David Chaparro

# Optically Detected Magnetic Resonance and Experimental Setup and Data Analysis

## Abstract

Optically detected magnetic resonance is an effective way to measure magnetic fields. Using microwaves and electron spin resonance (ESR), it is possible to measure the magnetic field of a sample as well as its magnetic moment. However, measuring the magnetic field of a sample under pressure presents considerable challenges in measurement. With NV center diamonds it is possible to measure the magnetic field on the order of nT while under pressure. With diamond anvil cells (DAC), it becomes possible pressurize a sample up to 10 GPa and measure the magnetic field using NV-Center Microdiamonds. To achieve these measurements, it is necessary to find the optimal voltage to run an ESR experiment. My work included finding the saturation point of various Designer Diamond Anvil Cell (DDAC) antenna geometries as well as determining the power with the best signal to noise ratio. Additionally, I will be developing the procedure to pressurize a DAC to begin pressurized NV-Center Diamond ESR measurements. Through taking the derivative of an ESR spectra we found that the optimal power to run an ESR experiment for our DDAC is 400mV. Taking the derivative of an ESR Spectra has been found as the best method to determine the optimal power for a DDAC.

## Introduction ODMR

Electron Spin Resonance (ESR) is a spectroscopy method which is used to measure the energy level of an electron in a magnetic field. Electrons in an energy level have a spin and a magnetic moment. When magnetic field $B_0$ is applied to an electron, the spins will align with the magnetic field, splitting the energy levels into different states $+\mu_B B_0$ and - $\mu_B B_0$. The distance between these two energy levels is proportional to the strength of the magnetic field. By measuring difference in energy it takes to excite an electron from the 0 state to the +/- $\mu_B B_0$, we can calculate the magnetic field or the magnetic moment of the sample. These are called resonance frequencies and are observed through electron spin resonance. This effect is especially noticeable in Nitrogen-Vacancy center diamonds (NV-Center Diamonds). The Nitrogen and vacancy site in the lattice creates a two electron system which can occupy energy levels with a spin of 1,0, and -1. [1] This material is also special because it is sensitive to magnetic fields in the nT range. Green laser light (532nm) excites electrons to a higher energy state and causes the material to fluoresce red when the electrons decay to their lower state. However, if you excite the electrons with its resonance frequency microwaves (around 2.75 Ghz) and green laser light, it is possible to excite the electrons to a higher energy which fluoresces with a different decay path [2]. Instead of fluorescing red, the electrons decay through an intermediate energy level causing a decrease in the fluorescence intensity *(Figure 1)*. The number of electrons decaying through this alternate process is about 4% of the total electrons decaying. To observe this decrease in fluorescence, our experiment utilizes a lock-in amplifier to detect and measure the small signal that would otherwise be indistinguishable from noise. The two ways to measure the ESR of a material include field varied ODMR and frequency varied ODMR. Field varied ODMR changes strength of the magnetic field and holds the microwave frequency constant. [3] This causes the degenerate states to separate from each other as the magnetic field strengthens.

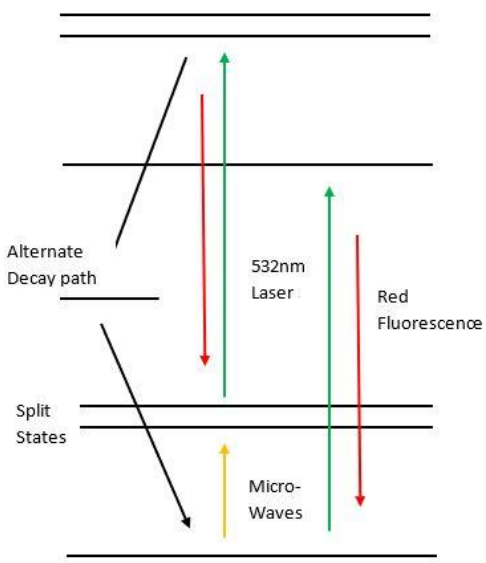

*Figure 1: Energy diagram of an NV Center diamond. Red indicates fluorescence, green indicates laser emission and yellow incident microwaves*

