Rubidium Packaging for On-Chip Spectroscopy

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Rubidium Packaging for On-Chip Spectroscopy

Cameron Louis Hill

A thesis submitted to the faculty of
Brigham Young University
in partial fulfillment of the requirements for the degree of

Master of Science

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December 2015

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ABSTRACT

Rubidium Packaging for On-Chip Spectroscopy

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This thesis presents rubidium packaging methods for integration using anti-resonant reflecting optical waveguides (ARROWs) on a planar chip. The atomic vapor ARROW confines light through rubidium vapor, increases the light-vapor interaction length, decreases the size of the atomic cell to chip scales, and opens up possibilities for waveguide systems on chips for additional optoelectronic devices. Rubidium vapor packaging for long-life times are essential for realizing feasibly useful devices. Considerations of outgassing, leaking and chemical compatibilities of materials in rubidium vapor cells lead to an all-metal design. The effect of these characteristics on the rubidium D2 line spectra is considered.

Keywords: rubidium spectroscopy, ARROW, on-chip, waveguide, rubidium D2 line, self-assembled monolayer, atomic vapor lifetime, atomic vapor packaging, rubidium pressure broadening, atomic vapor flow, electroplating, indium, slow light, electromagnetically induced transparency, Cameron Hill, Aaron Hawkins
ACKNOWLEDGEMENTS

I am grateful to all my fellow students and advisors that have supported me in this research. I specifically thank Dr. Hawkins for providing me with this research opportunity along with the great amount of advice and resources he made available to me. I would also like to thank Matthieu Giraud-Carrier for his frequent advice and for collaborating with me on this project. I thank Trevor Decker for his work creating the copper sulfate solutions, testing plating procedures and recording rubidium spectra for analysis. I would also like to give a special thanks to my loving wife, Christa, for her support and encouragement throughout this experience.
# TABLE OF CONTENTS

LIST OF TABLES ........................................................................................................................................ vi
LIST OF FIGURES .................................................................................................................................... vii
1 Background ........................................................................................................................................... 1
   1.1 Research Goals ................................................................................................................................. 2
   1.2 Explanation of Thesis Layout ........................................................................................................... 3
2 EIT and Slow Light .................................................................................................................................. 5
   2.1 History of EIT Experiments ............................................................................................................. 5
   2.2 Theory ............................................................................................................................................. 6
3 ARROW Platform for Atomic Spectroscopy ......................................................................................... 8
   3.1 ARROW Platform .............................................................................................................................. 9
   3.2 Rubidium Vapor Packaging ............................................................................................................ 10
      3.2.1 Chamber Attachment .................................................................................................................. 10
      3.2.2 Loading Environment and Vacuum ........................................................................................... 12
   3.3 Rubidium Vapor Flow in the Hollow Core ....................................................................................... 12
4 Rubidium Spectroscopy .......................................................................................................................... 16
   4.1 Rubidium Fine Structure .................................................................................................................. 16
   4.2 Rubidium Hyperfine Structure ......................................................................................................... 18
   4.3 Rubidium Spectral Line Shape ........................................................................................................ 18
      4.3.1 Rubidium Isotope Peak Locations ............................................................................................. 18
      4.3.2 Doppler Broadening .................................................................................................................... 19
      4.3.3 Natural Broadening ..................................................................................................................... 21
      4.3.4 Pressure Broadening and Shifts ................................................................................................. 21
   4.4 Standard Rubidium Spectrum .......................................................................................................... 22
      4.4.1 Rubidium Bulk Cell .................................................................................................................... 22
      4.4.2 Bulk Spectrum Comparison to Simulation .................................................................................. 24
5 Sealing Methods for Rubidium Compatibility ...................................................................................... 26
   5.1 Indium Versus Epoxy ......................................................................................................................... 26
   5.2 Donuts ............................................................................................................................................. 27
   5.3 Indium Soldering ............................................................................................................................... 28
   5.4 Copper Electroplating ...................................................................................................................... 28
      5.4.1 Process ....................................................................................................................................... 28
      5.4.2 Electroplating Solution ................................................................................................................. 30
LIST OF TABLES

Table 5-1: Main Ingredients in a Copper Sulfate Solution.......................................................... 32

Table 7-1: ODMS Versus DMDCS Thickness and Contact Angle.............................................. 56
LIST OF FIGURES

Figure 1-1: Setup of the integrated atomic vapor cell using the ARROW (not to scale). Rubidium is loaded into the left chamber in a vacuum and sealed. Heating the chip increases the atomic vapor density and increases the amount of rubidium atoms that can absorb the laser source and contribute to the spectrum detected by the photodetector................................. 3

Figure 2-1: The EIT three-level configuration schemes use a relatively high power coupling beam and a lower power probe beam. The probe beam contains the signal. Each scheme has a forbidden transition between two of the three states. (a) V-type scheme. (b) Λ-type scheme. (c) Ladder or cascade-type scheme. .............................................................................. 6

Figure 3-1: (Left) 3D depiction of the vapor-core ARROW. Parts are not to scale for visual purposes. The hollow core is light blue. The solid core is dark blue. (Right) ARROW chip ready for rubidium loading. ........................................ 12

Figure 3-2: Contact angle of water on a self-assembled monolayer (SAM). ........................................ 16

Figure 4-1: Rubidium fine and hyperfine splitting of the 52S1/2 and 52P3/2 orbitals and the D2 line transitions (red lines) for 85Rb and 87Rb. Each set of hyperfine splitting is to scale. Each set is not to scale to the other set......................................................................................... 18

Figure 4-2: D2 line spectrum of rubidium vapor. The 52S1/2 orbital splitting separation is shown. The total atomic angular momentum F in the ground state is shown. ......................................................................................... 20

Figure 4-3: Saturation absorption spectroscopy (SAS) involves splitting a beam and causing those beams to propagate opposite of each other in a vapor cell. Reading one of the signals causes there to be a Doppler-free dip in the absorption where the hyperfine transitions are located on the spectrum due to the pump beam saturating the atoms not moving in the beam axis direction........................................................................ 21

Figure 4-4: Rubidium bulk cell containing pure rubidium.......................................................... 24

Figure 4-5: Basic optical setup for observing spectra of rubidium vapor cells. The laser is swept around 780 nm to obtain the D2 line.................................................. 24

Figure 4-6: Rubidium D2 line spectra. The theoretical line and an actual spectral line recorded using the optical setup at BYU is shown. ........................................ 25

Figure 5-1: Chrome and nickel donuts are placed around the hollow core openings for indium soldering (left). The chrome and nickel are deposited using an electron beam vapor deposition (right). In an e-
beam deposition, electrons are directed at the source using a magnet. The source is vaporized and deposits onto the substrate. ................................................................. 28

Figure 5-2: Copper is oxidized at the anode and reduced at the cathode forming a thin layer of copper. (Left) Basic copper sulfate plating diagram. (Right) Copper plating the geometries of the copper stub on the ARROW calls for copper sulfate to flow through the anode of the electroplating system. ............................................................................................................. 30

Figure 5-3: Copper sulfate is flowed down the center of the anode of the electroplating system (a copper tube). The cathode is the copper stub and the indium. ................................................................................................................................. 30

Figure 5-4: Thick plated copper peeling off of the indium ................................................................................................................................. 33

Figure 5-5: Minicells are used to test rubidium compatibility. (left) Minicell with just rubidium and nitrogen inside. (right) Minicell with silver epoxy adhesive sample and rubidium inside. ........................................................................................................... 35

Figure 5-6: (left) Copper plated indium. This sample also has a nickel layer in between the copper and the indium. The plating recipe used here contains MPSA. (right) Minicell containing an adhesive sample. ................................................................. 36

Figure 5-7: (left) Silver epoxy on chip. (right) Indium on chip. ................................................................................................................................. 36

Figure 6-1: Optical setup used to obtain spectra for this research. ......................................................................................................................... 40

Figure 6-2: Plotted $^{87}$Rb F=2 D2 line peak FWHM vs. N2 pressure at ~70 °C. The equation of the linear fit is shown. The slope is 27 MHz/torr. The slope is roughly consistent with the slope of 19.3 MHz/torr from reports by Kluttz et al. .................................................................................................................................... 41

Figure 6-3: Epoxy minicell spectra on the same day of the test and after 18 days. Little broadening is apparent on the same day, but after 18 days, there is a lot of broadening. ................................................................................................................................. 43

Figure 6-4: Spectrum of copper-plated silver epoxy on select days over 35 days. All the spectra is shown in the appendix. ................................................................................................................................. 43

Figure 6-5: FWHM of the $^{87}$Rb F = 2 peak marked with a green circle in the previous spectra. The fail threshold was chosen to be above data noise. ................................................................................................................................. 44

Figure 6-6: Rubidium D2 line spectrum over time. The first spectrum is the shallowest. The spectrum deepens over 2 hour increments as shown and then settles. The hotplate was set to 70 °C, but the actual temperature in the minicell may have been close to 58 °C as is simulated.......................................................................................................................................................... 48
Figure 6-7: Rb D2 line of the minicell with epoxy present. The spectrum starts out peak and gets shallower and broader every 2 hours after. ................................................................. 49

Figure 6-8: FWHM of peak 1 over 5 days in stable conditions. The bulk cell ideally is flat. On the left, the bulk cell has noise that also appears on the other cells (system noise). On the right, the minimum value of the bulk cell is taken as 0 and subtracted from each of the 3 signals. This gives smoother data revealing a curved trend in the FWHMs (and a flat bulk FWHM trend). .............................................................................................. 50

