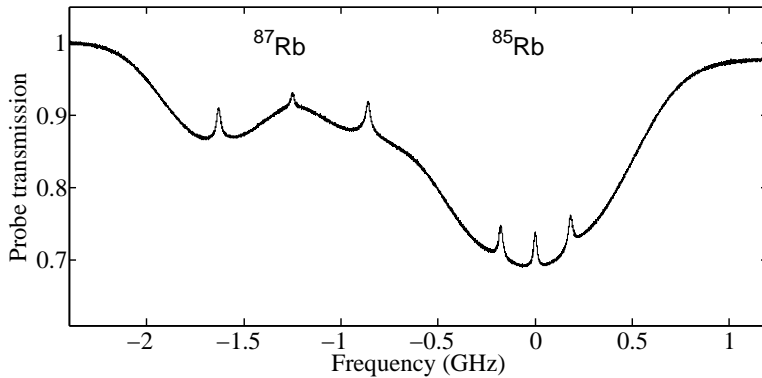


Fig. 2 Probe beam transmission through one of the miniaturized cells shown in Fig.1e in a saturated absorption spectroscopy setup [19]. The full-width-half-maximum of the saturated absorption resonances is ~ 16 MHz (essentially determined by frequency noise in the laser field). This spectrum was measured in the D1 line of Rb. The frequency detuning in this figure is measured from the cross-over of the $^{85}\text{Rb } 5S_{1/2}(F=3) \rightarrow 5P_{1/2}$ transition.

darkening the glass. To circumvent this issue, we have defined a cold point in the cell, away from the cutting place, where the Rb remains condensed. The ends of the cell were placed in good thermal contact with aluminum bodies of several cm^3 that fulfill as temperature reservoirs. Small cavities to accommodate the ends of the cell were machined in the metallic bodies, and thermal conductive paste was used to reduce the thermal resistance of the glass-metal contact. Using this procedure, the temperature of the glass at the cold point was kept below XX °C during the cutting procedure.

As a primary indication of a successful cutting, in Fig.2 we show an example of saturated absorption spectrum obtained with one of the cells shown in Fig.1e. In particular, this spectra was measured in a miniaturized saturated absorption spectrometer that was only possible after miniaturizing the spectroscopy cell [19].

3 Spectroscopy analysis

All our cell cutting procedures were done in the open air. Earth's atmosphere is composed essentially of N_2 (78 %) and O_2 (21%), therefore a possible leakage during the cut procedure would essentially introduce N_2 as contaminating gas. We assume that O_2 is eliminated in a fast redox reaction with the highly reactive rubidium atoms. Such N_2 impurities introduce inelastic collisions that in particular broaden the D1 and D2 transitions of Rb with a typical coefficient of 18 MHz/Torr [20]. This broadening could be measured using Doppler-free saturated absorption spectroscopy [21] and used to determine the pressure of the potential N_2 contamination. Using a carefully aligned setup, with an extended cavity diode laser and a reference cell with pure Rb vapor, we have

