Evaporative Coating of Rb Maser Cells

D. F. Phillips, A. Boca and R. L. Walsworth

June 3, 1999

1 Introduction

The double-bulb rubidium maser (DBRM) relies on a vacuum cell for its operation. This differs from the spin-exchange noble gas cells which contain Xe, He and N_2 as well as rubidium. These gases (in addition to playing active roles in the noble gas experiments) act as a buffer, keeping the Rb away from the walls of the cell. In the DBRM the lack of buffer gases allows rubidium atoms to effuse between the pumping and masing chambers of the device. Without a buffer gas, however, the device is very sensitive to rubidium-wall interactions which can shift the rubidium hyperfine frequency and even depolarize the atoms.

Long polarization times may be obtained by coating the inner surface of the bulb containing the rubidium atoms with a benign substance. Previous work [2, 3] has shown that Rb bounces hundreds of times from a surface coated with tetracontane, $C_{40}H_{82}$, – a component of standard paraffin – (table 1), without losing its polarization (Fig. 1). Note in the figure that octadecyltrichlorosilane (OTS) [4], the coating used for noble gas cells is much less effective with rubidium. (Similar results have been found by other groups [5] after careful studies.) The tetracontane coating process is, however, somewhat complicated as the tetracontane must be evaporatively coated under vacuum.

In the following sections of this document, we will describe the evaporative coating scheme as well as some of the details of Rb handling that may differ from the noble gas cells. We will also discuss the initial techniques used to characterize the coating quality as well as some earlier techniques used in coating DBRM cells.

2 Coating

The aim of the evaporative coating process, as described in detail below, is to apply a thin uniform layer of tetracontane to the cell. A metal filament covered with tetracontane is placed inside the cell. The filament is then heated so that the tetracontane melts and evaporates into the evacuated cell. When the tetracontane hits the room temperature walls of the cell it sticks, forming the coating. Contaminants inside the tetracontane such as any

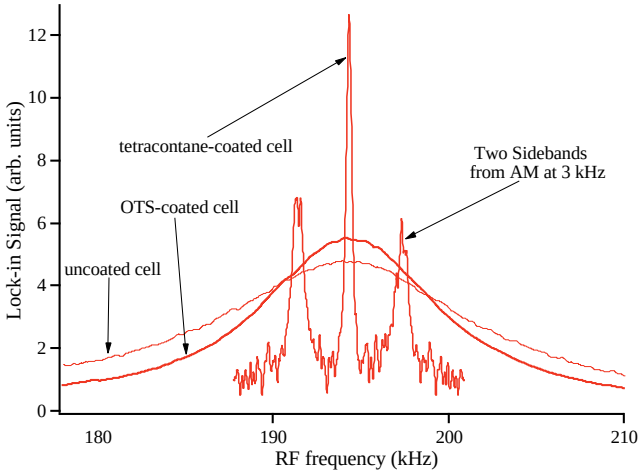

Figure 1: Zeeman resonances at B=0.28 gauss from bare, OTS coated and tetracontane coated cells. The linewidth is, in part, determined by the lifetime of the polarized atoms in the cell. If the atoms maintain their polarization for a very short time because of depolarizing wall collisions, then their linewidth will be very broad. The linewidths of uncoated and OTS coated cells correspond to roughly the time between wall collisions (i.e. one bounce depolarizes them). The tetracontane linewidth corresponds to several hundred bounces and is limited by magnetic inhomogeneities and power broadening in the measurement process rather than by polarization lifetime. (From [1])

solvent, water and oxygen will typically have different vapor pressures than the tetracontane itself. Therefore, they will either evaporate before the tetracontane and be pumped away or stick to the glass before the tetracontane or never evaporate at all. This allows us to produce a cleaner coating than simpler wet techniques which apply the tetracontane solution directly to the walls of the cell in air (see appendix B).

2.1 Cell manifolds

The rubidium bulb is initially made by our glass blower [6] or the quartz workers [7] and attached to a manifold. This manifold allows the cell to be evacuated by a turbo pump at the cell filling station and holds the filament containing the coating as well as the Rb which will be placed in the cell. Typical cell manifolds are shown in figures throughout the text. A glass, single bulb cell which was used for testing coatings is shown in figure 2. The filament enters the cell through the upper capillary

Table 1: Properties of tetracontane

formula	$CH_3(CH_2)_{38}CH_3$
formula weight	536.1 amu
melting point	81.3°C
boiling point	150°C

and the rubidium is brought in from the lower. Figure 5 shows the quartz double bulb used for initial maser tests. Figure 6 is a double bulb glass cell for studying light scattering problems. (Except for some additional labels added for this text, this was the drawing given to the glass blower.) Note in the figures that in addition to the cell itself the manifold contains a port where it is attached to the pumping system, a storage region for the filament after the coating is completed and a tube where the Rb ampoule is attached. (The single bulb cell (Fig. 2) is shown with the Rb ampoule attached while the double bulb cell (Fig. 6) is shown before the ampoule is attached.) During the pull-off (sec. 3.1), tubes leading from the cell are sealed, leaving only the single or double bulb cells with one or two tips.

2.2 Preparing the filament

Make filaments from steel brazing rod, magnet wire and coax inner conductor pressed together.