Figure 6-9: Absorption depth of peak 1 of the rubidium D2 spectral line. Depth increases rapidly for In-Ni-Cu and levels off. The bulk cell stays rather steady and the epoxy decreases almost linearly. ................................................ 51

Figure 6-10: Number of data points used for signal averaging over the testing time. No clear trend is evident besides an average step down in days 1, 3, and 4. ........................................................................................................................................ 52

Figure 7-1: Hydrophobicity of DMDCS and ODMS monolayers.............................................. 56

Figure 9-1: (a) Double stub design. (b) Single stub design. This design minimizes the distance rubidium needs to transport through hollow core to be in the laser interrogated portion of the waveguide. .............................................. 60

Figure A-1: Minicell control spectra over 35 days. ...................................................................... 67

Figure A-2: Minicell with copper-plated silver epoxy spectra over 35 days................................. 68

Figure B-1: (left) Rubidium D2 line peak 2 width over 5 days in continuous tests. (right) Peak 2 width over 5 days in continuous tests. ................................................................. 69

Figure B-2: (left) Rubidium D2 line peak 3 width over 5 days in continuous tests. (right) Peak 3 width over 5 days in continuous tests. ................................................................. 69
1 BACKGROUND

Rubidium atomic vapor has optical properties useful for many nonlinear optical experiments. One commonly researched experiment is electromagnetically induced transparency (EIT) [1], a mechanism often used for slow light [2]. Methods and structures for integration of waveguides capable of slow-light include photonic crystals [3], band-edge waveguides [4], microresonators [5], metamaterials [6], and stimulated Raman scattering (SRS) [7]. This thesis describes the process of integrating a rubidium-vapor-filled waveguide on a chip using the ARROW platform.

Integrating waveguides capable of EIT and slow light opens up many possibilities for optical communication and processing. EIT and slow light is used in many optical devices and experiments such as optical switches [8], optical traps [9], coherent optical memory [10], four-wave mixing devices [11] and improved interferometers [12]. Integration would allow for systems of such devices to be combined onto a chip.

Optical communication network devices use electronic circuits to synchronize, buffer, switch, and perform time-division multiplexing. All-optical devices based on slow light could replace these electronic circuits and lower error rates, power consumption, and cost inherent in optic-electronic conversion circuitry. All-optical devices may also increase bandwidth capabilities [13].

Of the means for slow light, atomic vapor has been shown to have high potential in all-optical image processing. In [14], images carried by optical pulses are delayed by up to 10 ns
using cesium vapor. They preserve the transverse phase and amplitude of the image thus increasing signal-to-noise ratios at low light levels.

Atomic-vapor slow light also has potential in single photon communication networks. Controllable single photon transmission and storage is crucial for quantum networks where memory nodes use atoms to process and store quantum states. Tunable single-photon pulses have been demonstrated using EIT and slow light in rubidium vapor [15].

1.1 Research Goals

The research presented in this thesis is for obtaining an integrated light-confining rubidium vapor cell using hollow-core anti-resonant reflecting optical waveguides (ARROWs). A not-to-scale depiction of the basic layout of the integrated vapor cell using the ARROW is shown in Figure 1-1. The light-vapor interaction length is potentially 4 mm depending on the diffusion depth of the rubidium vapor. This thesis presents the inherent difficulties of inserting rubidium into cells placed atop the waveguide entrance openings in a way that allows high rubidium vapor density in the channel and low amounts of contaminating and/or reacting materials. Much research has been performed based on this rubidium-vapor core ARROW by previous students at BYU [16, 17].

Specifically, this research is to determine if a method of copper-plating indium described in [16] can be applied on the atomic vapor cell ARROW chip. The copper coating is meant to protect the rubidium vapor from reacting with the indium. This research applies the procedures described in [16] and suggests additional procedures/recipes for obtaining an effective copper coating on indium.
This research also considers the effect of contaminants on the rubidium D2 line spectra. The majority of contaminants is considered to be from the sealant used to attach the chambers to the ARROW chip. The effects on pressure and Gaussian broadening of the rubidium D2 line peaks are analyzed. The conclusions in this thesis can apply to changes in the rubidium D1 line spectra as well.

1.2 Explanation of Thesis Layout

Chapter 2 will explain what EIT and slow light is and the basics concepts behind how it works. EIT by means of rubidium atomic vapor will be discussed. Chapter 3 will explain the platform for containing rubidium vapor on a chip in a way that a laser can be propagated through the vapor. The explanation will specifically apply to 780 nm light which corresponds to the rubidium D2 line. Chapter 4 will explain the rubidium D2 line spectrum. This will be a background to the tests used to analyze the effect of packaging methods on the rubidium vapor.
Chapter 5 will explain specific packaging methods. Indium as a rubidium chamber adhesive will be considered as it has preserved the rubidium spectrum well in the past. Chapter 6 will present some tests showing how several different adhesives perform. Chapter 7 will explain some results on wall passivation for the platform used for this thesis. The wall passivation is another method used to preserve the rubidium spectrum on chip. Chapter 8 will summarize the conclusions of this thesis. Chapter 9 will explain future work that is going to be worked on or is suggested should be performed.
2 EIT AND SLOW LIGHT

The concept of slow light is the significant lowering of the group velocity of an optical modulation in an electromagnetic wave. Slow light is achieved by obtaining high dispersion within a narrow, low-loss, spectral region. Means of achieving slow light include electromagnetically induced transparency (EIT) [18], coherent population oscillation (CPO) [19], and coupled resonator optical waveguides (CROW) such as photonic crystals [20, 21]. EIT and CPO rely on controlling the electron configurations of a material with multiple laser sources while CROWs use periodic structure waveguides to couple waves between resonators. EIT has shown the ability to slow light to a stop [22]. EIT is considered more difficult to implement on-chip than CROWs [21]. In my research, I consider the implementation of EIT on-chip using atomic vapor cells integrated with the ARROW waveguide.

2.1 History of EIT Experiments

EIT was first observed using a strontium vapor in 1991 [23]. A group led by Lene Hau at Harvard University reduced the group velocity of light to 17 m/s using EIT and Bose-Einstein condensates (BECs) in 1998 [24]. Hau led the same group to obtaining stopped light two years later [25]. In 2007, they demonstrated stopping light and starting it again preserving the amplitude and phase of the stopped wave [26]. They used BECs for their experiments. BEC involves cooling boson gas particles to near absolute zero temperature. For this reason, warm rubidium is more feasible for integrated EIT. Rubidium vapor has been used to slow light to 8 m/s using EIT [27].
2.2 Theory

EIT is using electromagnetic waves to cause a material to increase transmission at a specific frequency. The electromagnetic waves block electrons from transitioning between certain states. There are various schemes commonly used. Often, these schemes involve three electron levels. Of the three transitions between the three levels, only two are allowed. The schemes are shown in Figure 2-1. They are called the V-type, Λ-type and the ladder or cascade-type systems. A comparison of the various schemes and their uses are given in [28].

As an example, consider the Λ-type scheme in the figure above. |1> is the ground state. The transition from |1> to |2> is forbidden. |3> is the only decaying state, so it is the only way to absorption. As a result, the two most likely ways for coupling beam absorption are the direct |1> - |3> path and the indirect |1> - |3> - |2> - |3> path. The |3> - |2> - |3> path can continue to repeat. The coupling beam is made so strong compared to the probe beam that the second path (with however many |3> - |2> - |3> path repeats) essentially has the same probability amplitude as the first path. The probability amplitude is also opposite in sign. This leads to the probe beam
not being absorbed by the material (transparency). These configurations are considered in more
detail in [29].

Various three-level schemes have been used with rubidium vapor. The next section will
explain specific electron transitions in the rubidium atom such as those used for EIT [1, 30].
These excitations are analyzed using rubidium spectroscopy.
3 ARROW PLATFORM FOR ATOMIC SPECTROSCOPY

The 3 level schemes used for EIT can use levels such as the ones available in rubidium vapor. This chapter will discuss confining rubidium into a waveguide core in a way that will increase the ability to use the orbital structure of rubidium to obtain the 3 level scheme in EIT. The next chapter will explain where the peaks of the rubidium D2 line which originate from energy levels that can be used for the 3 level scheme.

In order to propagate laser light through rubidium vapor on a chip, an integrated hollow-core waveguide is needed. A waveguide confines the light to a single mode so that the interaction with the rubidium vapor is increased. Integrated waveguides allow for designs that involve multiple waveguides that can be combined to make devices discussed in chapter 1 on a single planar chip.

This chapter discusses the (anti-resonant reflective optical waveguide) ARROW design and how it is used to confine rubidium vapor and light. The first section will cover why the ARROW is used for this thesis and the basic dimensions of it for guiding at the wavelength for the rubidium D2 line. Section 3.2 discusses how the rubidium is packaged onto the chip. Section 3.3 discusses how the rubidium is expected to flow down the hollow core given the packaging methods used.
3.1 ARROW Platform

Conventional optical waveguides have a cladding material index that is lower than the core material index resulting in total internal reflection (TIR). Since the core will be filled with a vapor, its index will be very close to 1. In order to guide the light through a low-index core, a design that does not rely on TIR is needed. The ARROW (anti-resonant reflective optical waveguide) design meets these qualifications. The ARROW has the advantage of being made using conventional silicon microfabrication processes [31]. This allows fabrication to be done in BYU’s Integrated Microfabrication Lab and for efficient design changes.