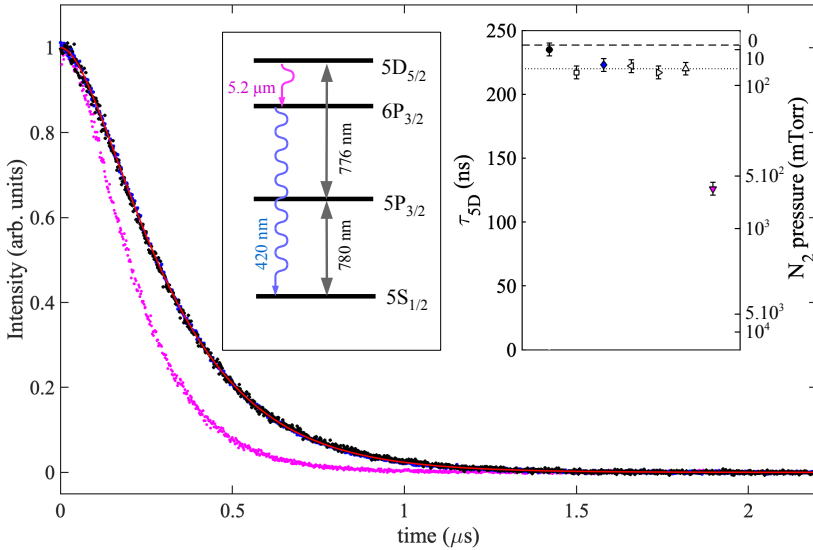


Fig. 3 (Color online) Transient decay of the blue fluorescence intensity, recorded after turning off the 776 nm laser. We show the transient for a reference cell (filled with pure Rb, in black), the transient for a miniaturized cell (blue), and the corresponding model fit (red). For comparison, we also show the transient for a slightly contaminated cell (magenta). *Insets:* Rb energy levels involved in the experiment. $^{85}\text{Rb}(5\text{D}_{5/2})$ level mean lifetime τ_{5D} measured by fitting the theoretical model. The corresponding N_2 pressure is shown in the right vertical axis. \bullet : reference cell, \square : a cell before cutting and \blacklozenge after cutting, \blacktriangleright : porous cell after cutting, \blacktriangleleft : porous cell from the same batch, \triangle : cell from another batch, \blacktriangledown : slightly contaminated cell.

measured the dependence of the saturated absorption resonance width with the light intensity. To neglect the power broadening, we have extrapolated to zero light intensity a width of 15 MHz (full-width half-maximum). This width is much larger than the expected natural width of approximately 6 MHz for the Rb 5P levels, most probably due to frequency noise of the laser source. This low spectral resolution limits the pressure of N_2 impurities that can be measured by the saturated absorption technique to a fraction of Torr.

We have considered an alternative technique based on the measurement of the lifetime of an excited state of Rb by measuring, in the time domain, the transient of the spontaneously emitted fluorescence. Using this method the pressure of N_2 impurities can be estimated with good accuracy without being limited by the linewidth or frequency noise of the laser sources. We have in particular measured the lifetime of $^{85}\text{Rb}(5\text{D}_{5/2})$ levels following the method described in [22]. These Rb levels were excited using the ladder-level excitation 780-776 nm technique [23] described in the inset of Fig.3. We have measured the transient decay of the blue fluorescence intensity at 420 nm after

Table 1 Measured values of τ_{5D} on different Rb cells. These cells were produced in 3 different batches which are arbitrarily numbered.

Cell	$\tau_{5D}(ns)$	Batch
Standard cell (reference)	235 ± 5	-
Standard cell (before cutting)	217 ± 5	1
Standard cell (after cutting)	223 ± 5	2
Porous cell (after cutting)	222 ± 5	2
Porous cell (batch reference)	217 ± 5	2
Porous cell (batch reference)	220 ± 5	3
Porous cell (failed cutting)	126 ± 5	2

suddenly shutting down the 776 nm field using a fiber-coupled Mach-Zender intensity modulator (model PHOTLINE NIR-MX800-LN-10), and measuring the blue fluorescence transient using a solid state photon counting detector (model Perkin Elmer SPCM-AQRH-13). The $^{85}\text{Rb}(5D_{5/2})$ level lifetime was then measured by adding approximately 10^6 measurements of the fluorescence transient and fitting the resulting curve with a theoretical model. Fig.3 shows the transients of the blue fluorescence in three different cells: a reference commercial cell that has pure Rb vapor (Toptica), one of the cells shown in Fig.1e, and one of the porous cell that was contaminated during the cutting procedure. We also show a fit of the theoretical model from which the τ_{5D} lifetime value was determined. The uncertainty on the value of this lifetime was estimated to be ± 5 ns [22].

The mean lifetime of the 5D level τ_{5D} can be reduced due to quenching collisions with N_2 molecules. This reduction can be estimated with the equation $\tau^{-1} = \tau_{5D}^{-1} + \sigma_q \sqrt{\frac{8}{\pi \mu k T}} p$ [21], where σ_q is the cross-section of quenching collisions for the 5D levels, μ is the reduced mass of N_2 - Rb two-body system, k is the Boltzmann constant, T the absolute temperature, and p the pressure. Unfortunately, σ_q has not been previously measured for the 5D levels of Rb. We can, however, make a rough estimate of the magnitude of this cross-section from the measurements of Supronowicz et al. [24], who report the quenching collisions cross-section of the 7D and 8D Rb level by N_2 collisions to be $\sigma_q = 30 \pm 30 \text{ \AA}^2$. We expect a similar cross-section for the 5D energy level considering that this value is also close in magnitude to those of intermediate nP states in Rb [25] and Cs [26], nS ($5 < n < 9$) [27] and D states in Na [28]. The $^{85}\text{Rb}(5D_{5/2})$ has a natural lifetime $\tau_{5D} = 238$ ns [29], that due to quenching collisions is then reduced as $\delta\tau \sim -\tau^2 \sigma_q \sqrt{\frac{8}{\pi \mu k T}} \delta p \sim 360 \text{ ns/Torr}$ of N_2 at 100 °C. Therefore, with this procedure, we can estimate the pressure of N_2 impurities with an accuracy of ~ 14 mTorr.