At resonance magnetic field, the microwaves will be at resonance with the +/-1 states causing a decrease in fluorescence. The other method is frequency varied, where you maintain constant magnetic field and vary the microwave frequency. This method allows you to detect two resonant frequencies and can be used to measure the magnetic field inside a material, or its magnetic moment. Using fluorescence to determine the resonant frequency is the primary feature of Optically Detected Magnetic Resonance (ODMR). Our work aims to find the saturation point of the absorption signal as well as pressurizing the diamond anvil cell to begin measurements of magnetic field under pressure.

## Experimental Setup and ESR Procedure

Our experimental setup involves an optical setup featuring a laser and photodiode. First, the green laser shines on the NV-Center diamond, causing it to fluoresce. Then, the microwave generator outputs an AM modulated microwave signal to the microdiamond antenna to be absorbed by the NV-Center. The photodiode signal is then passed to the lock-in amplifier along with the microwave source to detect the signal. The lock-in amplifier multiplies the two sine waves together, resulting in one high frequency and low frequency signal. Using a low pass filter, we can filter out the microwave frequencies longer frequencies. By

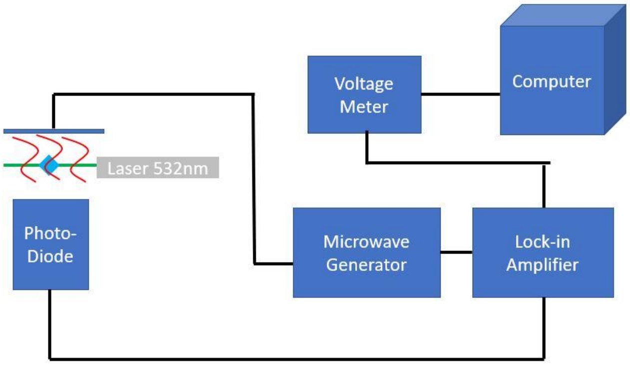

*Figure 2: Experimental setup of instruments*

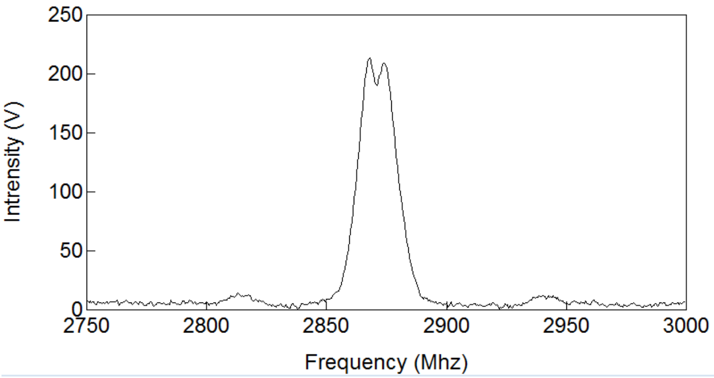

*Figure 3: Example ESR spectra*

minimizing the phase shift between the microwave source and the optical signal, we can measure the absorption directly. We then send the DC voltage signal from the photodiode to the Keithley voltage meter to be processed and sent to the computer for recording *(Figure 2)*. Our sample is placed on an adjustable gantry with a hole to allow a green laser light to shine on the sample. On the platform our sample is housed on is a small antenna which is connected to a microwave generator. Each experiment consists of sweeping the range of microwave frequencies from 2750MHz to 3000MHz in increments of 5MHz and plotting the corresponding voltage output from the system *(Figure 3)*. At each frequency, data was taken every .5 seconds for 5 times to ensure an accurate reading and eliminate noise. Additionally, noise was reduced by taking multiple passthroughs of the frequency range. For my first part of the project I was responsible for testing a large diameter NV center diamond with the optical setup to ensure that the experimental setup functioned correctly. My next responsibility was to complete the same experiment with a cluster of microdiamonds and a single micro diamond with NV Centers on multiple antenna geometries to determine the saturation point for the experiment *(Figure 4)*. Setup for the Designer Diamond Anvil Cell (DDAC) required placing the single micro diamond in the center of the DDAC using vacuum grease and an eyelash tool *(Figure 5)*. The DDAC has a microwave antenna built into the sample stand so that we can perform ODMR. Once the micro diamond is placed on the DDAC the DDAC mount is placed on