The ARROW uses cladding layers of low-loss materials with specific thicknesses to create the light guiding characteristics of the waveguide. The specific thicknesses cause the layers to act as anti-resonant Fabry-Perot (FP) etalons in the transverse direction at specific wavelengths. The antiresonance in the transverse direction is what guides the light into the core [32]. As little as one layer can be used. More layers are used to improve transmission. The layer thickness are calculated using [31]

\[ d_i = \frac{\lambda}{4n_i}(2N + 1) \left( 1 - \frac{n_c^2}{n_i^2} + \frac{\lambda^2}{4n_i^2 d_c^2} \right)^{-1/2} \]

\( \lambda \) is the desired wavelength. \( n_i \) is the index of the material. \( N \) is the order. \( n_c \) is the index of the core. \( d_c \) is the core thickness. A core index of 1 is used as a very close approximation for the nitrogen and rubidium vapor that will fill the core. The core thickness is generally between 5.5 and 6 µm.

Silicon dioxide and silicon nitride are common thin films deposited in microfabrication labs such as the BYU IML. PECVD oxide and nitride have indexes of 1.46 and 2.05
respectively. Equation 3.1, with \( \lambda = 795 \) nm (for the rubidium D1 transition) and \( d_c = 5.8 \) \( \mu \)m, results in a 111 nm nitride and 186 nm oxide for the bottom layers to maximize transmission.

Using rubidium’s D2 transition wavelength of 780 nm increases the thickness by approximately 3 nm. In the past, ARROWs have been made in BYU’s IML using 3 sets of oxide and nitride layers on bottom and top [16]. To simplify processing, the bottom layers consist of sputtered Ta\(_2\)O\(_5\) and SiO\(_2\) deposited by a commercial company. One top oxide layer, 6 \( \mu \)m thick, is used for further simplification. Ta\(_2\)O\(_5\), having an index of 2.275, requires a thickness of 95 nm for ARROW guiding.

The chip also consists of a solid core waveguide to couple the laser into the hollow ARROW core. The solid core portion of the waveguide uses (TIR) with the interface to ARROW layers on the bottom and the air on the sides and top to guide light.

### 3.2 Rubidium Vapor Packaging

In order to place rubidium vapor over the hollow cores that guide the vapor to the portion of the waveguide where the laser will propagate, chambers are attached to the chips, filled with rubidium, and then sealed.

#### 3.2.1 Chamber Attachment

Many adhesives have been considered for compatibility with rubidium [16]. The main ones considered in this thesis are Aremco 2310, indium solder, and silver epoxy.

Aremco 2310 epoxy is used because it has been tested and used many times for ARROW atomic cells and minicells and its effect on the rubidium D2 line spectrum is minimal over the first couple of weeks. Aremco 2310 is a high strength, high temperature, flexible epoxy. A further description is available on Aremco’s website [33].
Alternatives to Aremco 2310 epoxy have been sought in order to prolong the lifetime of the ARROW atomic cell. For this reason, copper-plated indium solder is considered in this thesis. Indium is applied by heating it up to its melting point and using flux to clean the metal surfaces that it will adhere to. The flux also serves to clean off the indium oxide that readily forms on the indium wire. Copper is plated onto the indium to protect the rubidium from reacting with it. Copper-plated indium has been found to produce long lasting compatibility results when used as the adhesive for the copper stubs on the ARROW atomic cell [16]. Details about chamber attachment using indium plated copper are given chapter 5.

Over concern that indium heating and cooling on the chip may be harmful to the cores on the waveguide, silver epoxy is also considered in this thesis. Silver epoxy is much more easily applied to the chips relative to indium solder. Silver epoxy, being conductive like indium, has the advantage of being able to be plated with copper using electroplating. Coverage is minimal.

Rubidium compatibility results using these adhesives are presented in chapter 6.

Figure 3-1: (Left) 3D depiction of the vapor-core ARROW. Parts are not to scale for visual purposes. The hollow core is light blue. The solid core is dark blue. (Right) ARROW chip ready for rubidium loading.
3.2.2 Loading Environment and Vacuum

As an alkali metal, rubidium is very reactive, especially with water and air. All handling of rubidium is usually done in an inert-gas-filled glove box. Rubidium is a silver-looking substance that melts at just above room temperature (39.48 °C). Standard procedure for loading the glove box with chips is to place the chips inside the loading chamber of the glove box and to vacuum it down to single-digit torr pressures and purge the remaining air with nitrogen. Several purge cycles take place over 1 to 3 hours. Then the loading chamber can be opened from inside the glove box and make available its contents.

Rubidium is heated up slightly to its low melting point so it is removable from its glass container. A steel syringe is then used to gather up the removed rubidium after it solidifies. The stickiness of the rubidium is advantageous as it stays on the syringe until it is placed inside the copper stub on the chips.

The loaded copper stub of a chip is then inserted into elastic tubing that is attached to a vacuum source. The vacuum is opened and brings the copper stub to a vacuum that generally reads 0 to 2 mTorr at minimum vacuum. It is accepted that the vacuum used at BYU may range from 0.1 to 2 mTorr at these readings. The copper stub is then crimped using a copper cold-weld crimping tool. If the pressure reading stays the same then it is assumed that the crimp effectively hermetically sealed the copper chamber. The unloaded copper stub of the same chip then undergoes the same procedure without the rubidium unless it has been filled with epoxy to plug the other end. This may be done if a rubidium pressure differential is deemed unnecessary.

3.3 Rubidium Vapor Flow in the Hollow Core

This section is intended to explain the difficulty of causing rubidium vapor to flow down the 5 X 10 µm hollow cores of the ARROW while preserving the polarization. There is a
significant amount of research on reducing the effect of atom-wall collisions on the orbital structure of the atoms [34-36]. This section will give an understanding of the significance of wall collisions, especially inside of the capillary. These collisions are considered significant to the spectrum of the atomic vapor inside the chip.

In the systems presented in this thesis, the rubidium in a loaded chamber for spectroscopy is heated to between 60 and 90 °C. Using the equations for vapor pressure given in [37], those temperatures correspond to vapor pressures between 8 and 92 uTorr.

As discussed previously, there is about 0.1 to 2 mTorr of pressure inside the system to start. The majority of the pressure is from nitrogen as a result. The mean free path can be calculated using [38]

$$\lambda = \frac{k_B T}{\sqrt{2}p \pi d_g^2}$$  \hspace{1cm} 3.2

Where $k_B$ is the Boltzmann constant, $p$ is the pressure, and $d_g$ is the effective diameter of the atoms. Ranging $T$ from 60 to 90 C, $p$ from 0.1 to 2 mTorr, and using $d_g$ equal to 496 pm (the diameter of rubidium), the mean free path ranges from 16 to 344 µm. The Knudsen number can then be calculated using [38]

$$K_n = \frac{\lambda}{d_p}$$  \hspace{1cm} 3.3

where $d_p$ is the size of the capillary. Using the smaller dimension of the ARROW core of 5.8 µm, $K_n$ ranges from 3 to 69 for rubidium. A Knudsen number greater than 10 means collisions between gas molecules and the walls of the ARROW are more dominant than between molecules. A Knudsen number below 0.1 means wall collisions are more dominant. Therefore, we are only somewhat concerned with interatomic collisions and are more concerned with gas-wall collisions when considering rubidium vapor flow through the ARROW core.
**ARROW Hollow Core Wall Passivation**

In order to decrease the probability of spin depolarization at each wall collision in the ARROW core, wall coatings for inside the core are considered. The spin depolarization can hinder the strength of the peaks of the rubidium spectrum.

Buffer gases help reduce depolarization, but contribute to collisional broadening. Paraffin coatings are commonly used in bulk vapor cells and are found to allow up to 10,000 atom-wall collisions before depolarization. Paraffin wax, though, is too thick for use in the ARROW core. Paraffin wax also tends to lose performance at temperatures above 60 °C [39].

Other coatings have been explored as a result, including polydimethylsiloxane (PDMS) [40], and self-assembled monolayers (SAMs) such as octadecyltrichlorosilane (OTS) [41, 42] (the latter source using OTS in microstructures), and octadecylmethyldimethoxysilane (ODMS or ODS) (in optical fiber) [43]. ODMS has been used here at BYU with preparation and insertion-in-ARROW methods being described in [16].

OTS is the same as paraffin wax in structure except that it has a silane head that attaches to glass or silicon dioxide surfaces. OTS has a performance of 2100 collisions before atomic relaxation (using potassium vapor), but tends to be inconsistent. Heavier alkali atoms generally produce lower performance. Since OTS and paraffin wax are similar, it is thought that the poorer performance of OTS is due to poor surface coverage. This may also explain varying performance results. A way to characterize OTS performance is described in [41].

One way to measure the effectiveness of a SAMs such as silane SAMs is to measure the hydrophobicity of the monolayer surface. This is done by placing equal amounts of water on the layer in the form of a droplet. The angle is then measured using a contact angle goniometer. This is a basic instrument that uses a microscope with a goniometer attached. Theoretically, the angle
of the water droplet contact angle with a surface can range between 0 and 180°. If the angle is less than 90°, then the surface is hydrophilic. If the angle is greater than 90°, then the surface is hydrophobic.

The hydrophobicity of DMDCS and ODMS were tested at BYU. Results are given in chapter 7.