The coating filament is constructed from three pieces of metal to absorb and transport the heat and to hold the tetracontane. First, a 1.2″ steel brazing rod, 0.1″ in diameter rests in the capillary tube outside the cell. This large diameter piece will be heated via induction heating. As it is magnetic, it can also be used to manipulate the filament with a permanent magnet from outside the manifold. Two strands of 2″ copper magnet wire transport the heat to the center of the bulb. Finally, a 1/2″ piece of multistrand inner–conductor from a coax cable holds the tetracontane. The multistrand wire wicks the tetracontane effectively, stopping it from dropping off the filament onto the bulb when it melts. These three pieces of wire are crimped together as the temperature necessary to vaporize the tetracontane and coat the cell will melt soft solder. (The pieces could also be spot-welded together. For details on earlier filaments, and their difficulties see appendix A.)

Aldrich Chemical Co. [8] sells tetracontane (table 1), in the form of flakes. It is melted onto the multistrand wire by heating the wire with a soldering iron far from the tetracontane and care

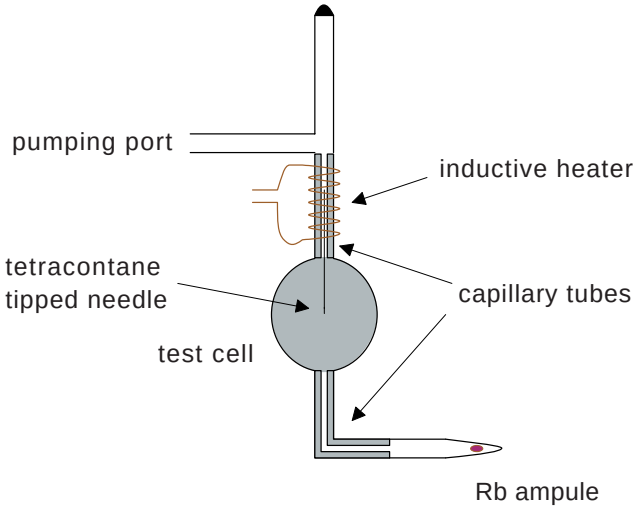

Figure 2: Single bulb manifold used for studying cell coatings. The filament and inductive heating coil are shown in place. A turbo pump is attached to the "pumping port" to evacutate the cell. After the coating process, the filament is stored in the region above the pumping port.

is taken not to burn the tetracontane.

Apply one or two flakes of tetracontane to the filament and gently heat with a soldering iron.

2.3 Cleaning the cell and attaching the Rb ampoule

Before the manifold is evacuated for coating, it must be cleaned and the Rb ampoule must be attached. The manifold is initially chemically cleaned following the same procedures used for other cells in the lab. First the manifold is cleaned with soap (Alconox powder) and warm distilled water in an ultrasound bath. The manifold is then rinsed with more distilled water. Then the manifold is cleaned with "piranha solution" which consists of 70% H_2SO_4 and 30% H_2O_2 by volume. This solution will bubble vigorously and become hot. While the solution is active, put it in the manifold and let it sit for an hour, turning and rotating it so that the acid comes in contact with the whole manifold. Next, rinse the manifold three times with distilled water and three times with methanol to remove the acid. This procedure will thoroughly clean the cell.

For more information on glass blowing, see references [9, 10].

Then the Rb ampoule (most recently purchased from Strem Chemicals [11]) is attached to the end of the manifold (Fig. 6) using a methane – oxygen torch If the outer diameter of the arm where the ampoule is attached matches that of the ampoule the

glass blowing is quite easy. Before attaching the ampoule, however, a piece of glass with which to open the break-seal must be inserted. (We have experimented recently with including some magnetic material in the breaker glass following suggestions of Hugh Robinson, but have not found it particularly helpful.) To check if the glass working is successful, put the manifold on the pumping station and pump it down. The pressure should fall quickly below 1×10^{-4} torr. If the pressure remains above this level, there is a leak. To further check for leaks, a little methanol may be sprayed on the joint. If this changes the pressure inside the manifold (either higher or lower), there is a leak and the glass joint must repaired.

The process of glass blowing will introduce vapor and other contaminants into the cell and the manifold will need to be cleaned again. The second cleaning begins with a rinse of the full manifold with methanol to remove any macroscopic water left in the cell by the glass blowing. Then the manifold is evacuated with a turbo pump to 10^{-6} Torr and heated to drive off any water and other surface contaminants in the glass. If the manifold does not pump to 10^{-6} Torr in one hour, there is probably a leak in the manifold which must be fixed.

To heat the cell, cover it in aluminum foil for good thermal conductivity, then wrap 110 volt heating tape around it. A thermocouple gauge is placed between the wraps of the heating tape as a temperature probe. A final layer of aluminum foil is applied around the tape to contain the heat. Power is then applied to the heating tape using a Variac (typically starting at 10% on the Variac) to slowly heat the cell, while still under vacuum. The power is increased on the Variac until, after several hours, the temperature is over 120°C. Recently, the manifold has been heated as high as 200°C for the overnight part of the bake-out. (At this temperature, one must be careful to keep the ground glass joint much cooler because the vacuum grease which seals it will break down and leak. Also, the valves may not take this high a temperature.) After pumping and heating overnight, the heat is removed. Pressures close to 1×10^{-7} torr should be reached when the manifold cools to room temperature. (Note that Robinson [12] and Bouchiat [2] bake their cells at much higher temperatures of 350°C and 400°C respectively.) More details on other glass cleaning techniques may be found in appendix C.

