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Title: Dispensing and Hermetic Sealing Rb in a Miniature Reference Cell for Integrated Atomic Clocks

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Abstract: An innovative method for fabricating the reference cell for a Rubidium (Rb) integrated atomic clock is presented. This method uses low-temperature solder sealing technique for producing mini-cells of the size of 14 x 10 x 3 mm, suitable for Rb miniature atomic clocks. Top and bottom of the cell consists of two glass slides. An LTCC (Low-Temperature Cofired Ceramic) module of 2 mm thickness, equipped with a small reservoir for confining the Rb is placed in between the two walls, acting as a spacer, increasing the total volume of the cell. A solder ring joins together the LTCC and the top of the cell. This paper also presents a new technique for handling the Rb, which allows its safe handling and long storage. The alkali metal is stored inside a pool of dodecane, which protects it from oxidation. Pure liquid Rb is then dispensed inside the adjacent reservoir using a glass micropipette; finally, the cell is heated in vacuum, in order to carry out the sealing. The achieved sealing hermeticity was tested, without Rb, by sealing N₂O gas and monitoring its pressure through absorbance measurements using FTIR spectroscopy. Hermeticity was also tested with Rb by integrating a pressure sensor in the LTCC module.

Dispensing and Hermetic Sealing Rb in a Miniature Reference Cell for Integrated Atomic Clocks

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Revised version

COVER LETTER

18.3.2011

Dear Editor,

Please find attached the files for the paper entitled “Dispensing and Hermetic Sealing Rb in a Miniature Reference Cell for Integrated Atomic Clocks” authored by Fabrizio Vecchio et al., which integrates considerable advances compared to the extended abstract presented at Eurosensors XXIV as contribution #3478.

This is a revised version, according to the referee's comments. Additionally, minor language mistakes were corrected, and precisions added.

Please let me know in case you find anything missing during the review of the paper.

Best regards

Thomas Maeder

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Abstract

An innovative method for fabricating the reference cell for a Rubidium (Rb) integrated atomic clock is presented. This method uses low-temperature solder sealing technique for producing mini-cells of the size of 14 x 10 x 3 mm, suitable for Rb miniature atomic clocks. Top and bottom of the cell consists of two glass slides. An LTCC (Low-Temperature Cofired Ceramic) module of 2 mm thickness, equipped with a small reservoir for confining the Rb is placed in between the two walls, acting as a spacer, increasing the total volume of the cell. A solder ring joins together the LTCC and the top of the cell. This paper also presents a new technique for handling the Rb, which allows its safe handling and long storage. The alkali metal is stored inside a pool of dodecane, which protects it from oxidation. Pure liquid Rb is then dispensed inside the adjacent reservoir using a glass micropipette; finally, the cell is heated in vacuum, in order to carry out the sealing. The achieved sealing hermeticity was tested, without Rb, by sealing N₂O gas and monitoring its pressure through absorbance measurements using FTIR spectroscopy. Hermeticity was also tested with Rb by integrating a pressure sensor in the LTCC module.

Keywords: atomic clock; mini-cell; Rubidium; LTCC.

1. Introduction

Precise time and frequency references are crucial aspects for a wide range of electronic-based systems such as communication networks, data processing devices, and global positioning [1]. Atomic clocks provide the most accurate time and frequency standards [2], but their relatively large size and power consumption have to date prevented widespread deployment of precise timing in mobile, portable, battery-powered applications. However, as the demand for high precision timing in portable devices rises with the rapid spread of communications and navigation systems, there is a strong need of portable, low power and precise frequency standards that can be efficiently produced with batch techniques.

Our overall goal is to achieve a miniature, low power atomic clock in the 10 cm³ and 100 mW volume and power range, in order to get a miniature, low-power device [1-5]. In our size range, we do not need the miniaturization level achieved by MEMS technologies; this research intends to fabricate reference vapor cells of size ca. \varnothing 5 mm x 3 mm. Nevertheless, we do aim at a batch fabricable process, i.e. more efficient than traditional glass-blown mini-cells.

In the development of a miniature atomic clock, obtaining a vapor cell that is reliable and stable, yet also easily fabricated is arguably the most challenging part. Besides being small, the cell must be filled with Rb (used in this work) or Cs, sealed, and heated during operation to generate a significant vapor pressure. However, alkali metals are very reactive with oxygen and water, and therefore they must be protected from air before and during the sealing process, and the resulting cell must be completely hermetic in order to avoid its rapid degradation. The most widely used approach to a hermetic mini-cell is by MEMS technology, using anodic bonding [6] to close the cells. The problem of this well-known and standard

sealing process is that it requires relatively high temperatures (~450 °C) and long times (a few hours), which often causes evaporation or excessive reactions of the alkali metal.

In this research, a fast and low-temperature soldering technique is used for sealing, which presents the following advantages with respect to anodic bonding:

1. Traditional eutectic solder alloys [7] have low melting point; for instance the Sn-Bi eutectic (58Bi-42Sn) melts at 138°C, allows to perform the sealing process at very low temperatures.
2. The sealing process is accomplished in a few minutes, with the peak temperature (solder melting time) being of the order of 10 s, minimizing evaporation and reactions of the alkali metal.
3. Despite the low melting point of both Rb and traditional solder metals, these systems form intermediate compounds with very high melting points (e.g. [8]), and exhibit very limited solubility and a very low levels of mixing in the alkali liquids. The joint is therefore expected to remain stable and hermetic.

Another big issue related with the fabrication of reference cells for atomic clock is to find a fast, safe and convenient method to dispense the alkali metal inside the cell. The strong reactivity of the alkali metals with oxygen makes their handling very difficult; during dispensing, the metal must be completely protected by air. The traditional techniques used so far are complicated and need sophisticated tools. Dispensing methods using azides (RbN_3 or CsN_3 or indirectly by reacting RbCl / CsCl with BaN_6) [9] introduce safety issues, as these compounds are highly toxic and explosive. Additionally, they emit gases inside the cell after sealing, an issue that is also somewhat present with laser activation of a latent alkali source / getter inside an anodically bonded cell [10]; even if this latter technique elegantly solves the reactivity problem, any gases are then trapped by the getter, making this method incompatible with buffer gas cells.

In this research, we present a comparatively facile technique involving dispensing a drop of pure liquid Rb using a simple glass micropipette. To efficiently protect the alkali from ambient oxygen and moisture, essentially all handling operations are carried out in a pool of hydrophobic solvent of relatively low volatility.

Lastly, instead of MEMS technologies, we take advantage of the excellent 3D structurability of LTCC materials [11-13] to fabricate a vapor cell structure acting as a spacer between two glass windows and equipped with a Rubidium reservoir for better confinement of the alkali metal (Fig. 1a). Moreover, in this work, a test variant of this spacer was produced, where the bottom of the vapor cell cavity was closed with an integrated pressure sensor, consisting of an LTCC membrane carrying a thick-film Wheatstone bridge [14] and thus allowing monitoring of the cell pressure (Fig. 1b).

FIGURE 1

2. Rubidium handling and dispensing method

The developed alkali metal manipulation procedure is illustrated in Fig. 2. As our vapor cell is comparatively large and contains a dedicated metal reservoir (see below), we can easily dispense a relatively large drop of Rb using a glass micropipette. The originality of our method lies in that all manipulations are carried out inside a large pool of dodecane, which protects the alkali metal from oxidation during handling and, being hydrophobic, does not readily take up water.