![Diagram of contact angle of water on a self-assembled monolayer (SAM).](image)

**Figure 3-2: Contact angle of water on a self-assembled monolayer (SAM).**
4 RUBIDIUM SPECTROSCOPY

This chapter will consider the orbital structure of the rubidium that is excited in the hollow-core of the ARROW and how it relates to the rubidium D2 spectrum. The some of the peaks of the spectrum originate from energy levels that have been used to obtain EIT using hot rubidium vapor. The more the spectrum is preserved, the more accurate the EIT experiments will be.

4.1 Rubidium Fine Structure

Rubidium’s single electron in its outer shells makes for a ground to excited state orbital system that is predictable and useful in optical experiments such as EIT [1, 30]. Rubidium is an ideal alkali-metal element because of its high vapor pressure at room temperature. Also, the D1 and D2 lines of rubidium occur at wavelengths that are available using relatively inexpensive laser sources [37]. The D1 line is at 795 nm and the D2 line is at 780 nm.

The rubidium D1 and D2 lines are made up of the $5^2S_{1/2} \rightarrow 5^2P_{1/2}$ and $5^2S_{1/2} \rightarrow 5^2P_{3/2}$ transitions respectively. The splitting of the 5P orbital is due to interaction between the orbital angular momentum $L$ and the spin angular momentum $S$ of the electron. The interaction creates two possible total angular momentums, $J = 1/2$ and $J = 3/2$. The $5^2P_{1/2}$ and $5^2P_{3/2}$ states make up what is called a fine-structure doublet [44].
Figure 4-1: Rubidium fine and hyperfine splitting of the 5S1/2 and 5P3/2 orbitals and the D2 line transitions (red lines) for 85Rb and 87Rb. Each set of hyperfine splitting is to scale. Each set is not to scale to the other set.
The D2 line transitions for $^{85}\text{Rb}$ and $^{87}\text{Rb}$ are shown in Figure 4-1. There are 6 transitions that make up the D2 line. These transitions are a result of hyperfine structure. The D1 line is similar. The differences are that the line goes to the $5^2\text{P}_{1/2}$ state and the energy jump is lower (corresponding to the higher wavelength). There are only 4 transitions that make up the D1 line [44].

4.2 Rubidium Hyperfine Structure

Hyperfine structure is a result of the total atomic angular momentum $F$. $F$ is dependent on the interaction of the total electron angular momentum $J$ with the total nuclear angular momentum $I$. $F$ in the $5^5\text{S}_{1/2}$ state can take on either 1 or 2 for $^{87}\text{Rb}$ and either 2 or 3 for $^{85}\text{Rb}$. In the D2 excited state, $F$ can take on 0, 1, 2, or 3. This would give rise to 8 possible transitions, but $F$ transitions are quantum limited. $F$ can only change by -1, 0, or 1. This means for $^{87}\text{Rb}$, the possible transitions are $F_g = 1 \rightarrow F_e = 0, 1$, or 2 or $F_g = 2 \rightarrow F_e = 1, 2$, or 3. Thus, there are only 6 possible transitions [44].

4.3 Rubidium Spectral Line Shape

The rubidium spectral line shape is a result of the transition frequency locations and the broadening of those peaks.

4.3.1 Rubidium Isotope Peak Locations

Naturally occurring rubidium consists of 72.17% $^{85}\text{Rb}$ and 27.83% $^{87}\text{Rb}$. The D2 line spectrum is shown in Figure 4-2. There are four distinct peaks in the spectrum. The outer 2 peaks are from the $^{87}\text{Rb}$ isotope. The smaller size of those peaks are mostly a reflection of the smaller proportion of $^{87}\text{Rb}$ atoms. The 6.8 GHz spread between the $^{87}\text{Rb}$ peaks are a result of the $5^2\text{S}_{1/2}$
orbital splitting as explained in section 4.2. The center 2 peaks are a result of the same orbital splitting, but of the more abundant $^{85}\text{Rb}$ isotope. Each peak consists of 3 separate peaks that are so close together, they are indistinguishable due to Doppler broadening (~0.5 GHz at room temperature). The 3 peaks are a result of the 3 possible transitions shown in Figure 4-1.

There is an associated D1 line that occurs at a wavelength of 795 nm that has a similar structure and shape. Unlike the other lines, the hyperfine structure in the D1 line is distinguishable in the spectrum for $^{87}\text{Rb}$. At 0.815 GHz, the hyperfine splitting is greater than 0.5 GHz broadening that occurs from Doppler broadening [44].

### 4.3.2 Doppler Broadening

The broadening of the peaks in Figure 4-2 is mostly a result of Doppler broadening. Doppler broadening occurs as a result of thermal motion of the atoms along the axis of the probe beam. Atoms moving toward the probe beam experience a blue shift in frequency while atoms
moving away from the probe beam experience a red shift [45]. The Maxwell-Boltzmann distribution of the beam-axis velocity of the atoms is Gaussian with a FWHM of about 0.5 GHz for the rubidium D lines at room temperature [37]. This broadening makes many of the hyperfine transitions indiscernible. A popular way to obtain spectroscopic peak locations of hyperfine spacing is by saturation absorption spectroscopy (SAS).

Figure 4-3: Saturation absorption spectroscopy (SAS) involves splitting a beam and causing those beams to propagate opposite of each other in a vapor cell. Reading one of the signals causes there to be a Doppler-free dip in the absorption where the hyperfine transitions are located on the spectrum due to the pump beam saturating the atoms not moving in the beam axis direction.

In SAS, a pump beam is taken from the probe source beam and redirected to go in the opposite direction of and same path as the probe beam in the vapor cell. The pump beam generally will be of higher intensity than the probe beam. The pump beam excites the atoms in its path. The ones that do not experience a Doppler shift will appear transparent to the probe beam and will not affect the spectrum. The atoms experiencing a red shift to the pump beam experience a blue shift to the probe beam and vice versa. They will still be absorbed. This results in what are called “Lamb dips” at each transition in the original Doppler broadened spectra [45].
4.3.3 Natural Broadening

Natural broadening is a result of Heisenberg’s uncertainty principle. The shorter the excited lifetime of a particle the more uncertain the energy is. At rubidium D line energy levels, this broadening is somewhat insignificant in the presence of Doppler and pressure broadening. This broadening results in a Lorentzian lineshape [46].

4.3.4 Pressure Broadening and Shifts

Pressure or collisional broadening is another common cause of peak broadening in atomic vapor cells. At wavelengths of 780 nm, pressure broadening is relatively insignificant below 1 torr. Above 1 torr, pressure broadening becomes increasingly significant. Around 25 MHz/torr is a typical amount of collisional broadening. Doppler broadening contributes about 500 MHz to the linewidth at room temperature.

Pressure broadening causes a Lorentzian line shape and a peak shift. The overall peak of a transition is formed by the convolution of the Gaussian shape from Doppler broadening and the Lorentzian shape formed from natural and pressure broadening. This creates a Voigt profile [47].

The relationship between the pressure of specific gases such as nitrogen, the noble gases, and several hydrocarbons and peak broadening and shifts are well documented for the rubidium D1 line. The shifts range from -10 to 6 MHz/torr while the broadening rates range from 20 to 34 MHz/torr [48]. For the rubidium D1 and D2 lines, broadening rates are given for several hydrocarbons and He in [49]. Additional rates for N₂, ³He, and Xe broadening and shifts are given in [50]. [51] adds rates for D2, CF₄ and he nonradioactive noble gases. All of these are taken at temperatures varying from 293 K to 394 K and some of them attempt to compare data by converting rates by temperature dependent relations they fit to. The results are reliable for
many purposes. All the rates stay below 34 MHz/torr and are very linear. One source scales to temperature using [49]

$$\gamma_{B,S}^{ADJ}(T_2) = \gamma_{B,S}(T_1) \left( \frac{T_1}{T_2} \right)^\Omega.$$  \hspace{1cm} (4.1)

This equation applies to broadening (B) and shifts (S). $\gamma^{ADJ}$ is the adjusted rate. $T_1$ is the temperature at which the broadening was measured and $T_2$ is the temperature at which the rate was taken. $\Omega$ is usually 0.5, but may vary depending on assumptions made about the type of interactions that occur at atomic collisions.

In [52], Kluttz et al. fits data to a line that is dependent on temperature. They give their measured parameters for the slope dependence on the temperature, the coefficient of the slope and the y-intercept. They advert that their broader definition of the slope dependence on temperature is not found to be consistent with [50]. The slope dependence on temperature is usually much less than 10%.

### 4.4 Standard Rubidium Spectrum

#### 4.4.1 Rubidium Bulk Cell

The rubidium spectrum of a bulk rubidium cell is used as the standard for comparison to spectra obtained using packaging methods applicable to the ARROW system. A bulk cell is shown in Figure 4-4. The bulk cell is an all-glass (pyrex) system so that the rubidium does not react with the chamber. An inert buffer gas is used. Triad Technologies makes the bulk cell used for this thesis. The rubidium purity is greater than 98%. The buffer gas purity is greater than 99.9% [53]. A D2 line spectrum of the bulk cell is shown in Figure 4-2 with labels of where the peaks are from and in Figure 4-6 in comparison to a computer simulation of the spectrum. The
laser interaction length is the length of the cell (7.5 cm). This spectrum is used as a reference for the spectra obtained for this thesis. The only significant mechanism for broadening is Doppler broadening. This broadening does not change over time.

The bulk cell is placed in spectroscopic setup as basic as the one shown in Figure 4-5. The beam spectrum is recorded after it passes through the vapor cells showing the absorption profile of the rubidium. A more complete diagram of the optical setup used to obtain spectra for this thesis is shown in Figure 6-1.