After the manifold has been cleaned, the vacuum space is gently back filled with nitrogen. The pressure is brought slightly above one atmosphere so that the manifold may be opened with-

Monitoring a typical bake out:

time	V	T	P
17:05	0	24°	$3 \cdot 10^{-5}$
17:04	10	24°	$3 \cdot 10^{-5}$
17:15	10	33°	$3 \cdot 10^{-5}$
17:15	20	33°	$3 \cdot 10^{-5}$
17:45	20	67°	$4 \cdot 10^{-6}$
17:45	30	67°	$4 \cdot 10^{-6}$
18:10	30	117°	$2 \cdot 10^{-6}$
18:10	40	117°	$2 \cdot 10^{-6}$
18:35	40	185°	$6 \cdot 10^{-6}$
18:35	45	185°	$6 \cdot 10^{-6}$
9:15	45	192°	$6 \cdot 10^{-7}$
9:15	0	192°	$6 \cdot 10^{-7}$
10:00	0	30°	$4 \cdot 10^{-7}$

out wet air rushing into the cell. The filament is then placed into the cell and it is evacuated.

2.4 Inductive heater

Rather than using an electrical feedthrough into the glass manifold containing the cell to heat the filament, power is coupled to the filament inductively from a coil wound outside the manifold (Fig. 2). This allows the filament to be removed from the center of the cell to a storage area inside the manifold while the manifold is under vacuum. In addition, this technique reduces the complexity of the manifold and eliminates difficulties of heating in the wires leading to the filament.

Wrap Teflon tape and then the induction coil around the cell entrance, covering one inch above the bulb.

The inductive heating coil is a single layer of magnet wire wrapped roughly 40 times around the section of the manifold containing the filament. The coil is wound from relatively thin 25 HAPT copper magnet wire. This allows a large number of turns on the manifold close to the bulb itself and produces more flux. However, there are losses in this thin wire, wasting power outside the cell and heating the coil and glass rather than the filament. Therefore, a layer of Teflon tape is wrapped around the glass underneath the coil to insulate the glass from the heat of the coil.

Resonate the coil with a capacitor to produce a tuned circuit whose frequency is about 10 MHz.

A large voltage must be applied across the coil to heat the filament enough to vaporize the tetracontane. Therefore, a capacitor is placed in parallel with the coil, forming a tuned circuit (Fig. 3) with a $Q \approx 4$ (Fig. 4). As the voltage induced in the filament will be proportional to the frequency as well as the amplitude of the drive, the frequency should be set as high as is reasonably achievable. Resonant frequencies between 1 and 10 MHz (Table 2) have been used for this tuned circuit. Note that the filament slightly shifts the resonant frequency and degrades the Q. (In one round of cell coating, the 2.5 MHz resonant frequency (table 2) was shifted down by 30 kHz.) Therefore, the drive should be adjusted for maximum power after the filament has been positioned in the cell. Similarly, warming the coil shifts its frequency slightly. For optimal power, the drive may need to be optimized during the coating process as the coil warms.

Drive the heater with a power amplifier.

The tuned circuit is driven resonantly with several watts of RF power to coat the cell. Figure 3 show our present equipment for driving the tuned circuit. The frequency generator which produces the resonant drive, has a maximum output of roughly 50 mW. As this will not melt the tetracontane, a power amplifier producing up to 15 watts amplifies the generator and drives the tuned circuit. This inductively couples enough power to the

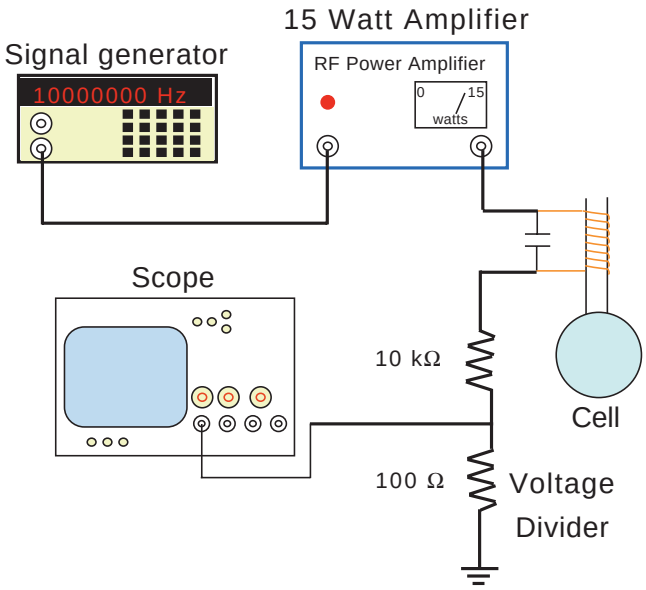

Figure 3: Electronics for inductive heating. An SRS DS345 generates the RF drive which is amplified by a 15 watt power amplifier. The RF is fed to the tuned circuit on the manifold and is monitored with a scope via a resistive voltage divider.

filament to melt the tetracontane. A voltage divider composed of 1/4 watt resistors (the large resistor dissipates more than 0.1 watts) is used to monitor the power delivered to the tuned circuit on an oscilloscope without damaging the scope.

2.5 Coating the cell

Turn off the overhead lights, put a black background behind the cell, and directly light the cell before coating.