FIGURE 2

First, the Rb is slightly heated up to its melting point, and dispensed into the reservoir located on the LTCC module, staying under the dodecane. Then, the lid is placed onto the cell, and the resulting assembly is rapidly transferred into a **vacuum-soldering** chamber. During the transfer, short-term

protection of the Rb from air is still ensured by the small volume of dodecane that stays inside the reservoir. In the vacuum soldering chamber, the cell is first progressively heated up to 80°C in 20 mbar of N₂ to gently evaporate the dodecane inside the reservoir while avoiding outright boiling. Boiling, i.e. a greater solvent vapor pressure than the ambient total pressure (the 20 mbar partial vacuum used in this work), is avoided because it may disturb the position of the Rb drop or even the position of the lid on the LTCC module.

After this evaporation step, the cell is further heated up to the soldering temperature for typically 20 s in the final desired atmosphere, ensuring hermetic closure.

This method prevents Rb oxidation during the sealing process, and allows easy handling under ambient conditions. For long-term storage, the box containing the dodecane pool is simply closed and placed into a glove box under dry nitrogen, where the dodecane provides additional protection against residual moisture. Of course, this technique may be further enhanced by adding to the dodecane pool oxygen- and water-trapping chemicals.

3. LTCC-based cell with integrated Rb reservoir & optional pressure sensor

3.1. Design of LTCC spacer

The LTCC spacers fabricated in this work for the envisioned mini-cells (Fig. 1) are rectangular-shaped of dimension 14·10·2 mm, with a vapor cavity of ø5 mm. The different modules are built using five different layers, designated T1-T5 (Fig. 3 & 4). T1 carries the metallization and the corresponding solder ring screen-printed on top of it, and is cut with a round hole for the vapor cell and a trapezoidal one for the Rb reservoir. T2, stacked many times to increase the thickness of the module, is identical to T1, but consists of only "bare" LTCC, i.e. without metallization or solder. T3 only has the hole for the membrane, and serves as the "floor" of the Rb reservoir.

For test purposes, a sequence of T4 & T5 may be used instead of T3; the bottom of the cell is closed in this case by an LTCC membrane (T4) with a screen-printed pressure-sensing thick-film piezoresistive bridge on the bottom, i.e. outside of the cell. T5 is used as a spacer to protect the resistor and conductor on T4 from contact and consequent sticking with the alumina carrier during firing. In our test configuration, the cell is under partial vacuum after sealing (Fig. 1b & 5), and the other side of the membrane is in atmospheric pressure, therefore, the output voltage of the Wheatstone bridge monitors the pressure difference across the membrane and, correcting for atmospheric pressure variations, allows measurement of the inside pressure. As the resistors lie on the outer surface and only the bare LTCC is exposed to Rb, no significant degradation of the membrane is expected. Additionally, the LTCC cell frame is very stiff, effectively insulating the membrane from parasitic stresses arising from the soldering of the glass lid.

FIGURE 3

FIGURE 4

FIGURE 5

3.2. Fabrication of LTCC spacer

The LTCC module was fabricated with the same materials as in our previous work [14]: DuPont (DP) 951 LTCC tape, DP 6146 co-firing solderable AgPd metallization paste and DP 2041 10 k Ω piezoresistive composition (test cell). The process for fabricating the LTCC spacer involves different repeatable and standard steps.

- Laser cutting of the different LTCC layers
- Screen-printing of the metallization layers, of the resistors for the membrane and the various connections
- Precise stacking and lamination of the LTCC module
- Co-firing, with a standard LTCC cycle having a top dwell of 20 min at 875°C
- Screen-printing and reflow of the solder paste on top of the metallization layer
- Thorough cleaning of the solder flux in an ultrasonic bath with ethanol

3.3. Glass windows

The top window is made from standard soda-lime float glass microscope slides (dimension 75·25 mm², 1 mm thick) that are metallized and "pre-tinned" with solder in a similar manner to the LTCC, i.e. the process consists of metallization, solder print & reflow and cleaning steps. The main difference lies in the metallization process (see following section): a different paste is used, and the metallization is post-fired onto the glass at moderate temperature (525-625°C), rather than co-fired at 875°C with LTCC. The slides are then diced with a diamond saw into the individual 10·10 mm² windows (it is possible to get 14 samples from one microscope slide).

For the final cell, a similar process may be used for the bottom window, or, alternatively, the bottom window may be bonded to the LTCC spacer prior to filling using a high-temperature process such as glass frit bonding, or even be co-fired as an insert with the LTCC.

3.4. Rb filling and sealing

Once all elements have been fabricated (Fig. 6), Rb must be dispensed inside the cell, and the sealing is carried out as described in section 2:

- Dispensing of Rubidium inside the adjacent reservoir;
- Precise alignment of top and bottom walls;
- Heating of the cell in vacuum to reflow the solder and realize the sealing.

FIGURE 6

4. Preliminary qualification of the process

4.1. Glass metallization and soldering studies

Before starting the production of the glass slide, different solder / metallization combinations were tried, in order to provide a first selection of promising alternatives. For the metallization layer, ESL 590G (Ag, 525°C firing temperature; ESL = ElectroScience Laboratories, USA), ESL 9912A (Ag, 625°C firing temperature) and ESL 9695 (AgPd, 625°C firing temperature) thick-film pastes were tested. Besides the metals, these pastes contained non-specified proprietary glass frits and oxides designed to allow bonding to nonmetallic substrates [15]. For the solder, standard eutectic 58Bi42Sn, 62.5Sn36.5Pb1Ag and Bi-Sn-Ag solder pastes were used (all proportions given in weight). The resulting cells were first visually

compared using an optical microscope, with the aim to find combinations exhibiting reliable and complete wetting of the metallization by the solder (results in Table 1 and illustrated in Fig. 7). ESL 9912A together with Bi-Sn / Bi-Sn-Ag was found to be a very attractive combination, featuring good wetting and a low melting point that both reduces Rb evaporation and leaching of the metallization by molten solder [15,16].

TABLE 1

FIGURE 7

The hermeticity achieved by some cells produced without dispensing the Rb inside the reservoir was tested by sealing 30 mbar of N₂O gas and monitoring its pressure through absorbance measurements using FTIR spectroscopy [17]. A first calibration test was done by soldering a pressure sensor on top of the cell and, comparing the FTIR peak with the pressure read by the pressure sensor, we found out that 30 mbar of pressure corresponded to a peak of magnitude 0.2. The results of this test are illustrated in Fig. 8.

The FTIR showed that the cell stayed sealed with 30 mbar of gas inside it for 2 weeks. After, the pressure started to slowly increase until reaching atmospheric value, indicating that further development work is necessary to improve the durability of the sealing.

FIGURE 8

4.2. Tests performed on cells with Rb

The first qualitative test was visual observation; after sealing, the Rb inside the cell remained silver in color for ≥ 10 days (date of writing this report), suggesting that the cell is hermetic. In the other case, Rb transformation to white powder, probably RbOH [18], would be very fast and clearly visible (Fig. 9).

FIGURE 9

For this cell, the output voltage of the Wheatstone bridge was measured before sealing and monitored periodically after sealing (with a constant, nominally 5.0 V, excitation voltage), in order to monitor the cell inside pressure. The output changed considerably during the sealing operation, reflecting transition from a zero differential pressure (both membrane sides at atmosphere) to ~ -1 bar immediately after sealing, indicating that the sealing operation was at least initially successful. However, a relaxation of this differential pressure, shown in Fig. 10, was observed over a few days, suggesting an increase of the pressure inside the cell.

The long-time stability of the cell is a critical issue. Therefore, all air ingress into the cell must be avoided in order to protect Rb from oxidation: Rb should stay in its original state during the lifetime of the cell, i.e. of the whole atomic clock. However, the evolution of the measured pressure apparently does not correspond to air ingress, as the Rb remained metallic. Possible causes include degassing of organics entrapped in the solder and drift of the pressure sensor caused by mechanical stresses. While the LTCC frame is rigid and expected to efficiently shield the membrane from stresses arising from the soldering, this supposition must still be tested, both by experiments and finite element modeling. Also, due to fabrication issues in this first generation of integrated membranes (partial overlap of the outer resistors by LTCC), the quality of the pressure output signal is somewhat poor.