**Figure 4-4:** Rubidium bulk cell containing pure rubidium.

**Figure 4-5:** Basic optical setup for observing spectra of rubidium vapor cells. The laser is swept around 780 nm to obtain the D2 line.
4.4.2 Bulk Spectrum Comparison to Simulation

The D lines of rubidium can be plotted using theory. In [37], Siddons et al. explains the theory behind the depth and width of the D line peaks. They consider natural and Doppler broadening. A simulated spectra was made following [37] and plotted in Figure 4-6. A spectrum of a rubidium bulk cell obtained using the optical setup at BYU is also plotted. The simulation temperature input was put at about 24 °C as this gave the best fit. It is important to note that the bulk spectra is scaled to fit the simulation. The middle 2 peaks were forced to align and the points outside of the middle 2 peaks were spaced according to the scale that the middle points were scaled to. The y-axis is normalized for both.

![Rb D2 Line Simulation vs Bulk](image)

**Figure 4-6**: Rubidium D2 line spectra. The theoretical line and an actual spectral line recorded using the optical setup at BYU is shown.

Some discrepancies are apparent. First, the peak heights do not match. This may be a result of noise, or nonlinear power distribution on the spectra. This also may be a result of actual changes in the energy levels of the rubidium atoms due to collisions or other interactions. Second, there is an obvious shift in the bulk data that happens at about 2 GHz. This may be a
result of nonlinear sweeping of the laser. This noise is important to consider when comparing data. There may be a little bit of collisional broadening occurring that the simulation does not account for.
5 SEALING METHODS FOR RUBIDIUM COMPATIBILITY

In order to preserve the rubidium spectrum in the ARROW system, several packaging sealants have been researched. The results are described by John Hulbert in [16]. Hulbert concludes that indium plated with copper is the most ideal adhesive for attaching the copper stub to the ARROW chip. Epoxy is considered an easier-to-apply alternative with negative effects on the lifetime of the ideal spectrum of the rubidium vapor. This chapter will explain how indium is used as an adhesive and what plating process variations can be considered.

This chapter will end by presenting a method of easily testing possible adhesives using a chromatography bottle as an alternative to the ARROW chip.

5.1 Indium Versus Epoxy

While epoxy has the benefit of being easy to apply to our system, it has been prone to outgassing/leaking which contributes to collisional broadening in minicell spectral tests. Indium is well known for its low outgassing and low leakage characteristics. Indium is commonly used in vacuums at cryogenic temperatures because it is very malleable at low temperatures; it is not prone to break or crack. Pure indium also has very low vapor pressures [54].

Indium readily reacts with rubidium. Indium has a high electrical conductivity allowing for electroplating. Previous experiments have shown that indium can be plated with copper to keep rubidium from reacting with the indium [16].
In order to use indium as a solder to attach the copper stubs to the chip, metal is evaporated and patterned in the shape of a donut around the hollow core openings of the waveguide. These donuts consist of 150 nm of evaporated chromium followed by 150 nm of evaporated nickel. Evaporation is performed using an electron beam evaporator in the BYU IML. The chromium adheres well to the SiO₂ underneath. Nickel is a commonly used solder surface and adheres well to chromium. The donuts are patterned using lithography and etched using a liftoff technique [16]. The nickel and chrome donuts are shown in Figure 5-1.

### 5.2 Donuts

In order to use indium as a solder to attach the copper stubs to the chip, metal is evaporated and patterned in the shape of a donut around the hollow core openings of the waveguide. These donuts consist of 150 nm of evaporated chromium followed by 150 nm of evaporated nickel. Evaporation is performed using an electron beam evaporator in the BYU IML. The chromium adheres well to the SiO₂ underneath. Nickel is a commonly used solder surface and adheres well to chromium. The donuts are patterned using lithography and etched using a liftoff technique [16]. The nickel and chrome donuts are shown in Figure 5-1.
5.3 Indium Soldering

Indium works well as a solder, having a low melting point of 156 °C. To solder copper stubs onto the donuts, the chip is heated to about 140 °C. A piece of indium is placed on the intersection between the copper stub and the donuts. Using a dropper, specialty flux is then applied to clean the surfaces on the copper and the donut. If the indium is not already melted completely, the flux causes the indium to melt and wick around the donut and copper stub bottom to create a solder joint. The flux directs the solder. The copper end that attaches to the chip is usually polished using sandpaper grades between 200 and 2000 grits.

5.4 Copper Electroplating

In order to keep the rubidium from reacting with the indium, the indium is copper plated using a copper sulfate plating bath. The basic concept is that a voltage differential causes copper to oxidize at the anode and reduce at the cathode. The copper moves through the solution in the form of copper sulfate.

5.4.1 Process

A copper tube smaller than the copper stub is inserted into the copper stub down to about 1 mm above the chip. Care is taken not to crush the hollow core. This smaller copper tube is the anode for the electroplating process. To promote copper on the anode oxidizing at the end of the copper anode and not along the outside of the anode, shrink wrap is placed around the copper anode leaving only the tip in direct line-of-sight of the cathode (see Figure 5-3). The cathode is the indium and the copper stub. The indium is closest portion of the cathode to the exposed to the anode. This is relied on to copper plate the indium more than the copper stub. Copper plating on the stub is a side effect that should not affect the performance of the copper. It is hypothesized
that plating that does occur on the stub helps to have a purified copper layer that acts as a barrier to the less pure, copper stub.

Figure 5-2: Copper is oxidized at the anode and reduced at the cathode forming a thin layer of copper. (Left) Basic copper sulfate plating diagram. (Right) Copper plating the geometries of the copper stub on the ARROW calls for copper sulfate to flow through the anode of the electroplating system.

Figure 5-3: Copper sulfate is flowed down the center of the anode of the electroplating system (a copper tube). The cathode is the copper stub and the indium.
5.4.2 Electroplating Solution

Various types of electroplating solutions are available for copper. The electrolyte is the main variant between solutions. The electrolyte may be cyanide, pyrophosphate, sulfate, or fluoroborate [55].

Copper alkaline cyanide solution, for example, is commonly used for copper plating where high adherence, high throwing power and fine-grained deposits are desired. As a result of these desirable characteristics, cyanide solutions are still often used as strike solutions – plating solutions used for the initial layer of copper over a surface. Over the past several decades, industry has moved away from cyanide solutions, often in favor of copper sulfate solutions. Environmental and human hazards that result from handling cyanide solutions has prompted this change. Laws are becoming stricter about copper cyanide waste management making copper sulfate a go-to solution to avoid cyanide waste [56]. Copper sulfate has made significant progress by the use of additives making it a useful alternative to cyanide solutions [57].

Sulfate Baths

Research in copper sulfate plating solutions has made significant breakthroughs in copper interconnect technology helping it to become the industry standard for plating. Additives in the copper sulfate solution recipes have proven essential to filling trenches and vias in copper interconnects [57].

A typical copper sulfate bath has 2 main ingredients. The main source of copper comes from copper sulfate pentahydrate. Sulfuric acid contributes to the conductivity of the solution and prevents copper crystals from forming. Often, minimal amounts of chloride (in the form of HCl or NaCl) will be added to the solution. Chloride suppresses the amount of copper oxidized
at the anode. The amount of chloride is to be kept between 20 to 100 ppm. If any of these ingredients are out of range, the copper will be rough, streaky, or burned [55].

Other considerations of an acid copper plating setup are current density, the ratio of the anode and cathode sizes, temperature and anode composition. Cathode movement or air agitation can improve copper seeding. Two typical recipe ranges are given in Table 5-1 [58]. These ranges for printed circuit boards are for higher throw capabilities. Current density is taken to be current per unit area of cathode.

<table>
<thead>
<tr>
<th></th>
<th>General Purpose Bath</th>
<th>Printed Circuit Board</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper Sulfate, CuSO$_4$ $\cdot$ 5H$_2$O</td>
<td>190-250 g/L</td>
<td>60-90 g/L</td>
</tr>
<tr>
<td>Sulfuric Acid, H$_2$SO$_4$</td>
<td>45-90 g/L</td>
<td>150-225 g/L</td>
</tr>
<tr>
<td>Chloride</td>
<td>20-150 ppm</td>
<td>30-80 ppm</td>
</tr>
<tr>
<td>Temperature</td>
<td>21-38 °C</td>
<td>21-32 °C</td>
</tr>
<tr>
<td>Current Density</td>
<td>2-20 A/dm$^2$</td>
<td>1.5-5 A/dm$^2$</td>
</tr>
<tr>
<td>Voltage</td>
<td>1-10 V</td>
<td>5-12 V</td>
</tr>
</tbody>
</table>

There are many proprietary additive recipes made available commercially. There are also additives that are often studied and published about in research papers such as Janus Green B [59]. These additives generally fall into 1 of 3 categories: carriers (sometimes called suppressors), brighteners, and levelers. Carriers have large molecular weight and are polyoxyl-alkyl type compounds. They attach to the anode to suppress the rate of oxidation. Brighteners are usually smaller sulfuric molecules that increase plating at the cathode. Levelers are often nitrogen based aromatic molecules that displace brightener species at high current density locations on the cathode (keep from forming pillars) [60].

These are important to consider for indium plating in the ARROW:

1) The anode size is much smaller than the cathode.
2) The distance between the anode and the cathode is very small, so any variation from a centered anode could cause unevenly distributed current density.

3) Sulfuric acid is corrosive to indium. The initial layer of copper needs to be laid quickly and uniformly.

4) Any corrosion pits in the indium may need to be filled by electroplating copper with leveling characteristics.