When the manifold with the filament in place has pumped down below 1×10^{-6} Torr, we position the filament using a small permanent magnet so that the tetracontane is centered in the cell. Then the RF amplifier is turned on at the resonant frequency of the tuned circuit to heat the filament. As the tetracontane on the filament begins to melt, its vapor pressure increases, it evaporates from the filament, and it then hits and sticks to the cell wall which is at room temperature. This deposits a thin layer of tetracontane on the cell walls. Several minutes after the tetracontane begins to evaporate, the RF power is shut off.

Cells should be coated thinly, but the coating must be visible throughout the cell.

Even with a very light coating, the tetracontane should be visible on the surface of the cell. A bright light and a dark background behind the cell may be necessary to observe the coating, however. While we see a milky white coating, others have described making coatings of thickness comparable to visible light

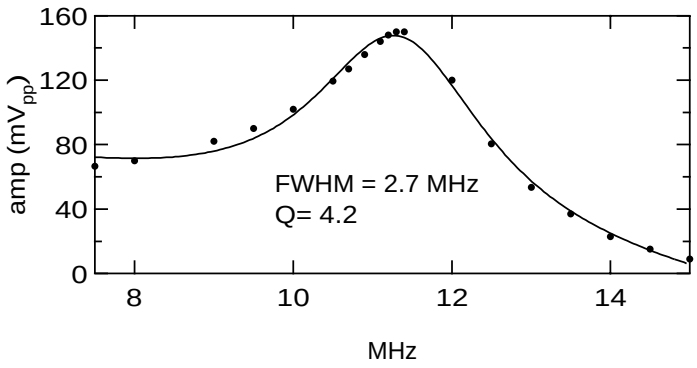

Figure 4: Output amplitude of induction heater drive divided by 100 (see fig. 3). For this circuit $Q \approx 4$. The drive voltage from the signal generator was 16 mV_{pp} giving a total voltage gain from the amplifier and tuned circuit of 1000 on resonance. Typically, to coat a cell, the input voltage is 50 mV_{pp}, which corresponds to 10 watts delivered to the coil.

wavelengths and seeing interference effects and rainbows when inspecting the cells under bright lights. (We have, however, coated one cell in which we could not convincingly see the coating and still observed long polarization lifetimes.)

Once the coating is sufficiently thick, the power is shut off and the filament is returned to the storage region outside the cell. The filament must be removed from the cell volume rapidly because the filament will remain hot for close to one minute after the power is turned off and may continue to coat the cell or the Rb storage neck if it is not removed from the cell region. Use the small magnet to move the) filament out of the cell into a temporary holding position.

After turning off the RF, quickly return the filament to the storage region as it will continue to coat for about one minute.

3 Chasing Rb and sealing the cell

After the cell has been coated, we must open the rubidium ampoule and chase rubidium into the tip of the cell using a torch. First, we open the ampoule containing the rubidium which was attached to the cell manifold before the coating was performed. There is a small glass hook on top of the ampoule. Breaking the hook allows Rb to enter the manifold. Others have done this by embedding some magnetic material in a glass rod above the hook and then using a magnet outside the manifold to force the rod into the hook. We simply rotate the cell so that the glass rod falls under gravity, breaking the hook. It is often conve-

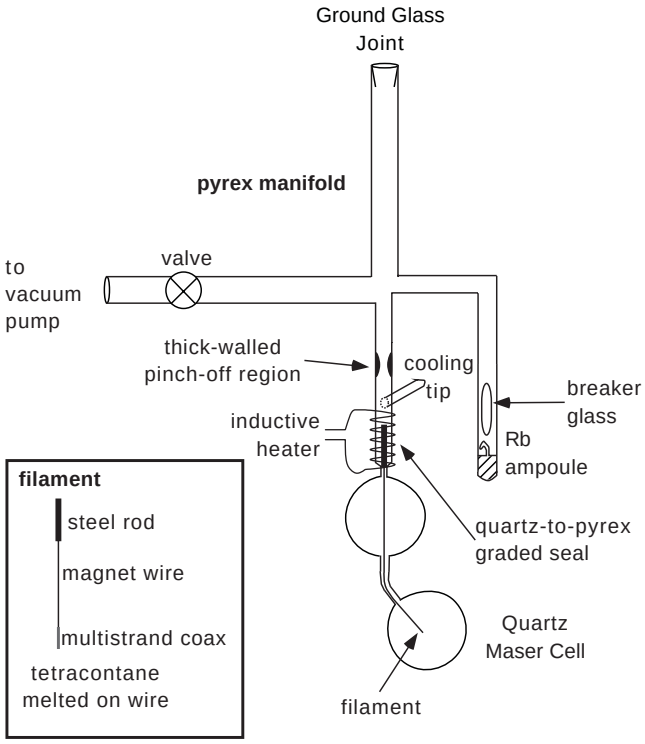

Figure 5: Drawing of double-bulb, quartz maser cell. Because of the bend in the "transfer-tube" between the two bulbs, the filament must be very flexible. This has led us to redesign our filaments using multistrand wire.

Table 2: Heating parameters from two coating runs

	1998 (Andreea)	1997 (Nolan)
N	38 turns	
L	10 μH	7 μH
C	35 pF	22 pF
ν	10 MHz	12 MHz
drive strength	50 V_{pp}	50 V_{pp}
solvent	none	xylene

nient, though not necessary, to have a valve on the end of the manifold so that it may be removed from the active pumping system while still under vacuum. This makes it easier to rapidly rotate or shake the manifold.