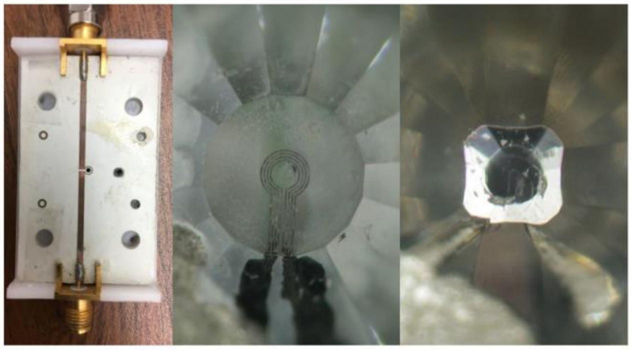

*Figure 4: Antenna A (left), Antenna B (middle), Antenna C (right)*

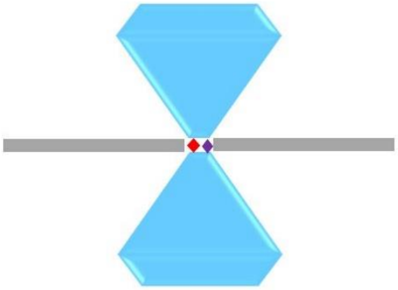

*Figure 5: Diamond Anvil Cell*

the adjustable gantry. To center the microdiamond, I had to search for the signal by adjusting the gantry multiple times until I found fluorescence. However, I managed to use the optics of the DDAC (round cut) to search for the microdiamond using the light that passed through the diamond. It was possible to both identify the antenna and microdiamond by staring at its projection on the ceiling and observing the dim shadows that passed through the projections *(Figure 6)*. Once the DDAC was centered and the microwave generator was connected to the DDAC and the phase shift from the microwave source to the lock-in amplifier was found using code that finds the optimal phase shift. The last experiment that I worked on was to recreate the pressurized ODMR experiment. To do this, the DDAC was aligned to the stage optical axis and secured using stycast 1266. This step was crucial to the pressurized ODMR experiment and we learned that stycast needed to be applied from the bottom of the diamond to halfway up the pavilion. Once that was complete, both the DDAC and the normal DAC were aligned and placed in their respective mounts. Once in their mounts the diamonds are aligned using set screws. With a thin metal gasket in between, the diamond is pressurized slightly to indent the gasket and prep it for drilling. An electronic drilling machine is used to create a hole <300 microns wide. Unfortunately, however, once we reached this step, we encountered problems with the DDAC and lost its electrical connection and were unable to proceed with the pressurized experiment.