5.5 New Plating Process for Improved Coverage

Copper plated indium tended to reduce the rubidium spectrum very quickly after the first test. After several days, the spectrum was barely visible and took computer processing to show that there was only a slight spectrum. Looking at the sample in the minicell, it was clear that the copper had peeled off of the indium, exposing the indium to rubidium vapor. A picture of copper peeling is shown in Figure 5-4. This peeling was common on many of the indium chips that were plated.

Further research revealed that this was, at least in part, a result of a brittle copper and indium alloy that readily forms. This had been considered previously on this project, but the nickel barrier deposition step between the indium and the copper was deemed unnecessary in order to simplify the plating process. This step was reapplied to the process.
Further tests seemed to still have problems with copper plating peeling off of the indium. It was hypothesized that this may be a result of too much stress in the nickel and copper. Nickel and copper had been put down with thicknesses of about 15 µm each. 15 µm was used in order to get full coverage of the indium. Shorter thicknesses left patches of nickel exposed. In order to reduce the thickness, while obtaining uniform coverage, another recipe for electroplating was used.

This recipe came from another research project at BYU in the Chemical Engineering department. The recipe calls for 230 mg CuSO₄, 1 ppm MPSA, 50 ppm NaCl, 300 ppm PEG, 10 mL distilled water, and 10 drops H₂SO₄. The mixing process is laid out as described by the recipe source. The key leveling additive is MPSA.

**New Recipe:**

1.) Mix 2 drops of 100 ppm MPSA solution with 2 mL of deionized water.
2.) Add 5 mL of the 100 ppm NaCl solution.
3.) Add 3 mL of the 1000 ppm PEG solution.
4.) Mix in 230 mg of CuSO₄.
5.) Slowly add 10 drops of H₂SO₄.
6.) Heat to 70 °C.
7.) Let stir and cool to room temperature.

The recipe was mixed as directed and an optimal currently density of about 2 mA/cm² was found. It is recommended that the proper current density be found each time a new solution of copper sulfate is mixed. Adjustments become necessary even as several chips are being plated. The voltage for proper plating tended to be between 4 and 5 V at this current density. The recipe gave very good coverage of the indium. Under a microscope, the pits appear to be much smaller and less frequent. Good coverage was obtained with just 1 to 3 um of copper plating. Peeling off of the copper plating was much more minimal than the previous recipe used.
5.6 Plating Conditions Used for Rubidium Compatibility Tests

Copper plating on indium needed to be tested in the presence of rubidium. In order to test copper plating on indium and not use up limited ARROW chip supplies, minicells have been used to model the ARROW atomic vapor system [16].

5.6.1 The Minicell Model

An example of a minicell loaded with rubidium is shown in Figure 5-5. Like the actual ARROW system, the copper is crimped using a cold-weld crimp tool as described in [16]. The copper end that is epoxied to the glass chromatography bottle is sharpened so that the copper fits tightly into the bottle. The copper is attached using Aremco 2310 epoxy. The tight fit of the copper limits the exposure of the rubidium vapor to the epoxy.

Figure 5-5: Minicells are used to test rubidium compatibility. (left) Minicell with just rubidium and nitrogen inside. (right) Minicell with silver epoxy adhesive sample and rubidium inside.

The same minicells have been used to analyze rubidium vapor capillary flow as well. In [61], a capillary having an inner diameter of 200 µm connects two minicells. One minicell is
filled with rubidium vapor. A laser is propagated through the other minicell and detected to check for a rubidium spectrum. Further details of the experiment are given in [62].

5.6.2 Chips Used for Plating

The silicon chips used as a base for the adhesive are made to be thin enough to fit into the copper stub of the minicell (about 0.3 cm). The adhesive area length is about 1 cm. The silicon chip is longer than the adhesive area length for easier handling. For silver epoxy and indium samples, nickel is deposited on the silicon chip to attach the indium and silver epoxy conductively to the chip. Examples of adhesives placed on chip are shown in Figure 5-6. These samples were electroplated before being placed in minicells.

Figure 5-6: (left) Copper plated indium. This sample also has a nickel layer in between the copper and the indium. The plating recipe used here contains MPSA. (right) Minicell containing an adhesive sample.

Figure 5-7: (left) Silver epoxy on chip. (right) Indium on chip.
Since copper sulfate, sulfuric acid and HCl are readily available, these ingredients were used to plate indium. PEG was also used as a suppressor. A copper wire was also attached to the indium to facilitate electrical contact for electroplating. For a majority of testing conditions, the conductive wire was attached using silver epoxy. Later on in testing, copper wire was inserted into the indium before it solidified on cool down. A sample of copper and nickel plated indium on a nickel pad is shown in Figure 5-7.

These samples were then put into minicells. Rubidium was loaded using the same conditions as loading actual chips. The rubidium D2 spectra was then recorded several times a week and the broadness and the deepness of the peaks were analyzed. The results of the peak trends over time are presented in chapter 6.
6 RUBIDIUM SPECTUM IN TEST CELLS

Rubidium spectrum deformation was described in chapter 3. The main sources of rubidium D2 line spectral deformation are collisional broadening from contaminating vapors and peak weakening as a result of the rubidium vapor pressure decreasing.

6.1 Optical Setup

The optical setup for collecting spectra for multiple devices is shown in Figure 6-1. There are 3 photodetectors and therefore 3 lasers beams that can be used for testing minicells. There is also a rubidium bulk cell signal and a reference signal. The 780 nm laser diode is powered by a laser controller. The laser is swept around 780 nm using a function generator. There is a collimating lens that is not shown in the figure. There are 2 mirrors in the beginning to facilitate controlling where the laser is directed. One of the mirrors was later bypassed for simplicity. There is also an iris just after the two mirrors shown that keeps the beam size small. Just after the iris is a neutral density filter which attenuates the signal for all wavelengths equally. These devices are described in more detail in [16].

6.2 Pressure Broadening in Minicell System

To confirm that pressure causes broadening in the minicells described in section 5.6.2, 5 minicells were filled with rubidium and sealed at varying temperatures. Then the spectra were
recorded and the FWHM of the peaks were plotted and compared to [52]. The ideal FWHM is that of a bulk cell spectrum as shown in Figure 4-6 (FWHM of the $^{87}$Rb F = 2 D2 line peak is about 700 GHz). Figure 6-2 shows the FWHM of the D2 line $^{87}$Rb F = 2 peak plotted versus pressure. Two of the data points were unreadable and are not shown. One of those data points was from a minicell that was loaded at atmospheric pressure (760 torr). An unreadable spectra is expected at these temperatures as the broadening could be up to 20 to 30 GHz wide. Our vacuum setup was not conducive to accurate pressure readings as the valve was placed after the pressure gauge in the system. The gauge could not be used to modulate the pressure. The valve was simply closed as soon as the desired pressure was read on the gauge. As a result, the pressure readings are imprecise, but is somewhat consistent with papers previously discussed. Also, the

Optical Setup

Figure 6-1: Optical setup used to obtain spectra for this research.
FWHM of the combined 3 peaks that make up the $^{87}\text{Rb F = 2}$ peak of the D2 line was measured. The slope is theoretically higher than actual linewidth broadening because the width is a measure of 3 peaks that overlap. This should be small, though, since the peaks vary significantly in height and are only separated by 10’s to 100’s of MHz.

The peak width at the minimum pressure we were able to obtain is about 720 MHz which is very close to the bulk peak width of about 700 MHz. This is as expected.

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure6-2.png}
\caption{Plotted $^{87}\text{Rb F=2 D2 line peak FWHM vs. N}_2\text{ pressure at ~70 °C. The equation of the linear fit is shown. The slope is 27 MHz/torr. The slope is roughly consistent with the slope of 19.3 MHz/torr from reports by Kluttz et al.}}
\end{figure}

6.3 Adhesive Sample Spectrum Testing

In order to test the compatibility of rubidium with copper-plated indium as well as copper-plated silver epoxy and Aremco 2310 epoxy, samples of each were inserted into minicells and their spectra were recorded and analyzed versus time. Indium and silver epoxy without copper plating were also tested.

6.3.1 Procedures

Ten minicells were loaded with rubidium – 2 controls and 8 loaded with samples. The samples contained about 0.6 cm$^2$ area of adhesive on silicon chips. One sample had Aremco
2310 epoxy, 2 had silver epoxy without anything on them, 2 had silver epoxy with copper plated on them, 1 had just indium solder on a chip, and 2 had indium with copper plated on them. The 2 controls have no samples in them. They are to compare how the minicell spectra should look if the adhesive samples did not affect the system.

Minicells are prepared by attaching a copper tube about 1 inch in length to a glass chromatography bottle. The glass is cleaned with acetone, isopropanol, and an HF dip. Copper is standard copper tubing that is cleaned in sulfuric acid. Sometimes borax cleaner and a scrubber is used before using sulfuric acid. Copper-etching solutions have also been used, but surfaces appear rough after this solution is used, which may not be conducive to rubidium not sticking to the walls.

The minicells were loaded with rubidium using the same procedure for loading and crimping as ARROW atomic vapor cells.

### 6.3.2 Results

The indium minicells are not analyzed graphically because their spectra failed within several days. Copper plating only helped the spectra last a couple of days longer. The fast failure rate is because of the peeling of the copper plating that could be observed visually as addressed in chapter 5.