Once the rubidium ampoule is opened and the Rb exposed

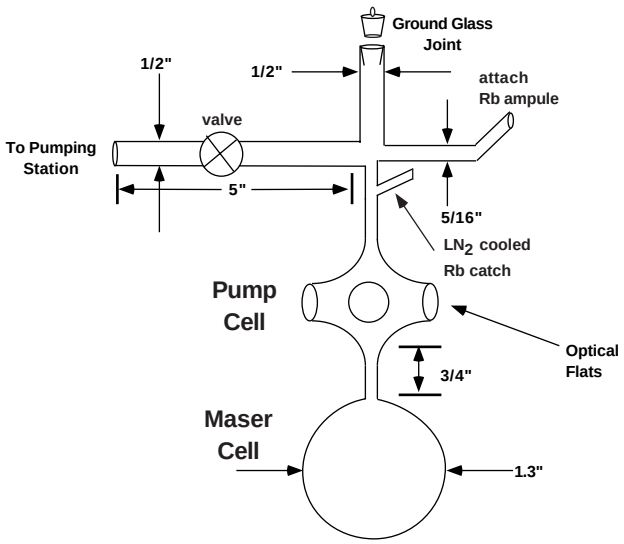

Figure 6: Drawing of double-bulb, glass cell manifold. Note the optical flats in the pump bulb to reduce scattered light in the maser cell. The valve on the left side allows us to remove the manifold from the pumping station while under vacuum and the tube on the upper right is where the Rb ampoule is attached. The filament is admitted to the manifold through the ground glass joint and may be stored before and after coating in the space immediately below it. The ground glass joint is sealed with a glass stopper.

Table 3: Properties of rubidium

natural abundance	^{85}Rb	72%
	^{87}Rb	28%
melting point	T_{melt}	39.3°C at 1 atm
boiling point	T_{boil}	688°C at 1 atm
specific heat	C_p	0.363 J/g · K
vapor pres.	p	$p = 10^{4.312-4040/T}$ atm
		$T > T_{melt}$, T in Kelvin

to the manifold the Rb must be warmed so that it will flow. (We use the term "chasing Rb" to refer to the process of heating the Rb above its melting point and allowing it to flow towards cooler regions of the manifold. This is also known as "distilling" the Rb.) To keep rubidium from entering the cell and damaging the coating during this procedure, a small glass block is placed

at the neck of the cell. This block contains a small piece of magnetic material so that it may be moved inside the manifold using the usual external magnet. (We typically store it during coating near the valve.)

After blocking the cell entrance with the stopper, the cell itself is wrapped in wet paper towels (to keep it cool) and aluminum foil. The foil will both keep the cell at a uniform temperature and protect the paper towels from the flame of the torch. We then warm the Rb using a weak flame from the torch with the oxygen turned off. Initially, there is argon trapped behind the Rb. The argon will escape when the Rb is first warmed above its melting point near 35°C (table 3). As the argon escapes, it will push the Rb out of the way, sending it down the manifold. The pressure in the manifold will suddenly rise and slowly fall as the argon is pumped away. The rubidium may then be chased towards the cell using a weak flame. A thin layer of Rb on the surface of the glass will appear blue whereas bulk Rb is a silver color. Thus a blue tint will lead the Rb as it is forced along the tube. We want to chase the Rb to the neck of the pump cell. (In the most recent cell (Fig. 6), we had a tip before the neck of the cell which we could cool with liquid nitrogen and catch the rubidium as it is chased towards the cell.) When the neck of the cell has a bluish tint, there will be plenty of Rb in the cell.

3.1 Sealing the cell

After the Rb has been chased, we are ready to seal the cell. First, to protect the coating, we gently pour about a cup of LN_2 over the foil and wet paper towels covering the cell. This freezes the water on the towel and provides more protection for the coating. Next, we gently warm the region of the glass neck where the seal will be made with a weak flame (by stopping the flow of O_2 the torch). This melts and then evaporates any coating in the seal region so that it is pumped away. A flame hot enough to melt glass, applied to coated regions of the cell, can burn the coating leaving contaminants that can degrade the coating in the cell.

With a hot torch, we separate the cell from the manifold. After the cell has been pulled off, the manifold should remain under vacuum. Once the pull–off is completed and the sealed cell tip has been annealed with a weak flame and then cooled, the aluminum foil and the paper towels are removed. The paper towels should still be cold, indicating that the coating hasn't been damaged by excessive heating. Also, an air leak into the cell will oxidize the Rb and discolor it. Thus, our signature of a

successful pull-off is that the manifold sustains a good vacuum, the Rb stays silver colored and the wet paper towels surrounding the cell remain cool.

3.2 Post pull-off rubidium chasing

Heat the sealed cell to 50°C and cool the Rb tip in an ice bath to transfer any Rb inside the cell back to the Rb tip.

After the pull-off tip has been allowed to thoroughly cool, any Rb which has entered the cell must be chased back into the tip. The body of the cell must be heated above the Rb melting point while the tip is kept cool. The cell is again wrapped in aluminum foil, heating tape and more aluminum foil (as in sec. 2.3), leaving the Rb tip free and oriented downwards so that liquid rubidium will flow towards it. The tip is then cooled with an ice bath so that the Rb which flows there will stay. (LN_2 is too cold and should not be used. The vapor rising from the bath will cool the cell through the aluminum foil and heating tape.) The temperature of the bulk of the cell is then brought slowly to 50°C as monitored on a thermocouple inserted in the heating tape. After several hours with this temperature gradient the Rb should migrate to the tip, producing a functioning tetracontane coated vacuum sealed Rb cell.