FIGURE 10

5. Conclusions and outlook

In this work, a facile low-temperature process for the fabrication of hermetic Rb vapor cells for miniature atomic clocks has been presented, involving handling of the alkali metals under a protective solvent pool, and rapid low-temperature sealing by soldering. The introduction of an LTCC spacer provides an elegant manner to achieve a large cell volume, i.e. increased depth, while also allowing the sealing of the bottom window to be carried out before filling by a stable high-temperature process such as glass frit bonding.

Preliminary results are encouraging, Rb apparently remaining in its original metallic state, sealed in the cells, for ≥ 10 days. The LTCC spacer provides a convenient test platform, as it allows easy integration of a pressure sensor to monitor the cell after sealing.

However, the work presented here still suffers from some issues, and materials, procedures and cell design must be further improved:

- Additional functions can be added to the LTCC module, such as a temperature control and RF electrodes, in order to achieve a fully integrated LTCC resonance cell.
- The metallization and soldering materials must be further investigated and optimized, to minimize leaching, enhance wetting of the metallization by solder and eliminate contamination by solder flux residues.
- Long-term accelerated high-temperature aging treatments, accompanied by in- and ex-situ characterization will be carried out to fully assess the reliability and potential lifetime of this solution.

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Biographies

Fabrizio Vecchio received his BSc degree in Electrical Engineering from Università di Cassino in 2006. He received his second BSc degree in Electrical and Electronic Engineering from University of Wales in 2008. Finally, he received his MSc degree in Electrical Engineering from Università di Cassino in 2009. His research is focused on thick-film and LTCC technology, and their application for the packaging of miniature atomic clocks.

Vinu Venkatraman received his Bachelor of Engineering in Electrical & Electronics from Birla Institute of Technology & Science (India) in 2006, Master of Science in Electrical Engineering from Portland State

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Herbert R. Shea has a PhD (1997) and a MA (1993) in physics from Harvard University, and a BSc (1991) in physics from McGill University. After 2 years as a post-doctoral fellow at IBM's T.J. Watson Research Center he joined Lucent Technologies' Bell Labs in Murray Hill, NJ, USA, first as a member of technical staff (1999–2001), then (2001–2004) as the technical manager of the Microsystems Technology group. Since April 2004, he is an assistant professor at the EPFL in Lausanne, Switzerland, with a focus on ultra-reliable MEMS for space applications. Research interests include nanosatellites, polymer MEMS, ionic propulsion, atomic clocks, and the reliability and accelerated testing of silicon and polymer based microsystems.

Thomas Maeder graduated at the Ecole Polytechnique Fédérale de Lausanne (EPFL) in materials science, and continued with a PhD in piezoelectric thin films, then a post-doc at IBM Rüschlikon in single-crystal conductive oxides. He now heads the thick-film technology group at the EPFL, where current areas of interest are thick-film and LTCC technology for advanced sensor, packaging, biomedical and harsh-environment applications.

Peter Ryser received a Master degree in Physics (University Neuchâtel 1979), a PhD in applied Physics (University Geneva 1985) and a Masters Degree in Corporate Management (Lucerne 1993). His professional background includes several R&D activities. From 1990-1998 he was the head of research at Siemens Building Technologies. Since 1999 Peter Ryser is Professor at the Swiss Federal Institute of Technology EPFL in Lausanne and act as a director for the micro engineering section.

Figure captions

Fig.1. Schematic cross section of the final (a) and test (b) vapor cells.

Fig. 2. Schematic drawing of Rb handling and storage in dodecane pool.

Fig. 3. 3D view of the five different layer types that compose the LTCC module.

Fig. 4. Photographs of the 5 different tapes, T1-T5.

Fig. 5 Schematic drawing of piezoresistive pressure-sensing membrane integrated in LTCC test spacers, (a) before and (b) after sealing. Piezoresistors R1-R4 are connected in a Wheatstone bridge configuration.

Fig.6. Glass slide = lid of test cell (a) and LTCC test module with membrane = bottom & side wall (b).

Fig.7. Example of good (left) and poor (right) solder wetting.

Fig. 8. FTIR transmission spectrum of two glass lids directly soldered in N_2O , without an LTCC spacer.

Fig. 9. Sealed test cell (a), with Rb still silvery in color (b).

Fig. 10. Voltage output of the piezoresisive membrane Wheatstone bridge, showing the shift in pressure due to sealing (partial vacuum), followed by a slow apparent return to atmospheric pressure.

Table captions

Table 1. Visual comparison of the different metallisation-solder combinations on glass.

Figure 1
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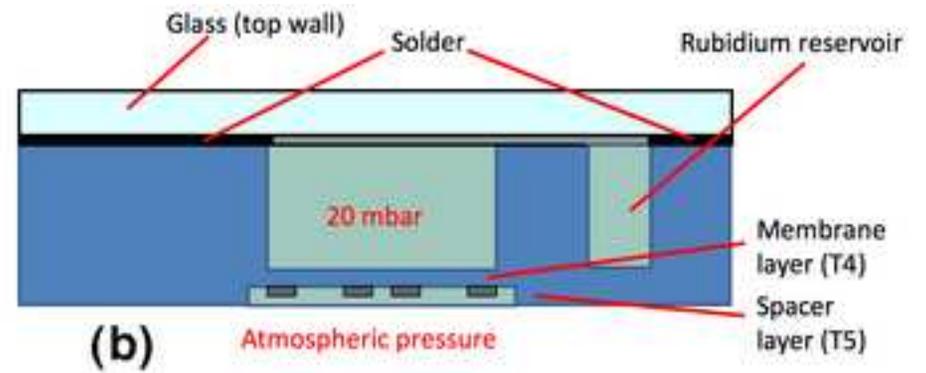
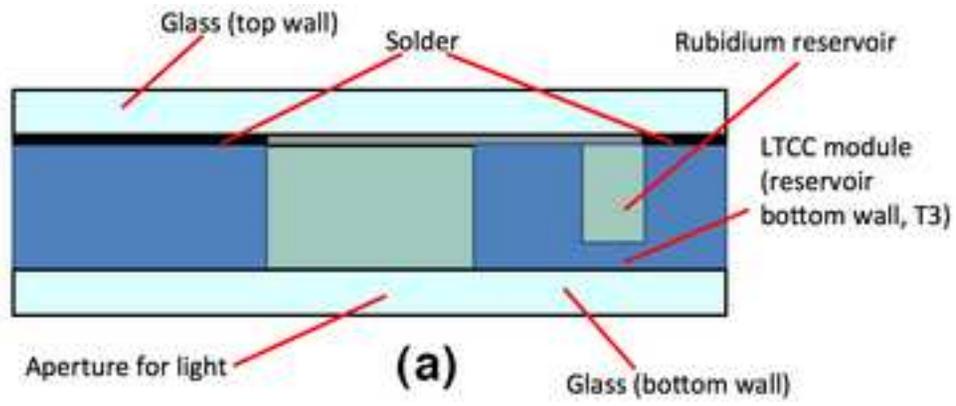