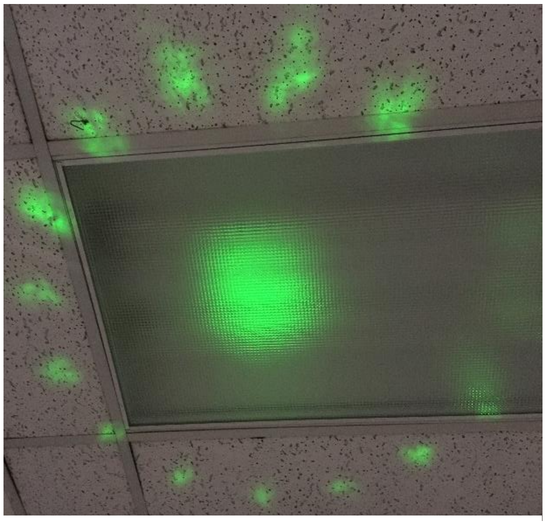

*Figure 6: Optical observation of Diamond Anvil Cell*

Results

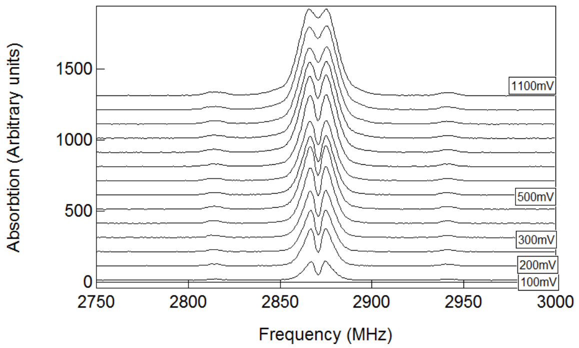

*Figure 7: ESR Spectra of varying microwave intensities*

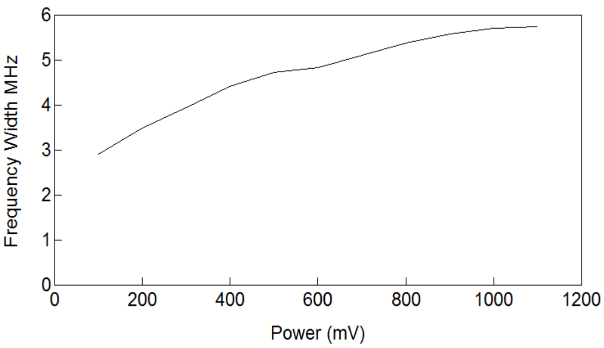

*Figure 8: Absorbtion peak width as a function of power. Width of peak was determined using a gaussian fit to each peak.*

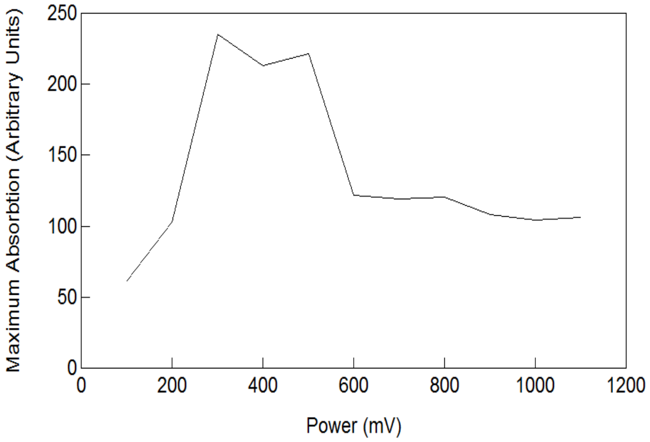

*Figure 9: Maximum intensity of peak absorption. Arbitrary units*

The first ESR Spectra was taken with the large diameter NV-Center Diamond on frequencies between 2750 MHz to 3000 MHz every 5MHz at powers from 100mV to 1.1V every 100mV *(Figure 7)*. The power spectrum showed that the height of the ESR Spectra peaked at 300mV, however, the width of each curve almost continuously increased as the power was increased. While this was enough for finding the optimal power setting for the antenna A with a large microdiamond, it was not enough for finding the saturation point on the DDAC antenna geometries. It is suspected that the two peak structure comes from an asymmetry of the microwave antenna generating a magnetic field. The next experiment utilized a cluster of micro diamonds on antenna B with an ESR Spectra taken at the same microwave frequencies, however, the range of powers used was from 100mV to 500mV in 50mV intervals. Graphing the maximum values of each power spectrum, we found that there was a peak at 200mV, however this was determined to not be the saturation point because the maximum value increased beyond the peak height after 500mV *(Figure 9)*. We next decided to