Alternative techniques to measure the presence of N_2 impurities could be considered. One of them is the Electromagnetically Induced Transparency (EIT) spectroscopy of Rydberg atoms [30]. In the particular case of Rb, the cross-section of dephasing collisions of Rydberg states with N_2 molecules lie

in the range of 1000 \AA^2 [31]. This implies a broadening coefficient of approximately 25 MHz/Torr at 100 °C. The width of EIT resonances for Rydberg states of Rb could be as low as a fraction of MHz [32]. Assuming such narrow resonances, this technique could also be used to detect a pressure of N_2 impurities of few tens of mTorr. An other possible technique is to detect the shift and broadening of the Rb $5\text{S}_{1/2} \rightarrow 5\text{D}_{5/2}$ two-photon transition, due to collisions with impurities and particularly N_2 [33]. In this case an accuracy on the N_2 pressure of several mTorr to few tens of mTorr could be achieved, although requiring a precision on the measure of the width and/or shift of this transition of few hundred kHz. However, these two techniques are affected by the frequency noise of the laser sources.

In the inset of Fig.3 and Table.1, we show some measurements of τ_{5D} on several cells. For the reference cell, we obtain a τ_{5D} value of 235 ± 5 ns, close to the theoretical value. We have measured τ_{5D} on the cells shown in Fig.1e before and after the cutting procedure and we do not observe a significant difference. We have also measured approximately the same value for τ_{5D} in the unconfined Rb vapor region (see Fig.1d) in a cut porous cell, a reference porous cell from the same batch, and in another cell from a different batch. We attribute the small difference observed on these measurements of τ_{5D} with the theoretical value to some imperfection in our filling procedure of spectroscopy cells. We can conclude that the pressure of N_2 impurities potentially introduced during a cutting procedure can be considered lower than the sensitivity limit of our measurement method, i.e. few tens of mTorr.

In the inset of Fig.3, we also show the value of τ_{5D} measured in a porous cell that is clearly detected as contaminated after a failed cutting procedure. This value of τ_{5D} corresponds to a N_2 contamination pressure of about 595 mTorr. When we have used this cell in a saturated absorption spectroscopy setup, where we have measured an absorption resonance full-width-half-maximum of 17 MHz (at the limit of zero light intensity), only slightly larger than the value obtained for a pure Rb cell. This result is consistent with our previous considerations about the low sensitivity of the saturated absorption technique.

4 Discussion and conclusion

The present work is a proof of principle of a new method for miniaturized cell fabrication. In Fig.4 we sketch a geometry that could be potentially used to produce miniaturized spectroscopy pre-filled cells. The minimum size of the cells that could be cut is limited by the maximum stress created by the glass temperature difference between the hot cutting point and the glass near the metallic Rb, that should be kept at a much lower temperature. After our experiments, in the case of pyrex glass this minimum size is well below 5 mm. This procedure has the advantages that is simple, fast and inexpensive. Moreover, most miniaturized atomic clocks and magnetometers require the use of

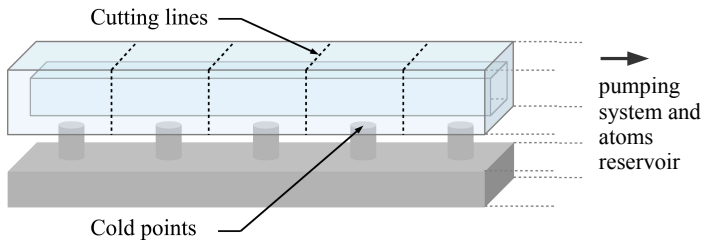


Fig. 4 A possible configuration to fabricate small spectroscopy cells from a larger one. Black dashed lines show the cutting places. A metallic structure could be glued to the cell to reduce the fragility of the system and to define cold points where alkali atoms remain condensed during the cutting procedure.

an alkali vapor and a mixture of buffer gases with specific partial pressures. In principle, the technique presented here would allow to fill a large "mother" cell with an alkali and buffer gas mixture that would be replicated in the tiny "daughter" cell.

The technique presented here should be further improved, reducing the risk of failure of the seal, by performing the cutting procedure in a chamber with an atmosphere of controlled composition and pressure. The cutting element could be also improved using a more complex device. For example, instead of using a single cutting element that works as a knife, as in this work, a two or multiple elements cutting device, acting like scissors, could be used to reduce burrs and have better control of the shape of the miniaturized cell.

Acknowledgments. We gratefully acknowledge fruitful discussions with Arturo Lezama and financial support from CSIC (Universidad de la República).

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