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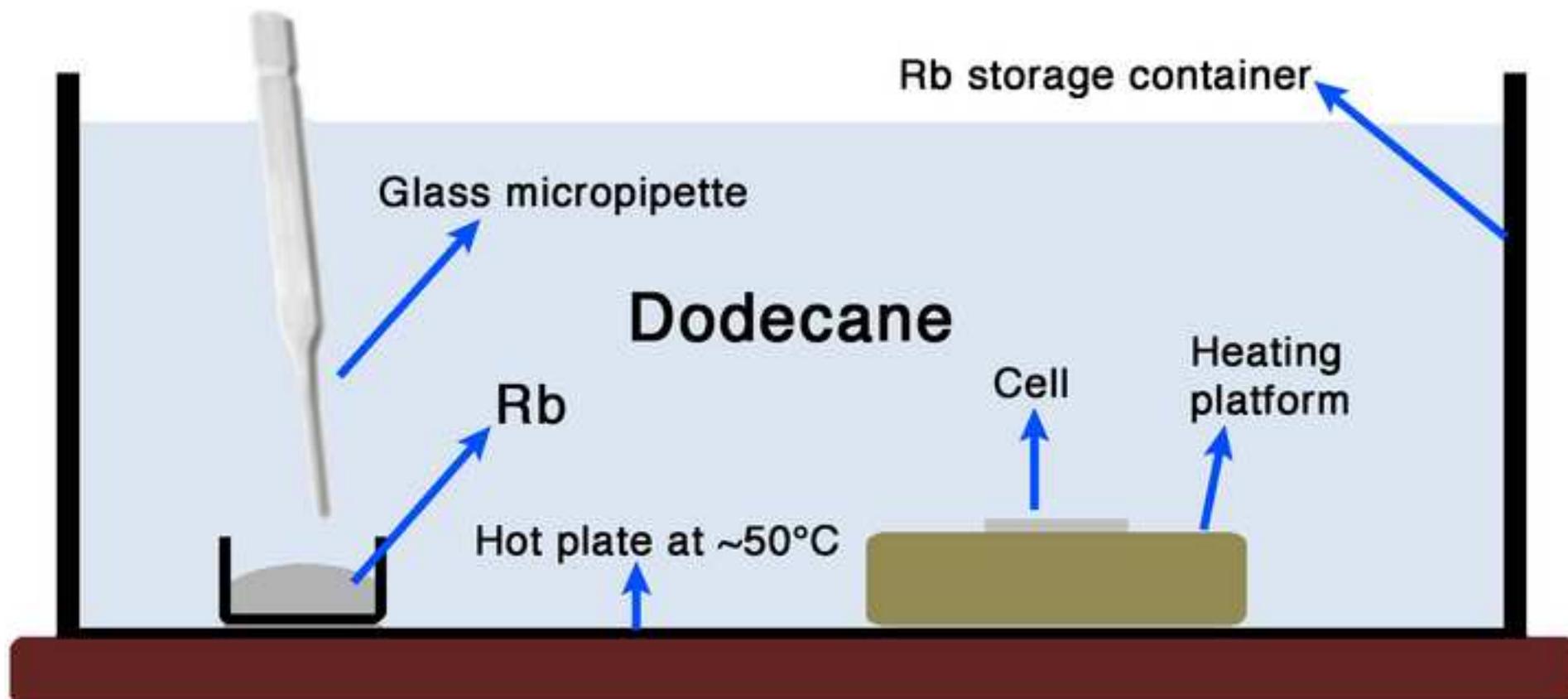


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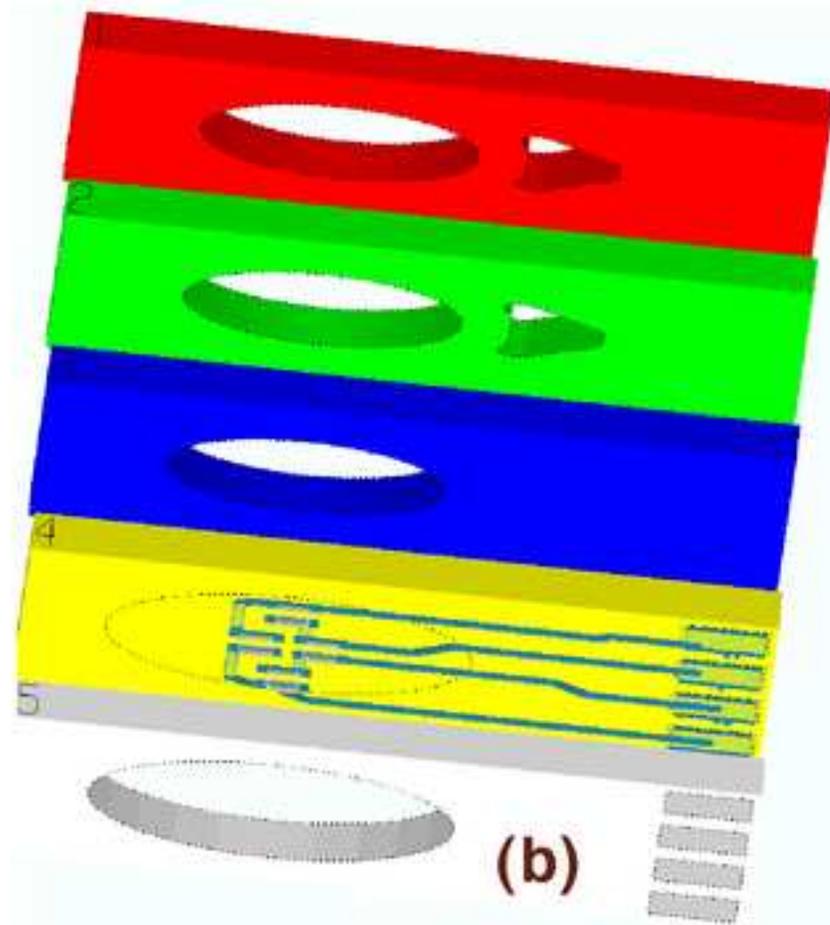
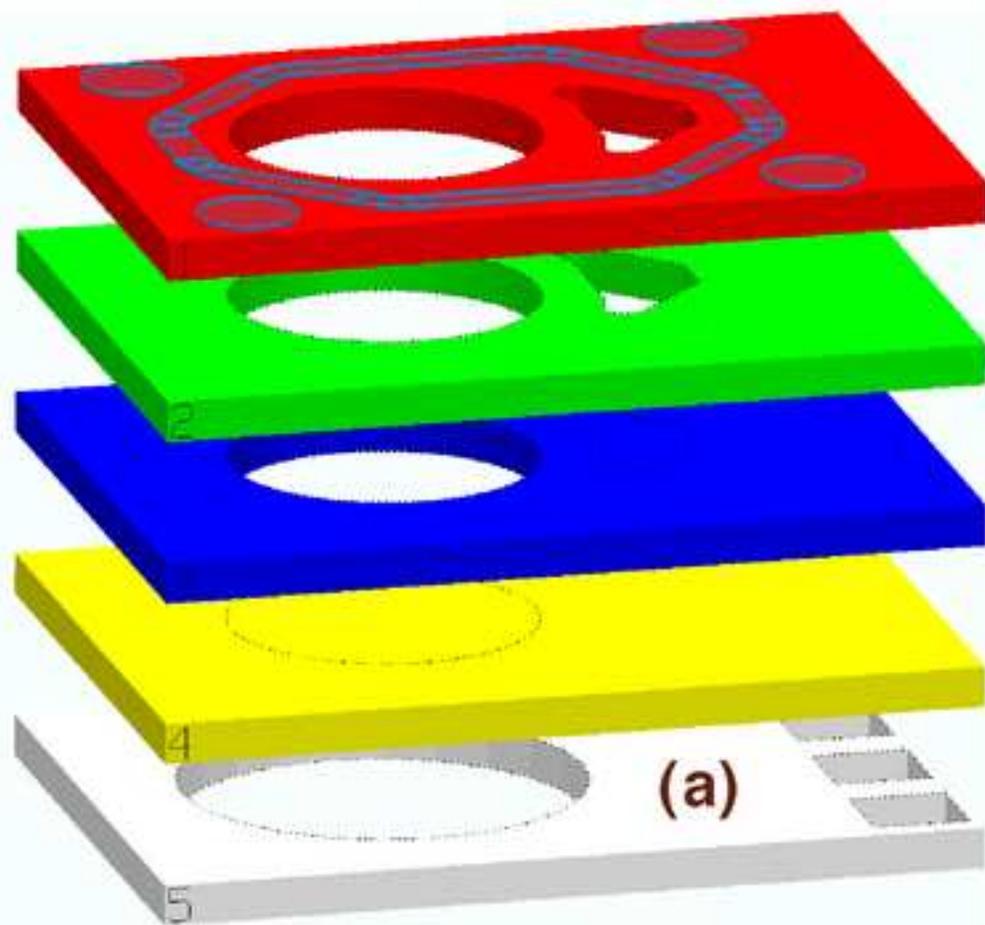