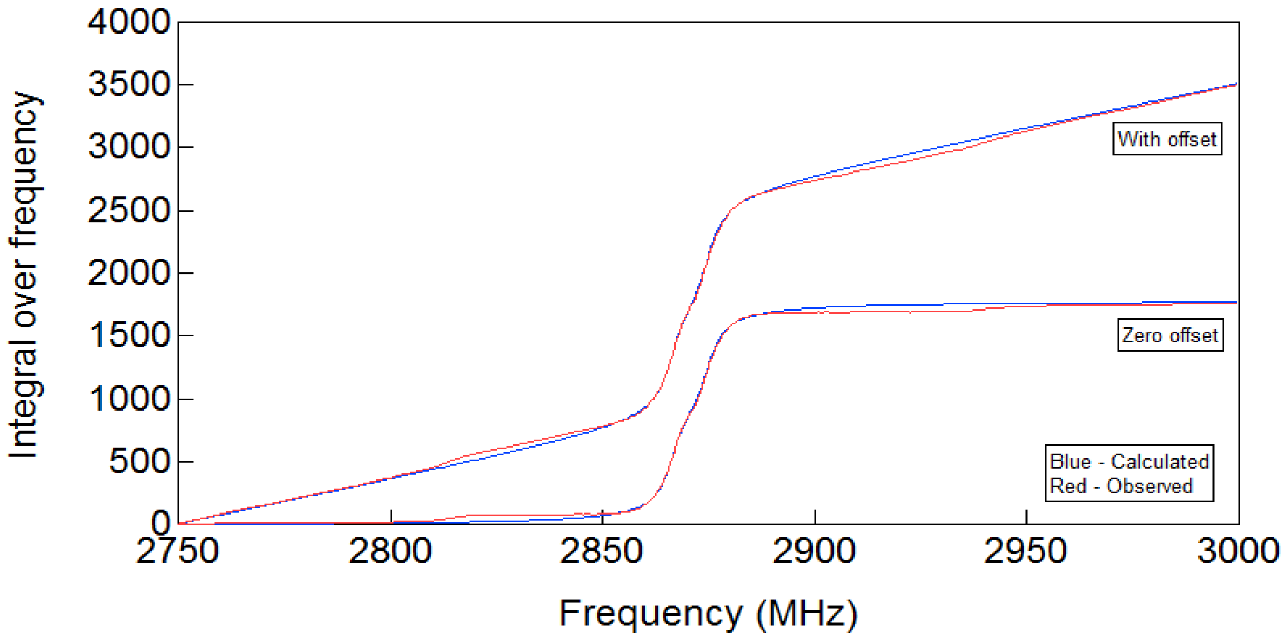

*Figure 10: Integral of sample ESR Spectra. Blue lines are predicted values based on gaussian fit parameters while red is actual data.*