4 Remelting the coating

After the cell has been sealed and the Rb chased to the tip, we often find that we need to melt the coating to produce a long hyperfine coherence time (T_2). We therefore heat the cell to about 85°C, a few degrees above the melting point of tetracontane. We heat the cell inside an air oven constructed by wrapping heater tape around a Pyrex beaker. This allows us to observe the coating as it melts.

Attach an RTD to the cell with thermal joint compound and yellow tape for a good thermal connection.

We first, attach a temperature sensor (typically a 100 Ω RTD — Resistive Temperature Detector — from Omega) with yellow scotch electrical tape and thermal joint compound (Thermalcote can be found in the tool chest) to the cell to monitor its temperature. The cell is then placed inside the beaker with the Rb tip sticking out so that it will remain cool. A rubber piece is used to close the bottom of the beaker. The Rb tip pertrudes through a hole in the rubber piece. Then the heating tape is wrapped around the beaker at the height of the cell and attached to a Variac.

Voltage is now applied to the heater tape to begin the remelting of the coating. Over roughly one hour, increase the voltage on the Variac to raise the temperature of the cell. We typically

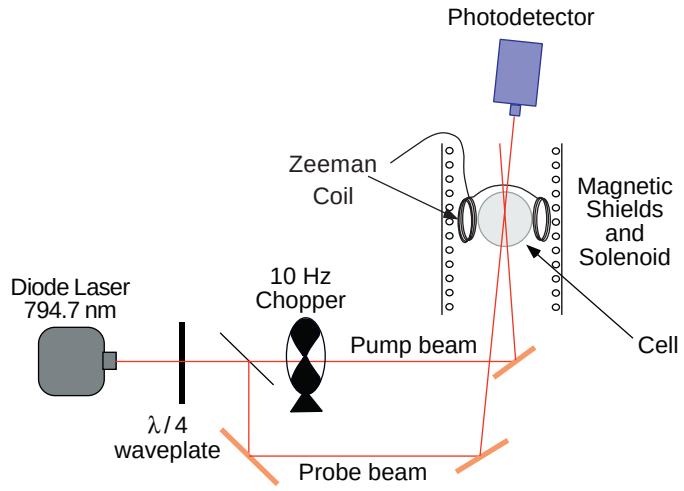

Figure 7: Schematic of optical setup for measuring T_1. A neutral density filter and a fast chopper may also be placed in the probe beam path.

increase the voltage by 5% per step and then allow the cell to re-equilibrate before further increasing the power. When the temperature monitored on the RTD reaches 80°C, we carefully watch the coating. Once it melts, we reduce the heat by a few %, allowing the coating to remain melted for several minutes. Then we slowly cool the cell back to room temperature. After we take the cell out of the beaker and remove the RTD, it is ready for polarization and hyperfine studies.

With a 500W heater, we start the Variac at 5% and raise it in 5% steps every 15 min. up to about 25% where the coating melts.

Cell remelting data:

time	Variac	T
6:20	$0 \rightarrow 5$	26°
6:25	$5 \rightarrow 10$	26°
6:35	$10 \rightarrow 15$	30°
6:50	$15 \rightarrow 20$	39°
7:00	20	49°
7:20	$20 \rightarrow 25$	57°
7:30	25	67°
7:40	$25 \rightarrow 27$	73°
7:50	$27 \rightarrow 28$	77°
8:00	28	81°
8:10	$28 \rightarrow 20$	83.5°
8:20	$20 \rightarrow 10$	71°
8:30	$10 \rightarrow 0$	47°

5 Optical T_1 measurements

The Rb polarization time (T_1) of the cell may be measured using an optical technique based on work by Corney [13]. Two laser beams, of significantly different intensities, are focused on the cell (Fig. 7). The strong pump beam polarizes the Rb atoms in its path while the weaker probe beam, which is not strong enough to noticeably affect the polarization of the cell, is absorbed by unpolarized atoms in its path. By polarizing the atoms with the pump beam and then measuring the polarization over time with the probe beam, T_1 can be determined.

Depending upon the polarization of the light, the T_1 of different transitions can be measured. Linearly polarized light tuned to one hyperfine level will depopulate the entire level whereas circularly polarized light will leave atoms in the highest m_F sublevel. Thus linearly polarized light will make us sensitive to

T_1(hyperfine) while circularly polarized light will be sensitive to T_1(Zeeman).

Initially the pump beam is turned on to polarize the sample. The pump is then turned off and the transmission of the probe beam through the cell is monitored. (We control the pump beam with a slow chopper whose period is set so that that the pump stays off and on several times longer than T_1.) With the pump beam off, the Rb atoms begin to lose their polarization. More of the probe beam is absorbed and a weaker signal is measured at a photodetector down stream from the cell. By fitting measured intensities to decaying exponentials ($I = I_0 \exp(-t/T_1) + I_{\text{offset}}$) we determine T_1 of our cell.