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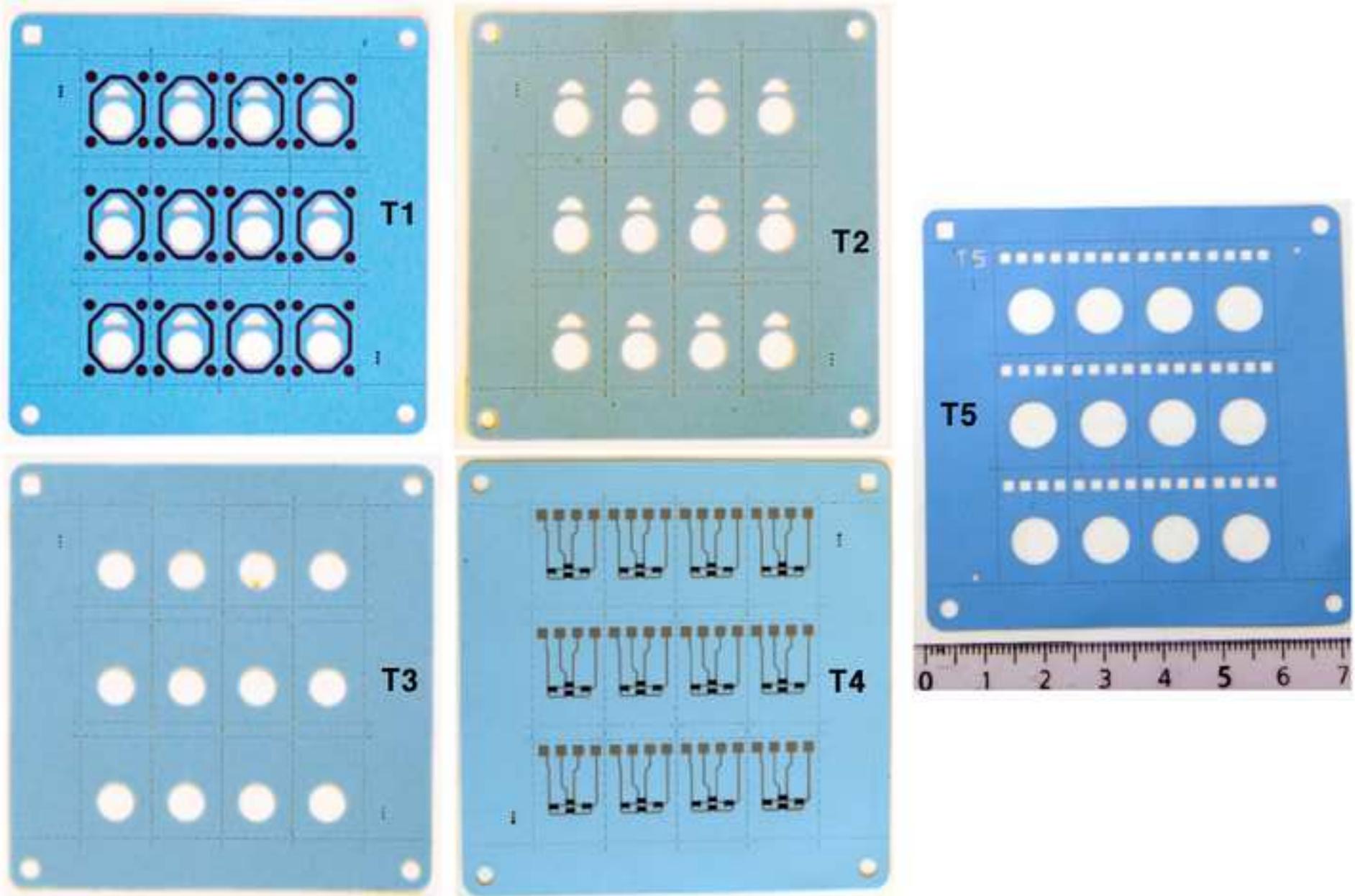


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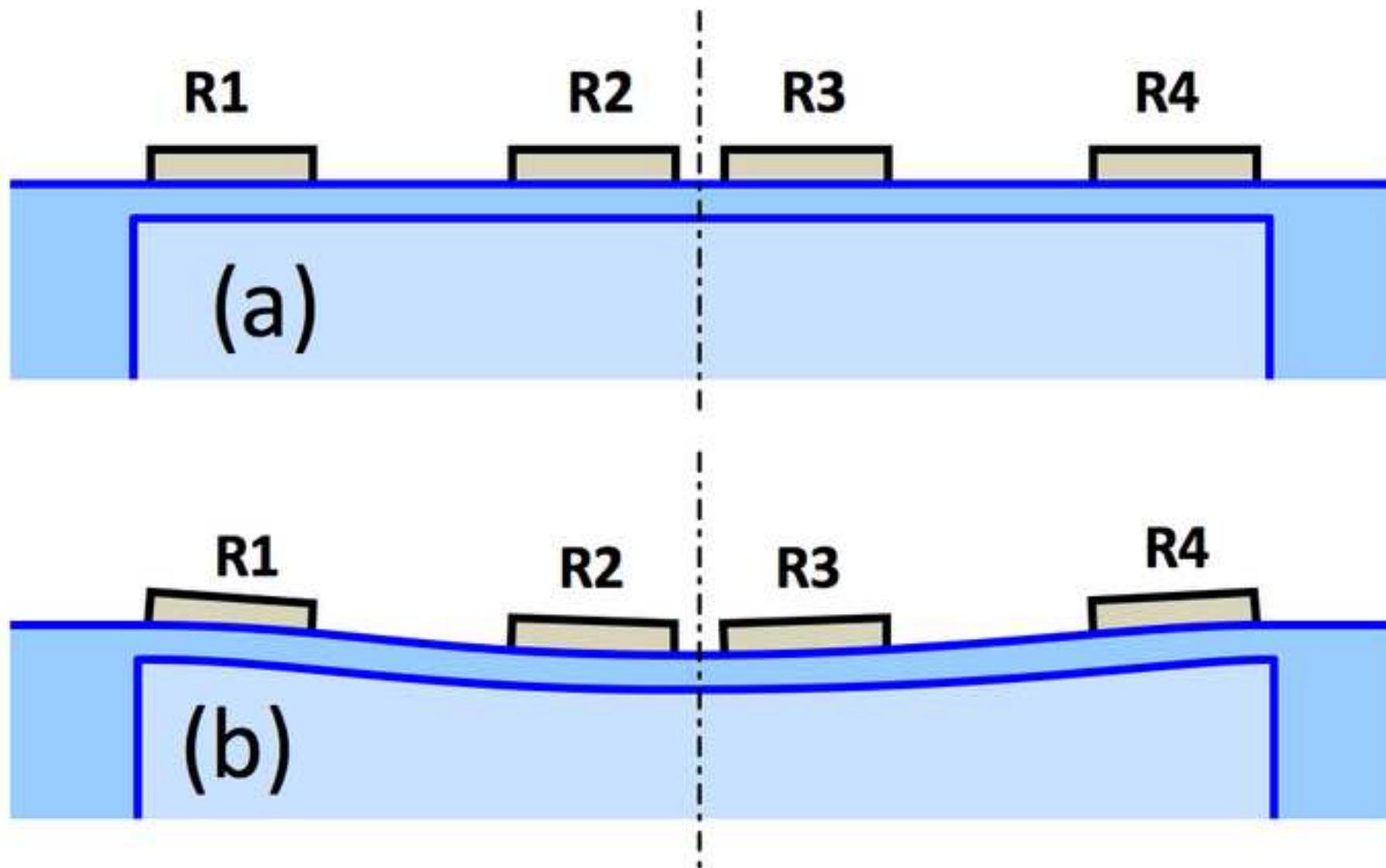