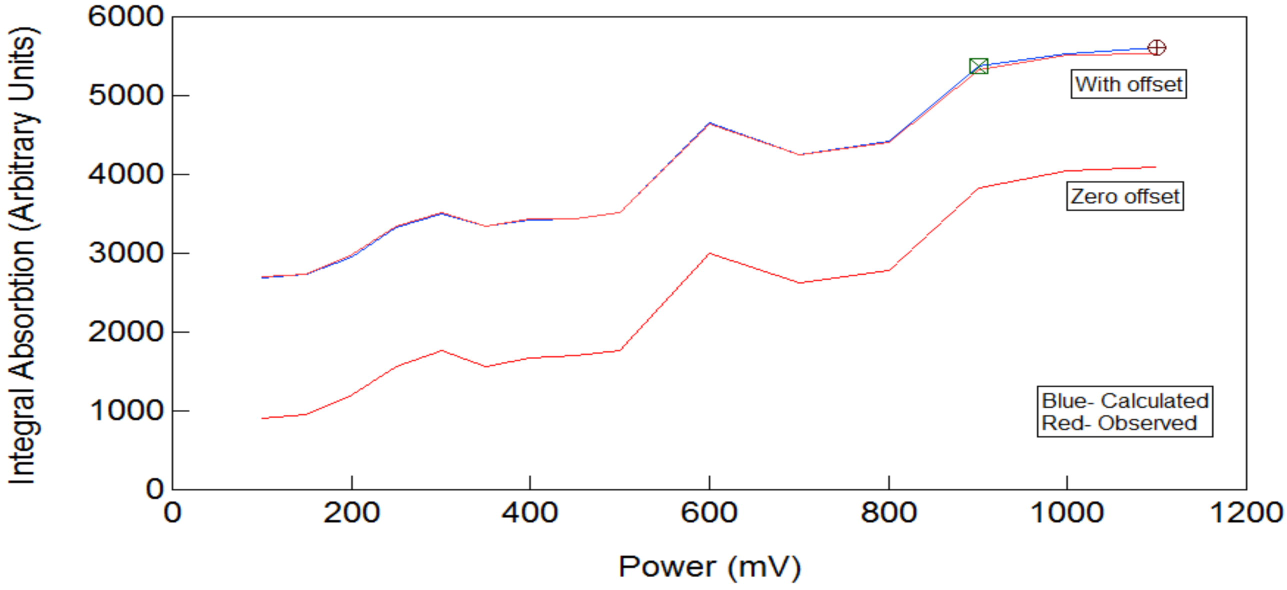

*Figure 11: Total value of integral for varying powers*

use antenna B to find a single micro-diamond and run a power spectrum. This experiment we decided to take the integral of one of the spectra to see if the noise from the system was contributing to the lack of a saturation point. We fit the data to two Lorentzian curves and used their fit parameters to recreate the datasets with a baseline and without it. Plotting the calculated

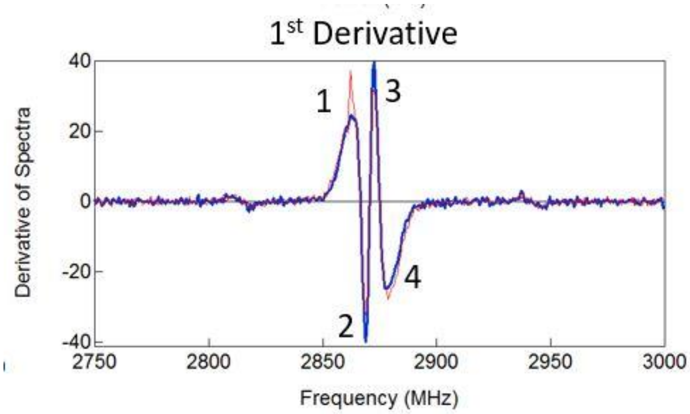

*Figure 12: Derivative of sample ESR spectra.*

vs experimental curves showed that both with and without a baseline, the noise did not significantly contribute the value of the total integral *(Figure 10)*. The total integrals of the experimental and calculated data were then graphed with and without offset and showed no significant differences *(Figure 11)*. Our last experiment was on antenna C with a single microdiamond. Power spectra were taken from 100mV to 1.1V in increments of 50mV. We then differentiated the data plotted the rate vs power *(Figure 12)*. The derivative for each power spectra was calculated, and the local maxima and minima were recorded to look for any patterns. Looking at the data the first local maximum and the second local minimum (1 and 4 on the image) were found to constantly increase in magnitude. However, the first local minimum and second local maximum (2 and 3) were found to peak at 400mV, giving us a definite power with the highest signal to noise ratio *(Figure 13)*. This is because the higher the derivative, the higher the rate of