5.1 Testing the probe strength

To be certain that there are no repumping effects from the probe beam, the measurement should be performed at several probe intensities. We insert an attenuator in the probe beam (not shown in figure 7) to reduce the light intensity. In the past, to make repumping insignificant we have had to reduce the intensity such that we can just barely observe the signal above the noise even with averaging of 50 measurements. To increase sensitivity, a second fast chopper, if available, can be added to the probe beam to chop it at rates much faster than T_1. Then the chopped probe beam is detected with a lock-in amplifier referenced to the chopping frequency. This second chopper should run at over 1 kHz to resolve depolarization times of a few milliseconds.

In addition to varying the probe strength, it is important to measure T_1 as a function of the Rb oven temperature. Reducing the temperature reduces the Rb vapor density and therefore Rb–Rb collisional spin-exchange effects which can reduce T_1. However, as the density is reduced, the dynamic range of the probe beam absorption signal also drops. Therefore, we usually start at Rb temperatures around 40°C and lower it towards 30°C, extrapolating to zero temperature to determine T_1 of the cell in the absence of Rb spin-exchange. In several Pyrex test cells, we have observed Rb $T_1$of 30 milliseconds at low temperatures. For our cells, this corresponds to ≈ 250 Rb bounces off the tetracontane coated walls before depolarization.

APPENDICIES

A Old filaments

We experimented on many different filament designs. Initially, we soldered multistrand wire to piano wire, but found that before the induction heating melted the tetracontane, the soft solder joints melted. Also, at these temperatures, the piano wire discolored and coated the bulb in black soot. We found empirically that the brazing rod from the model shop does not suffer these problems, and switched to crimping the wires together to avoid the melting solder. To apply tetracontane to the filament, we have tried dissolving it in xylene, but were unable to deposit enough tetracontane on the filament after dissolving it in this manner.

B Previous cell coating techniques

Earlier cells were coated using a wet technique, rather than vapor deposition. The tetracontane was dissolved in toluene and the solution was placed directly into the cell in air and mixed so that all surfaces of the cell were coated with the solution. The cell was then dried under vacuum. This left behind a very visible waxy coating which was extremely uneven. These cells had a very short T_1, which was improved by the techniques described below. It is difficult to determine, however, which changes were important.

First, the cell was heated above the melting point of the tetracontane ($\approx 80°C$) overnight. This allowed the tetracontane to partially redistribute itself inside the bulb. When this was completed, however, the tetracontane was still uneven, thick and waxy looking.

Second, during the initial studies, the Rb tip was not kept cooler than the main bulb. This allowed Rb to collect inside the bulb which could drastically shorten the T_1. When a better oven was constructed which allowed the Rb tip temperature to be separately controlled, the cell was allowed to sit with a large temperature gradient for several days to chase the Rb back to the tip. (Note that on one test cell in which large quantities of Rb were accidently let into the main cell, the tip was placed in an ice bath and the bulb was warmed to 50°C overnight. This successfully removed the Rb from the bulb and allowed a 30 msec T_1 to be observed.)

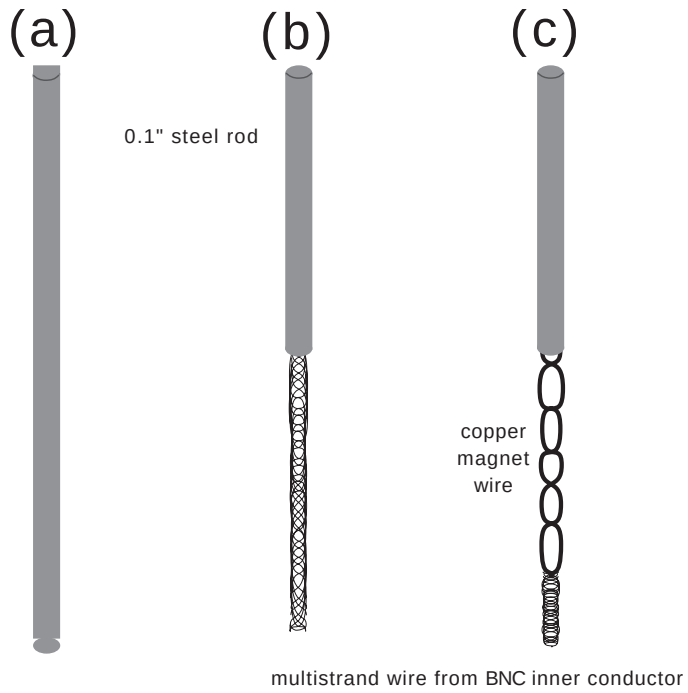

Figure 8: Drawing of different filaments with which we have experimented. In (a) a single piece of piano wire is used. This design has several problems including: lack of flexibility for the double bulb, outgassing of carbon when hot, and dripping of tetracontane. In (b), brazing rod replaces piano wire to eliminate outgassing and multistrand wire inside the bulb allows the filament to traverse bends and enter the bulb. The problem with this filament is that the multistrand wire wicks the tetracontane away from the bulb. In (c) magnet wire is used to transmit the heat into the bulb where multistrand wire holds the tetracontane and keeps it from dripping onto the bulb.

C Cell Cleaning

There are three standard techniques for cleaning glass. One can wash it in acid, heat it under vacuum, or set up a gas discharge in a dilute gas. In principle, since our cells are coated with wax which does not chemically bind to the glass/quartz substrate, one might hope that we would not be very sensitive to the cleanliness of our cells. However, we have dramatically improved T_2 since we started cleaning more carefully. We use the first two of these techniques in our cell cleaning procedure.