As an example of how spectra compares to the normal, Figure 6-3 shows the spectrum of minicell containing a sample of epoxy after it was loaded and after 18 days. The simulation is what the ideal spectrum would be if there was no pressure broadening, a 0.9 cm interaction length (an estimate of the interaction length through the 1 cm diameter minicell. The middle 2 peaks are aligned for all the spectra shown. The detuning is specific to the simulation detuning. The simulation follows [37].
Figure 6-4 shows an example of 3 sample spectra taken over 35 days for the minicells containing copper plated silvering epoxy (SEC). The FWHM is drawn in red. The FWHM increases over the 35 days. Day 35 shows a FWHM with 2 peaks that are not measurable.

In order to compare how the spectra look compared to each other, the FWHM of the first peak was plotted over time (Figure 6-5). A fail threshold chosen to be above the noise is also drawn at a width of 1.1 GHz. With this as the defined fail threshold, SEC1 fails at 9 days, epoxy

Figure 6-3: Epoxy minicell spectra on the same day of the test and after 18 days. Little broadening is apparent on the same day, but after 18 days, there is a lot of broadening.

Figure 6-4: Spectrum of copper-plated silver epoxy on select days over 35 days. All the spectra is shown in the appendix.
(E1) fails at 15 days, a control (ControlX) fails at 25 days and ControlY fails at 33 days. All the spectra for SEC1 and ControlX are given in the appendix.

The depth of the first peak was also plotted, but was so noisy and trendless that it is not shown.

![Figure 6-5: FWHM of the 87Rb F = 2 peak marked with a green circle in the previous spectra. The fail threshold was chosen to be above data noise.](image)

### 6.3.3 Analysis

**Noise**

The noisiness of the trends in both the width and the depth plots makes any conclusions formed questionable. Looking at the sample spectra in the appendix, the spectra seems to vary as much as the plots are noisy. There are many reasons for this that come from the procedures for obtaining the spectra.

First, the minicells are stored in a tube furnace, and taken out to test each time. There are many ways this can lead to inconsistent results. The minicells are agitated each time they are...
carried to the hot plate, releasing any contaminating vapor molecules that have settled on the minicell surfaces. From observing many spectra of minicells, it is known that shaking the cell seems to allow the rubidium to release more vapor. It is theorized that there is a small reaction that takes place at the surface of the liquid rubidium that causes the surface tension to increase and not allow vaporization. This effect may be greatly contributing to the noise.

Second, the laser table alignment is sensitive. The laser table gets out of alignment every once in a while and has to be realigned. Inevitably, each time the table is realigned, the location of the propagating probe beam is changed from where it was previously. Also, there are markers on the hotplate for consistent placement of the minicells, but the placement is sensitive down to the millimeter or less, so these markers are not enough to keep placement consistent. If the beam does not propagate through the minicell the same distance as the previous test, the depth (intensity) of the peaks will change according to lambert’s law [37],

\[ I(z) = I_0 e^{-\alpha(\nu,T)z}. \]

\( I_0 \) is the intensity of the light at the entrance of the medium, \( \alpha(\nu,T) \) is the absorption coefficient of the medium, and \( z \) is the position inside the medium.

Third, besides alignment problems, the beam size may change. This should not be a problem if the beam intensity is low. At high intensities, variations in intensity will cause variations in optical saturation – an effect that changes the lineshape. Optical saturation or optical pumping causes certain transitions to decrease while others are left the same due to forbidden transitions [37].

Other possible sources of noise include varying laser temperature settling time and varying hot plate temperatures (it is only accurate to within 5 °C).
Conclusions

The noise is too great to come up with any solid conclusions, but some hypotheses can be formulated from the data.

First, since both controls failed after 25 days, there are considerations that need to be taken with making minicells. Perhaps the copper is cleaned well enough or not outgassed enough. The copper only sits in a vacuum oven for more than a week or two. Higher quality copper that is oxygen-free should be used. [63] suggests that the copper be oxygen free (OFHC grade) and that outgassing copper to vacuum specifications is difficult without OFHC copper. Also, the copper is attached to the minicell using epoxy. Although care is taken to minimize the epoxy exposure to the inside of the cell, this is an obvious deviation from the ideal testing conditions and will only contribute to noise in the data.

Second, silver epoxy may be the best sealant tested so far. Both silver epoxy cells without copper did not fail before 35 days. The only other one that had not failed by 35 days was a minicell containing copper-plated silver epoxy. It can be concluded that silver epoxy does not outgas as much as the other materials and may even contribute to absorbing unwanted vapor since these cells outlasted even the control cells. For whatever reason, silver epoxy performed well.

Third, copper plated indium has significant hurdles to become a viable candidate as a lifetime extending adhesive. The electroplating procedures need to be improved for copper on indium to keep the copper from peeling off of the indium. This experiment is what led to using nickel as a copper barrier and a copper sulfate recipe that allowed for lower stressed films.

To eliminate the noise, the next test was planned with the minicells staying on the hot plate throughout the whole test. We will call this the “continuous tests”.
6.4 Adhesive Sample Continuous Tests

A continuous test is recording the spectra of sample minicells over a given time without agitating them or changing any possible variables in the laser table setup. The minicells are left on the hot plate and the spectra is recorded over several days. The minicells are “continuously” in their testing conditions. This would serve to eliminate the noise inherent when placing the cells for each test iteration.

6.4.1 Testing Conditions

To conduct continuous tests, automation in the testing setup is needed. The hot plate is set to 70 °C over the length of the test (several days). The function generator and the photodetectors are left on (depending on the battery lifetime for the photodetectors). The laser diode and the laser temperature controller should not be left on though, as these are sensitive devices. For this reason, the LabVIEW software that controls the laser controller and records the spectra from the photodiodes was modified to turn the laser temperature control and the laser diode on and record spectra for 2 to 3 minutes automatically with a specified frequency. After each recording, the software would turn the diode and the temperature control off. This functionality was implemented previously for rubidium diffusion tests since it was hypothesized that rubidium would diffuse completely through a 100 µm capillary after no more than several days.

The downside of this test is that only 2 samples can be tested at a time. Because 2 of the available photodetectors are close together, only 2 photo detectors were used (see Figure 6-1). A sample of Indium with a layer of nickel and a layer of copper was selected to test as this was the latest success in plating indium uniformly (Figure 5-7). Epoxy was also selected.

These two samples were placed in an aluminum holder on the hot plate set to 70 °C. The aluminum served to distribute the heat all around the minicell. These aluminum holders had
holes in them to allow the laser to propagate through the minicells. The laser table was set so that each spectrum had the same amount of laser power. The tests were set to record spectra every 2 hours. The tests lasted 5 days.

6.4.2 Results and Analysis

Figure 6-6 shows the rubidium D2 line spectrum of the minicell with In-Ni-Cu present. The spectrum started shallow and deepened over time until it reached an equilibrium. The simulation shown is using 58 °C and a 0.9 cm interaction length. The minicell is 1 cm in diameter. 0.9 cm is an estimate of the actual interaction length. While the hotplate heating the minicells was set to 70 °C, the actual temperature is unknown. 58 °C is presumed to be close to the actual temperature.

![Rb D2 Line Spectra with In-Ni-Cu Present](image)

**Figure 6-6**: Rubidium D2 line spectrum over time. The first spectrum is the shallowest. The spectrum deepens over 2 hour increments as shown and then settles. The hotplate was set to 70 °C, but the actual temperature in the minicell may have been close to 58 °C as is simulated.
Figure 6-7 shows the rubidium D2 line spectrum of the minicell with epoxy present. The spectrum with the deepest peak is the first spectrum measured. The spectrum goes shallower slightly every 2 hours. What is much more noticeable is that the peaks also broadening over time. These observations give need for trend line graphs of the depth and width of the spectra.

Consider the peaks in Figure 6-6 to be numbered 1 to 4 from left to right. Peak #1 is the smallest peak. The plot of the FWHM of the first peak for both test minicells and the bulk cell is shown in Figure 6-8. The bulk cell should be flat. On the left, the bulk cell has noise that also appears on the other cells. On the right, the minimum value of the bulk cell is taken as 0 and subtracted from each of the 3 signals. This gives smoother data revealing a curvature trend in the FWHMs (and a flat bulk FWHM trend).
Figure 6-8: FWHM of peak 1 over 5 days in stable conditions. The bulk cell ideally is flat. On the left, the bulk cell has noise that also appears on the other cells (system noise). On the right, the minimum value of the bulk cell is taken as 0 and subtracted from each of the 3 signals. This gives smoother data revealing a curved trend in the FWHMs (and a flat bulk FWHM trend).

The FWHM of peak 1 increases at a decreasing rate for the first 24 hours and then tends to level off. Epoxy seems to cause the FWHM to have a slight linear increase after 36 hours. The FWHM of In-Ni-Cu (Indium with nickel and copper plating) seems to even decrease slightly with time. This is very surprising and contradicts previous tests.

Equally interesting is the absorption depth of the first peak over 5 days shown in Figure 6-9. The depth of In-Ni-Cu peak increases dramatically over the first 18 hours and then flattens out. As expected, the bulk cell is steady (besides “shark teeth” and a small step in the last 2 days). Epoxy’s depth decreases at a decreasing rate.

For the In-Ni-Cu peak depth to increase as dramatically as it does, the vapor density must increase. It can safely be assumed that the hotplate is done heating to 70 °C within 20 minutes and that it oscillates around that temperature fairly tightly (given that there is no apparent cyclical behavior). There must be an equilibrium being reached inside the cell that takes about 0.6 days to reach. Residue could be reacting with the rubidium in liquid form and creating a surface that does not allow rubidium to vaporize as readily. The residue saturates and is removed.
from the liquid rubidium surface, allowing rubidium to vaporize. The increased width in the first 24 hours is probably outgassing of the epoxy and copper used to seal the minicell. The epoxy used to attach copper stubs to the minicells is minimized. The surface area that attaches the copper to the minicell is purposely made to be much less that the indium surface area.