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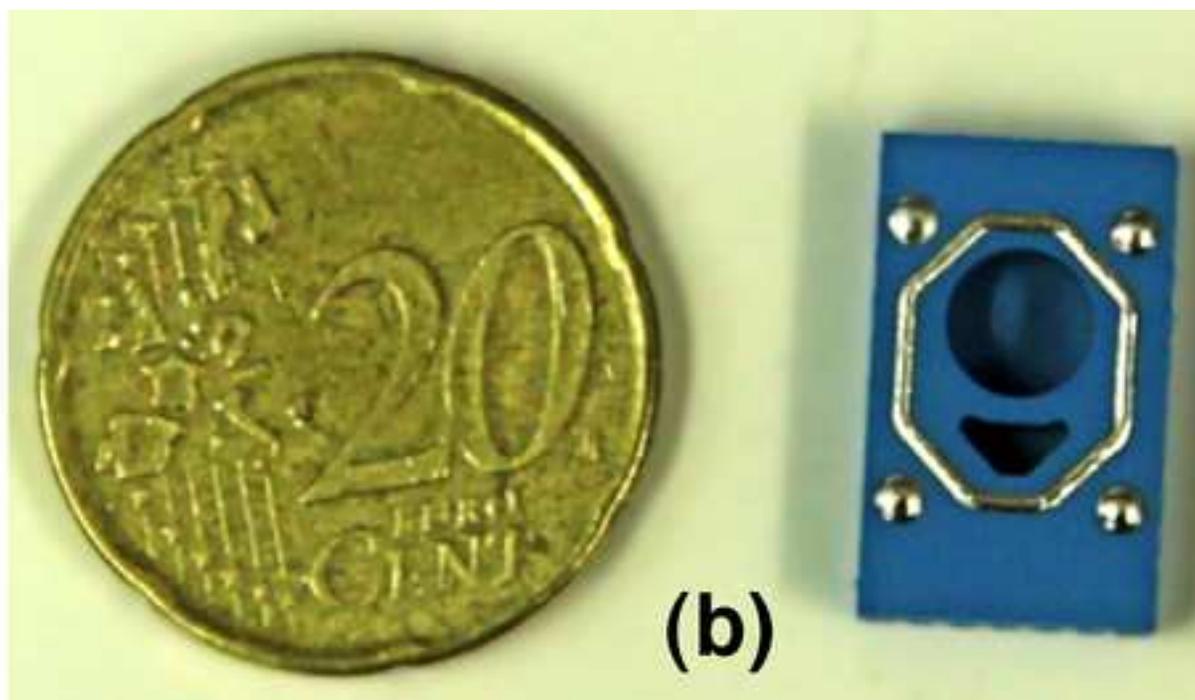
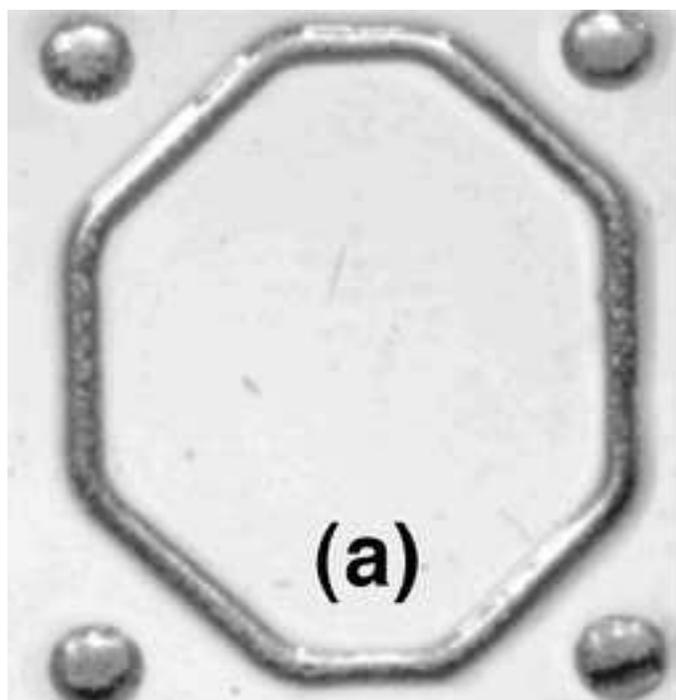


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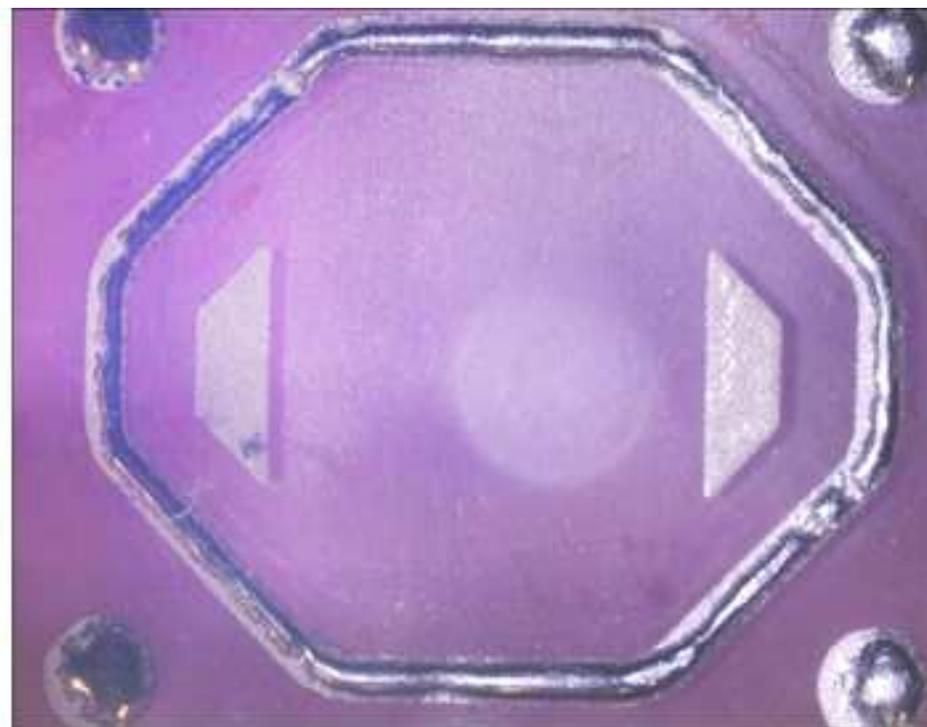
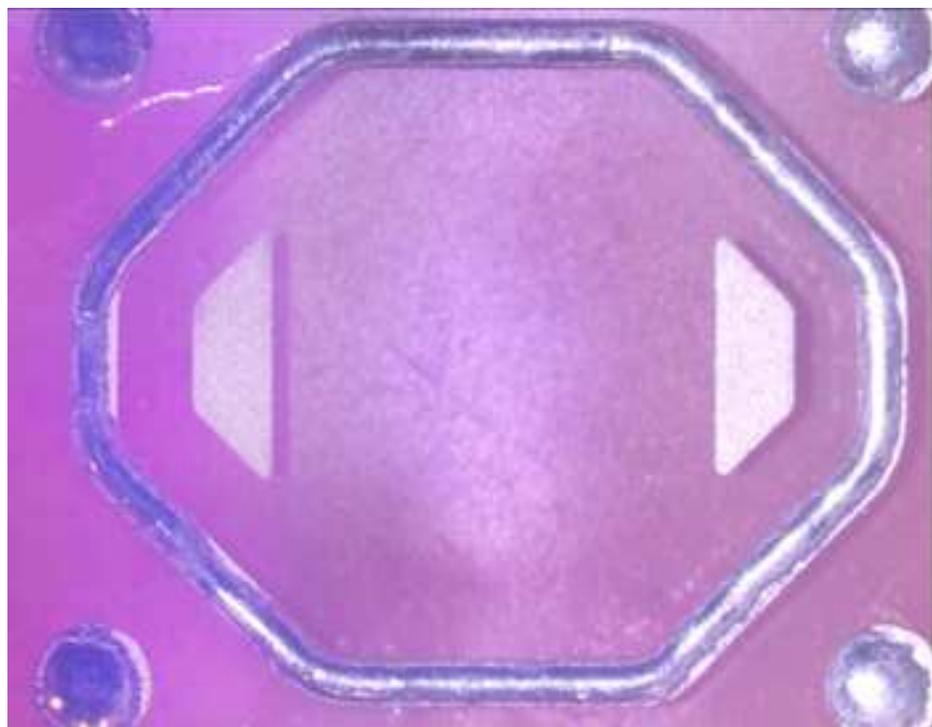