Our chemical cleaning follows the techniques of the noble gas experiments [14] as well as standard procedure in glass handling [9]. We clean the cell with detergent and then rinse out the detergent with distilled water. Then we clean with "piranha solution" consisting of 70% sulfuric acid and 30% hydrogen peroxide, which we rinse with distilled water and methanol.

C.1 Baking

The next step in the cleaning process is to bake the system out while it is under vacuum. We have typically baked overnight at around $100^\circ - 200^\circ$C. Robinson [12] and Bouchiat [2] both bake their cells at much higher temperatures of 400°C or 350°C.

C.2 Discharge Cleaning

In making cells for hyperfine studies of Rb, Hugh Robinson used a discharge cleaning process he refers to as *plasma scrubbing*. Robinson's description of the process is as follows: "Cell cleaning included a 12-h bake out at 350°C followed by plasma scrubbing with both hydrogen and helium gases. [12]" Discharge cleaning ignites an atomic discharge inside the cell and uses the active ions and metastable atoms to clean the surface of the cell. After the cell has been evacuated, a small quantity of gas is admitted to the cell. (H, He, Ar and N_2 at a few Torr are typically used.) The discharge is ignited with a Tesla coil and maintained by an RF coil driven with a few watts of power. After some minutes or hours, the gas is pumped away. New, clean gas is then admitted and the process is repeated. Many groups describe discharge cleaning cells for spectroscopy and NMR. Several are summarized below.

A Pipkin student [15], in producing a light source for ^{3}He, cleaned Pyrex cells by first cleaning in acid, then baking the cell by taking a torch to its exterior rather than using a slow uniform baking procedure. Finally, he discharge cleaned by admitting a

few torr of ^{4}He and igniting a discharge with a 50 MHz oscillator driving a coil external to the cell. The discharge is allowed to run for 15 minutes. With discharge still running, he opens the cell to the pump and pumps away the helium. A rosy pink glow is then seen on the Pyrex walls even at pressures of $\sim 10^{-8}$ torr. He states that "this glow was taken as evidence of a clean, out-gassed walls."

Lusher et al. [16] report a similar process in cleaning Pyrex cells for a low temperature ^{3}He NMR experiment. The cleaning process included baking at 470°C for 2 days under vacuum followed by a discharge process. 2.5 torr of ^{3}He were admitted to their system and a discharge was ignited and run for 3 hours before the helium is pumped away. "[t]his process was repeated several times until a pure helium optical spectrum was observed during the discharge, the discharge cleaning normally taking two days." No mention is made, of course, of the spectroscopic methods used to determine the purity of the spectrum.

With respect to cell cleaning, Lusher et al. cite Horvitz [17]. Horvitz was also doing ^{3}He spectroscopy and reported large improvements in T_1s after cleaning. His discharge cleaning technique is appealingly straightforward. After chemically cleaning the cell, he admits a few torr of He. Then he takes a torch and a Tesla coil and attacks the cell. He heats it near to the melting point and ignites the discharge there. He repeats this process all over the cell. He writes that when impurities are present, the discharge will be blue but will turn pink when all that is left is helium. He also reports using a "pocket spectrograph" to check the spectral lines.

References

[1] K. Tan, E. Mattison, E. Oteiza, R. Stoner, R. Vessot, I. Yu, and R. Walsworth, Charcaterization of wall coatings for a double-bulb rubidium maser, unpublished.

[2] M. Bouchiat and J. Brossel, Phys. Rev. **147**, 41 (1966).

[3] H. Robinson and C. Johnson, IEEE Trans. Instr. Meas. **32**, 198 (1983).

[4] J. Sagiv, J. Am. Chem Soc. **102**, 92 (1980).

[5] J. Camparo, R. Frueholz, and B. Jaduszliwer, J. Appl. Phys. **62**, 676 (1987).

[6] Wilbur Scientific, Inc., 37 Leon St., Boston, MA 02155, Phone 536-2609, Charles Raworth does all of our standard glass blowing and some of the less critical quartz work, as well.

[7] Mikoski Fused Quartz and Glass, 296 Gleasondale Rd, Stow, MA 01775. Phone (978) 562-7372.

[8] Aldrich Chemical Company, 1001 West Saint Paul Avenue, Milwaukee, WI 53233, http://www.aldrich.com/, cat. no. 381713.

[9] J. H. Moore, C. C. Davis, and M. A. Coplan, *Building scientific apparatus, second edition* (Addison-Wesley, New York, 1989), see chapter 2.

[10] Corning Glass Works, *Glass blowing with pyrex glasses* (Corning, New York, 1954).

[11] Strem Chemicals Inc., 7 Mulliken Way, Dexter Industrial Park, Newburyport, MA 01950-4098, http://www.strem.com, $18.40 per 1 gram ampoule.

[12] H. Robinson and C. Johnson, Appl. Phys. Lett. **40**, 771 (1982).

[13] A. Corney, *Atomic and Laser Spectroscopy* (Oxford University Press, Oxford, 1988), see chapter 17.

[14] D. Hoffman (unpublished).

[15] S. D. Rosner, Ph.D. thesis, Harvard University, 1968.

[16] C. P. Lusher, M. F. Secca, and M. G. Richards, J. Low Temp. Phys. **72**, 25 (1988).

[17] E. P. Horvitz, Phys. Rev. A **1**, 1708 (1970).