The epoxy can be simply explained by a greater amount of outgassing that continues for all 5 days. This would also explain the broadening that occurs. Just as with any distribution, the broader the peak, the shorter it will be, when given the same amount of data points (excited atom).

![Graph](image)

**Figure 6-9:** Absorption depth of peak 1 of the rubidium D2 spectral line. Depth increases rapidly for In-Ni-Cu and levels off. The bulk cell stays rather steady and the epoxy decreases almost linearly.

Although the change is small, the bulk spectrum depth does increase in the beginning. This is explained by the slow heating of the air inside the cubicle of the optical setup. The purpose of the cubicle is to keep light out of the experiment.
The 2 small spikes and the 1 step in the depth signal for all 3 spectra may be a result of a switch of some sort in the system components such as the power supply, the laser controller, or the function generator. They may also be a result of light getting into the system from the computer monitor or room lights. The spikes and step show how sensitive the cells are to their environment.

It is unlikely there are other factors causing the overall trends in the spectra besides those things that have been mentioned. Trends could have been the result of drift in the photodiode power (since they are battery powered), laser sweeping, or the laser diode power, but these possibilities are probably insignificant contributors to the given trends as the bulk cell does not have any variation besides noise.

The spectra measured are averaged from hundreds of raw data spectra. This averaging could possibly be causing trends. The raw data that is scanned has significant amounts of noise.

![Figure 6-10: Number of data points used for signal averaging over the testing time. No clear trend is evident besides an average step down in days 1, 3, and 4.](image)
The optical setup scans 1200 times in just under 3 minutes. Each scan contains 1500 points. There is obvious drift in the peaks as the scans are taken. For each set of 1200 scans, the location of the minimum of each scan (the tallest peak’s tip) is fitted and that plot is used to obtain a fit of all 1200 scans (Hulbert). In this process, some of the scans are truncated, leaving the overall signal truncated. The variance in the number of scan points is plotted in Figure 6-10. There is no clear correlation between the number of points used and the spectral peak FWHM and depth. As a result, it is accepted that the trends are not from the averaging algorithm.
7 ARROW CORE SURFACE PASSIVATION

This measurement technique was applied to analyze ODMS and another SAM called dimethyldichlorosilane (DMDCS or DDMS). DMDCS is similar to ODMS, but the process for depositing ODMS is more sensitive since it takes multiple measurements of several chemicals [16].

For this experiment, the substrates were silicon with about 100 nm of SiO₂ deposited using PECVD. The first substrate was placed in DMDCS for 25 minutes and then cyclohexane for 5 minutes. Then it was baked for 30 minutes. The second substrate was placed in ODMS, mixed according to [16], for 40 min and then cyclohexane for 5 minutes. It was then baked for 50 minutes. The thickness of the 100 nm SiO₂ layer was measured using a spectroscopic ellipsometer. As seen in Table 7-1, the thickness for ODMS was found to be 2.639 nm and the thickness of the DMDCS layer was 0.168 nm. These results indicate that the ODMS surface is not ideal. A proper coating of ODMS should have a contact angle of about 100° [64]. The source of ODMS may have been out of date. The thicknesses are both within expected margins of error. DMDCS has a contact angle close to expected. [65] uses vapor phase deposition for ODMS and DMDCS and get contact angles of 109° and 104° respectively. They find that the ODMS contact angle degraded over time with the SiO₂ substrate, but the DMDCS contact angle did not.

Figure 7-1 shows a picture of DMDCS, two ODMS coatings, and a substrate with no SAM coating. The first ODMS coating followed the recipe in [16]. The second ODMS coating
used cyclohexane mixed with ODMS at a ratio of 20:1 since that is how the DMDCS was prepared. There was little difference in contact angle. The contact angle obtained for both coatings is much larger than if there was no surface, so there must be some sort of layer on top of the oxide. As a result of these tests, DMDCS is used in the latest experiments using SAMs.

**Conclusion**

The hydrophobicity indicates that DMDCS layer is effectively depositing onto SiO2 surfaces while the ODMS is not. As a result DMDCS should be used in the hollow-core of the ARROW to reduce spin depolarization as described in section 3.3.

From experiment, DMDCS layer has no apparent effect on the spectrum of a loaded minicell, but the atom-wall collisions are much less significant in a minicell than in the core of the ARROW as explained in section 3.3. The significance to the spectrum in the ARROW core will need to be studied.

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**Table 7-1: ODMS Versus DMDCS Thickness and Contact Angle**

<table>
<thead>
<tr>
<th>Monolayer</th>
<th>Oxide Thickness (nm)</th>
<th>Thickness After (nm)</th>
<th>Difference (nm)</th>
<th>Contact Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>ODMS</td>
<td>108.404</td>
<td>111.043</td>
<td>2.639</td>
<td>50°</td>
</tr>
<tr>
<td>DMDCS</td>
<td>113.921</td>
<td>114.089</td>
<td>0.168</td>
<td>100°</td>
</tr>
</tbody>
</table>

**Figure 7-1: Hydrophobicity of DMDCS and ODMS monolayers.**

The hydrophobicity indicates that DMDCS layer is effectively depositing onto SiO2 surfaces while the ODMS is not. As a result DMDCS should be used in the hollow-core of the ARROW to reduce spin depolarization as described in section 3.3.

From experiment, DMDCS layer has no apparent effect on the spectrum of a loaded minicell, but the atom-wall collisions are much less significant in a minicell than in the core of the ARROW as explained in section 3.3. The significance to the spectrum in the ARROW core will need to be studied.
8 CONCLUSIONS

This thesis considered the rubidium packaging materials and techniques that can be used to trap rubidium vapor over the hollow-core openings on an ARROW waveguide while preserving the spectra of the rubidium. DMDCS is important to the setup in the tight geometries of the ARROW. In-Ni-Cu is the top viable candidate for long-lasting rubidium spectra as long as the chips are not agitated. A recipe is described. Epoxy is still useful for its ease of application.

Despite positive minicell performances, plating indium with nickel and copper adds many contaminants to the system that could enter into the hollow-core openings of the ARROW. The flux for soldering the indium is also a great concern. The heating and cooling necessary for rubidium may also cause cracking in the waveguides and the cores. Alternatives need to be considered. Suggestions for alternative adhesives to test are given in the next chapter.

Ideal System

There is an ideal system that combines the results of this thesis. The ideal system uses indium plated with copper as the sealant for the copper stubs. The core of the ARROW is coated with DMDCS. This will lead to the most well preserved spectrum on the ARROW system.
9 FUTURE WORK

9.1 Further Adhesive Studies

Epoxy having low outgassing specifications, and high chemical resistance need to be considered for further testing. Aremco 2310 has only “good” chemical resistance and does not meet NASA outgassing requirements.

The author recommends Aremco 2315. Aremco 2315 has the following advantages:

1.) Meets NASA outgassing requirements
2.) “Excellent” chemical resistance – two steps up from “good”.
3.) Lower coefficient of thermal expansion.
4.) Thermally conductive – the heat transfer to the copper stub should be improved.
5.) It is a potting compound – it was made to seal.

The properties of several epoxies including Aremco 2315 and 2310 are given on Aremco’s website [66]. Other brands may also have adhesives with desirable properties.

9.2 Complete Tests

If Aremco 2315 works well on minicells, it needs to be applied in a complete ARROW system. In-Ni-Cu should also be applied to the complete ARROW system. Once these tests reveal a long lasting rubidium spectra, the ARROWs can be applied in optical systems on-chip and slow-light experiments can be performed.
9.3 New Design

A concept being implemented for testing is shown in Figure 9-1 (b). In this single-stub design, the hollow core arms that reach out to the stubs are eliminated in order to minimize the distance the rubidium vapor needs to travel in order to reach the laser probe that goes down the center of the core. The need to eliminate these “arms” is based on the results obtained in [62]. The concept requires that the solid core waveguide be a buried channel waveguide in order for the copper stub to be placed on top.

![Image of double and single stub designs](image)

Figure 9-1: (a) Double stub design. (b) Single stub design. This design minimizes the distance rubidium needs to transport through hollow core to be in the laser interrogated portion of the waveguide.

The single stub design have all the same packaging procedures applied to it. The only changes are that the DMDCS will not be flowed through the core. It will have to diffuse through. The effectiveness of relying on diffusion will need to be tested. The copper-plating will be easier to implement since the stub is about twice as large.
REFERENCES


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M. H. Giraud-Carrier, Hill, Cameron; Decker, Trevor; Black, Jennifer; Almquist, Soren; Schmidt, Holger; Hawkins, Aaron, "Rubidium Diffusion in Microscale Spectroscopy and Slow Light Platforms," *Journal of Vacuum Science & Technology A*, Submitted November 2015.


APPENDIX A

Figure A-1: Minicell control spectra over 35 days.
Figure A-2: Minicell with copper-plated silver epoxy spectra over 35 days.
APPENDIX B

Figure B-1: (left) Rubidium D2 line peak 2 width over 5 days in continuous tests. (right) Peak 2 width over 5 days in continuous tests.

Figure B-2: (left) Rubidium D2 line peak 3 width over 5 days in continuous tests. (right) Peak 3 width over 5 days in continuous tests.