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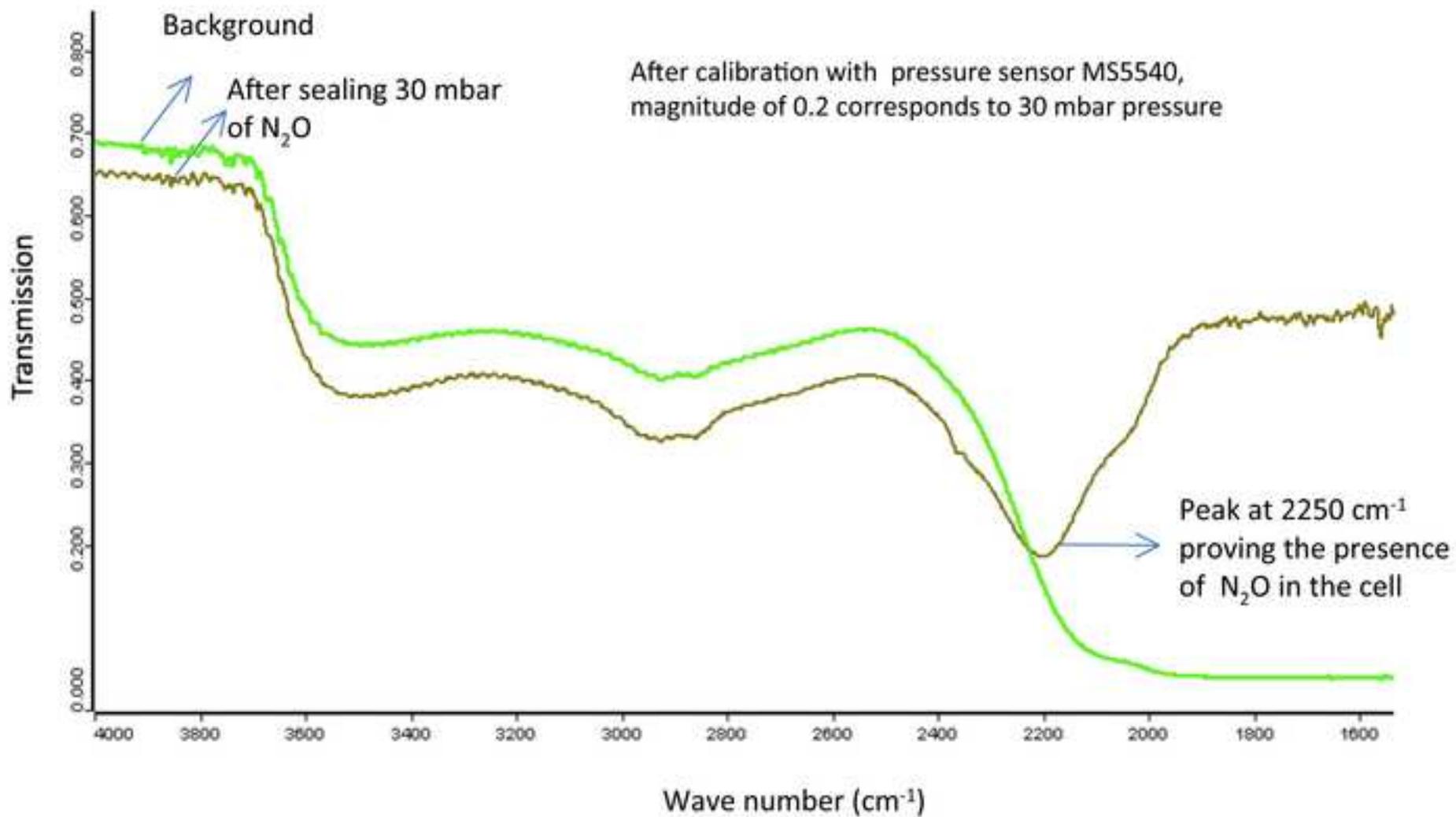


