

## Nondestructive Raman Spectroscopy for Hermetic Package Reliability Analysis

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*The capabilities of nondestructive Raman spectroscopy for reliability analysis are demonstrated with the case study of a rubidium (Rb) gas cell.*

Hermetic packages are mandatory for MEMS applications such as accelerometers or pressure sensors, as well as for implantable medical devices. A quantitative analysis (nature and partial pressure) of the gases inside such sealed cavities is thus critical for reliability assessment. A simple and nondestructive technique would thus be a valuable complement to the sensitive but quite complex and destructive residual gas analysis (RGA) method.

Confocal Raman spectroscopy was proposed as a means to analyze gases in devices having an optical access to the sealed cavity [1]. Raman is a vibrational spectroscopy complementary to infrared spectroscopy (IR). It has, in particular, the crucial capability of measuring diatomic gases, the vibrations of which are forbidden in IR but allowed in Raman spectroscopy.

We report here on the development of Raman as a practical tool to study rubidium gas cell reliability and lifetime issues. Such cells are used for miniaturized atomic clock (MAC) and developed at CSEM within the Swiss-MAC [2] project. Our cells (Figure 1) are batch fabricated and filled with metallic rubidium (Rb) by UV irradiating RbN<sub>3</sub> deposited and sealed in the cavities.

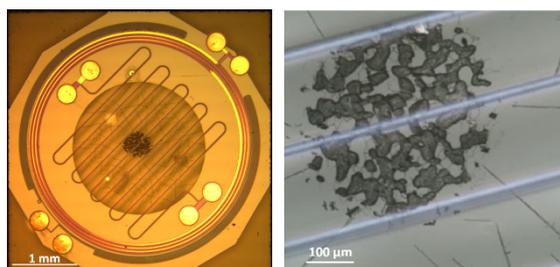
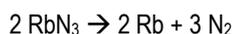


Figure 1: Pictures of the rubidium gas cell and the deposited RbN<sub>3</sub>.

The presence of metallic rubidium in the cells is assessed by measuring the absorption spectra of the Rb vapor. In some cells, a loss of the Rb signal was observed and was assumed to be related to Rb oxidation by oxygen or water i) generated during the anodic bonding process or ii) desorbed from the glass covers while operating the cell at ~ 100 °C. In order to elucidate the failure mechanism, Raman spectroscopy is used here to measure the partial pressures of nitrogen (N<sub>2</sub>), oxygen (O<sub>2</sub>) and hydrogen (H<sub>2</sub>) inside the cells. N<sub>2</sub> is present after UV irradiation due to the decomposition reaction:



The nitrogen pressure thus gives a direct indication of the UV irradiation yield. O<sub>2</sub> and water vapors cannot be present simultaneously with metallic rubidium because of the direct formation of rubidium oxides. Oxygen would, nevertheless, still be detected in cells without metallic rubidium. The presence of

H<sub>2</sub> is an indication of the presence of water inside the cell as it is a product of the reaction of Rb with water.

The confocal Raman configuration is schematically depicted in Figure 2. Thanks to an optimized calibration procedure, a detection limit of 5 mbar could be achieved. This value is sufficient with regard to i) the ~ 120 mbar of N<sub>2</sub> generated by the formation of 1 μg of Rb in the ~3 mm<sup>3</sup> cell, ii) the ~ 80 mbar of O<sub>2</sub> necessary to oxidize Rb completely, or iii) the ~ 40 mbar of H<sub>2</sub> generated by the reaction of Rb with water.

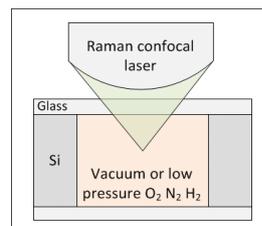


Figure 2: Confocal μRaman analysis inside the sealed cell cavity.

The absence of measurable quantities of O<sub>2</sub> or H<sub>2</sub> in our experiments led to the conclusion that formation, desorption or penetration of O<sub>2</sub> or water inside the cavity can be excluded as the main cause for the loss of the rubidium signal. A different mechanism has thus been postulated, and it is currently being further investigated.

The same Raman configuration was used in order to analyze in-situ the composition of the RbN<sub>3</sub> salt deposited in the sealed cavities. A batch of contaminated cells could thus be identified. The incriminated RbN<sub>3</sub> salt (Figure 3) was then removed from further fabrication lots.

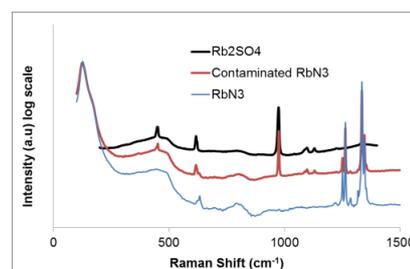


Figure 3: Raman spectra of pure RbN<sub>3</sub> and RbN<sub>3</sub> containing Rb<sub>2</sub>SO<sub>4</sub>.

Confocal Raman spectroscopy was successfully applied as a practical and nondestructive analytical method for atomic gas cell reliability analysis. The same method can thus be extended and used for the assessment of hermetic packages with an optically transparent window.

This research is performed within the frame of a multidivisional research program, and CSEM would like to thank the Swiss Confederation and the Canton of Neuchâtel for their financial support.

[1] W. H. Weber, *et al.* "Using Raman microscopy to detect leaks in micromechanical silicon structures", *Applied spectroscopy*, vol. 51, no. 1, pp. 123–129, 1997

[2] T. Overstolz, *et al.* "Highly Integrated, Miniaturized Gas Cell for Atomic Clocks," CSEM Scientific and Technical Report (2013), 28