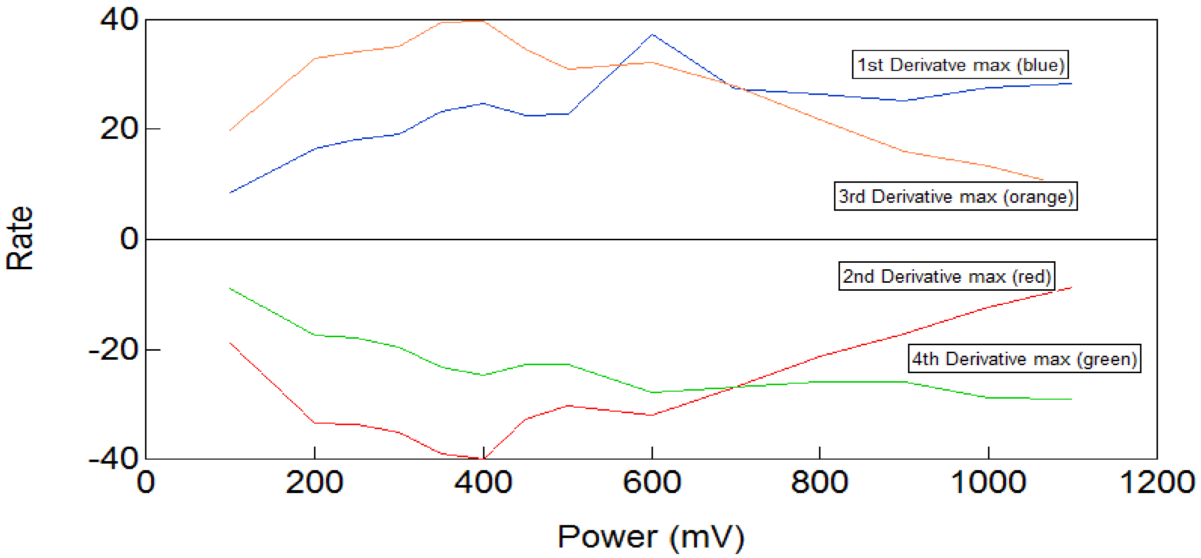

*Figure 13: Plot of maximum value of derivative for each power.*

change. The rate between the peak and the background noise is the highest when the background noise is the lowest. Thus, the derivative tells us the proportion of the maximum value of signal vs the background noise. Because of this, the highest derivative gives us the signal to noise ratio. The maximum value for the derivative represents the point with the optimal power to run the ESR experiment. However, we did not reach saturation because the power needed to reach the saturation point was well beyond what was possible with the microwave generator. Each antenna has a resistance from 50 ohms to 600 ohms; as the resistance increases, the power dissipated through microwaves decreases. Thus, we were unable to reach saturation because the resistance on the antenna was too high.  Since antennas A and B were not used for the pressurized ESR experiment, the optimal power was not determined for these antennas.

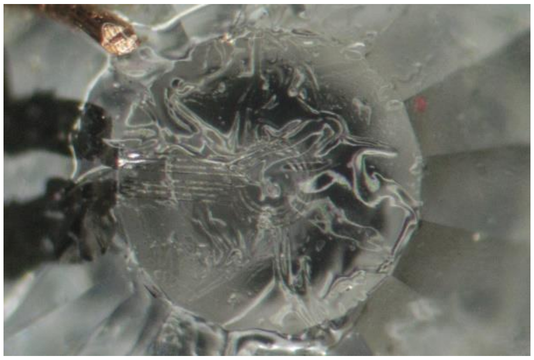

*Figure 14: Image of single microdiamond on antenna B*

## Conclusions

Using the derivative of the ESR experiment spectra, we determined that the optimal power to complete experiments on Antenna C is 400mV. Using this method of finding the maximum value of the derivative, we can determine the optimal power for future diamond anvil cells and can run pressurized ESR experiments. Future work for this experiment includes adding a Helmholtz coil to run a field varied ESR experiment which would be useful for measuring the magnetic field of a material under pressure. Additionally, with a pressurized diamond anvil cell, it will be possible to measure the magnetic field near a material while it is under pressure, as well as its magnetic moment. Additionally, it is possible to add a cryo-stat to measure these properties at different temperatures as well.

[1] A. Lenef, S. W. Brown, D. A. Redman, S. C. Rand, J. Shigley, and E. Fritsch, Physical Review B **53**, 13427 (1996).

[2] V. R. Horowitz, B. J. Alemán, D. J. Christle, A. N. Cleland, and D. D. Awschalom, PNAS **109**, 13493 (2012).

[3] J. Palmer, L. C. Potter, and R. Ahmad, J Magn Reson **209**, 337 (2011).