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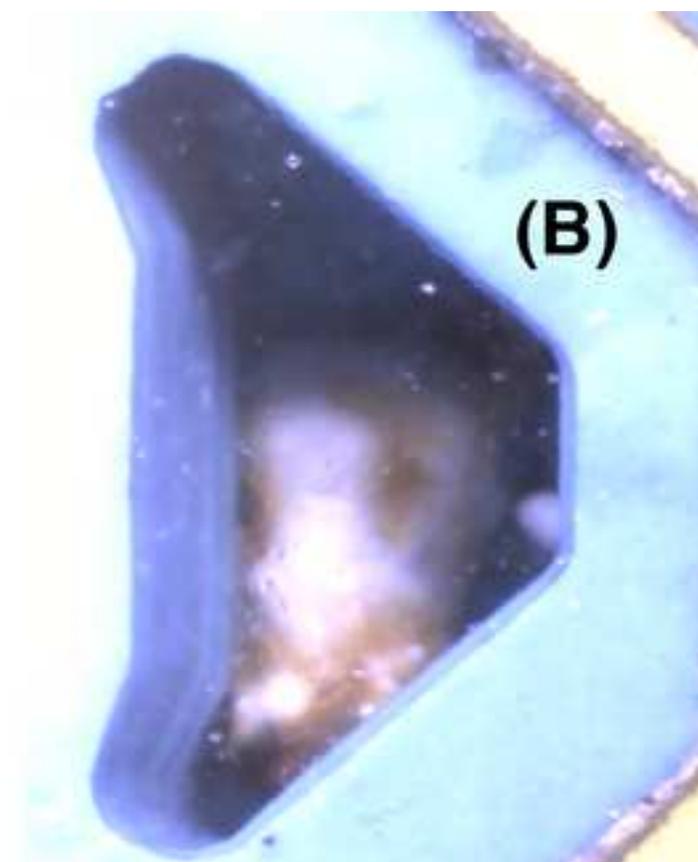
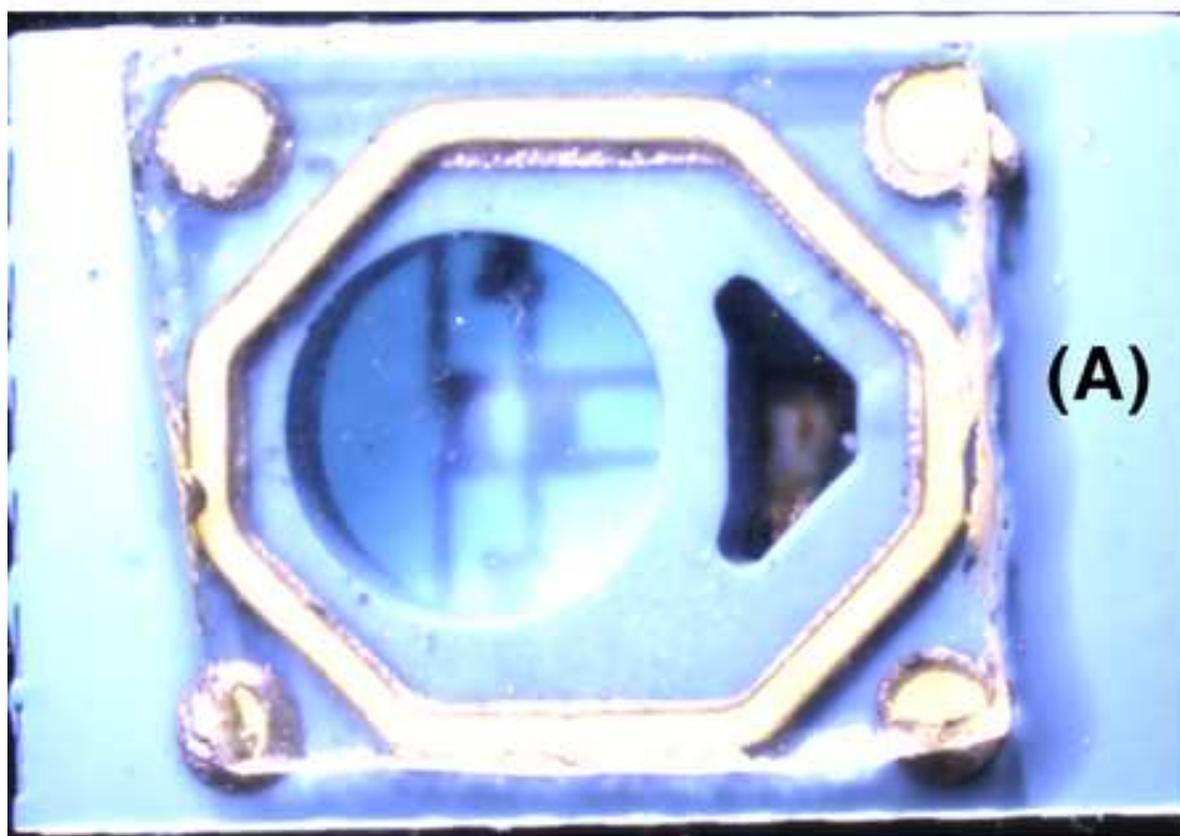
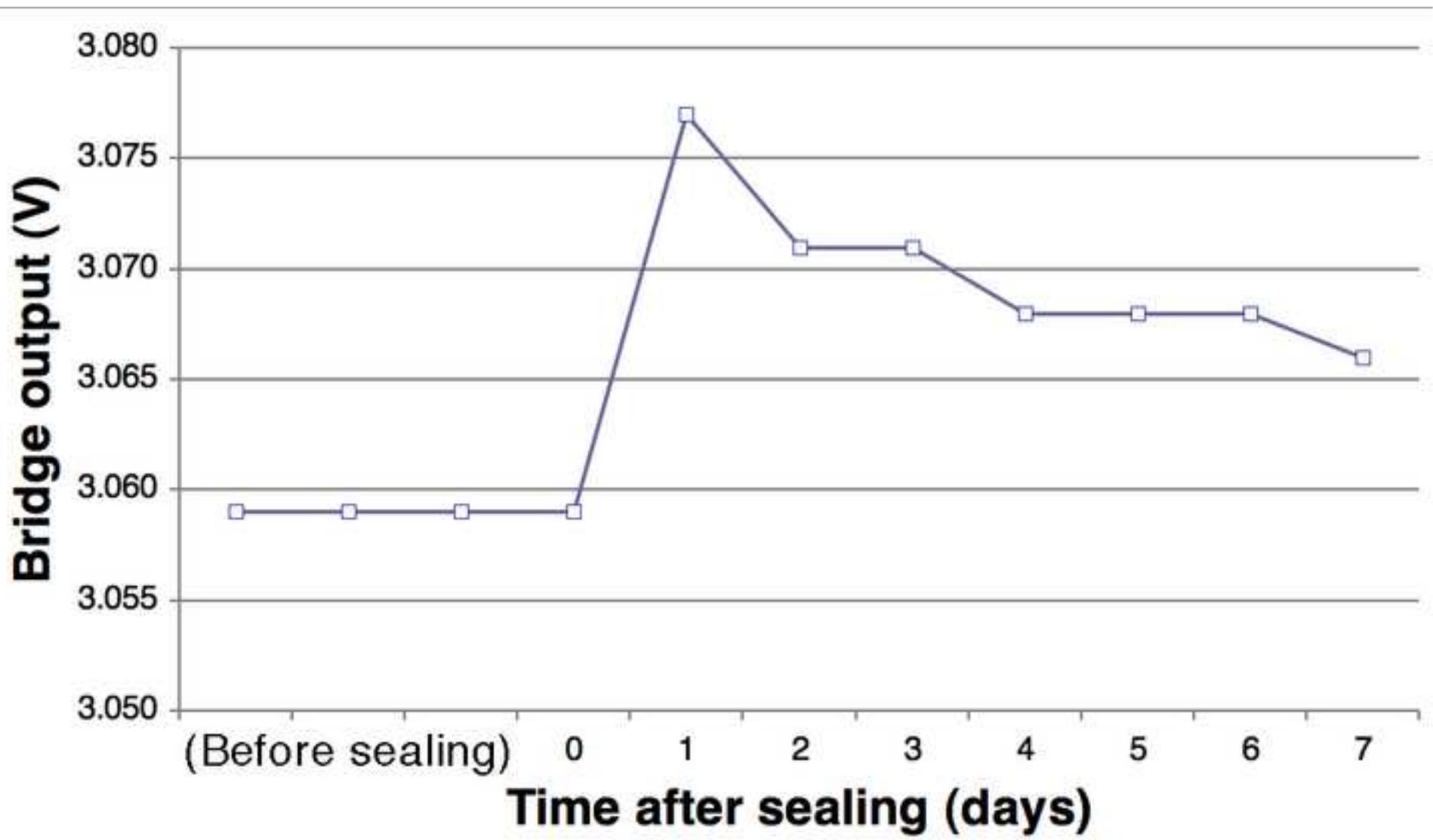


Figure 10
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Dispensing and Hermetic Sealing Rb in a Miniature Reference Cell for Integrated Atomic Clocks

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Tables

Table 1. Visual comparison of the different metallisation-solder combinations on glass.

Solder	Metallisation ESL 590G (Ag)	Metallisation ESL 9912A (Ag)
Sn-Pb-Ag	Bad wetting	Good wetting
Bi-Sn	Bad wetting	Good wetting
Bi-Sn-Ag	Good wetting	Good wetting