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Development of a Vacuum Encapsulated, Hermetically Sealed Diamond Amplified Cathode Capsule

A Thesis Presented

by

Elizabeth Gangone

to

The Graduate School

in Partial Fulfillment of the

Requirements

for the Degree of

Master of Science

in

Physics

(Scientific Instrumentation)

Stony Brook University

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Abstract of the Thesis

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This thesis will discuss the purpose, theory, and fabrication procedure of a hermeticallysealed, diamond amplified cathode capsule as well as results and progress to date. The objective of this thesis is to develop a procedure to fabricate a successful capsule, in which a successful capsule can contain at least $\sim 10^{-9}$ Torr pressure during leak checking and hold off at least 5000 V during voltage hold off measurements.

The main purpose of the capsule is to create a secondary electron emitter which acts as a high brightness, low emittance source in electron guns for accelerator systems and can be easily transported from the fabrication site to electron guns. The diamond amplified cathode capsule uses the diamond amplifier concept to overcome current problems associated with photocathodes and drive lasers for high average current accelerator systems. The capsule contains primary electrons emitted from a cathode in an insulating spacer under vacuum. The primary electrons are accelerated across the spacer by a high voltage bias into a diamond. Secondary electrons are

produced and emitted from the diamond, which results in a gain of two orders of magnitude. Material selections for the capsule were carefully chosen and described.

Sample preparation and fabrication procedures for both the low and high temperature were developed. Leak checking and voltage hold off measurements were used to refine the fabrication procedure. These integrity checks were also used to quantitatively define a successful capsule. Future research will be focused in improving the success rates for the integrity checks, refining the sample preparation and fabrication process, and performing an emission measurement to calculate gain.

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1. Introduction

This thesis will discuss the purpose, theory, and fabrication procedure of a hermeticallysealed, diamond amplified cathode capsule as well as results and progress to date. The objective of this thesis is to develop a procedure to fabricate a successful capsule, in which a successful capsule can contain at least $\sim 10^{-9}$ Torr pressure during leak checking and hold off at least 5000 V during voltage hold off measurements.

1.1 Purpose

The main purpose of the capsule is to create a secondary electron emitter which acts as a high brightness, low emittance source in electron guns for accelerator systems and can be easily transported from the fabrication site to electron guns.

Various applications require high average current electron beams greater than 100 mA in accelerator systems, such as energy-recovery linear accelerator systems (ERL) [1]. Limitations of producing average currents greater than approximately 50 mA exist due to current capabilities of conventional photocathodes and drive lasers. Another limitation arises in the electron gun of the accelerator system itself, as there is a risk of photocathode contamination thereby shortening cathode lifetime. A diamond amplifier, developed in recent years [2], [3], is a concept in which a primary electron beam is accelerated to a few kV. The primary electron beam is then amplified in diamond, producing a cascade of secondary electrons. The main components of a diamond amplifier consist of a cathode, an insulator, and a diamond. This concept overcomes the limitations of the cathodes and drive lasers by increasing emitted current with amplification

factors over two orders of magnitude [4],[5]; however, if a sensitive cathode was to be used, such as cesium potassium antimonide (CsK₂Sb), a new method must be implemented to protect the cathode surface. The hermetically sealed, diamond amplified cathode capsule encapsulates the accelerating gap between cathode and diamond. This is accomplished by containing vacuum inside of a hollow, ceramic spacer with the cathode and the diamond at either end. This protects the cathode from contamination in the electron gun, as well as allowing for transport from development site to the injector without exposure to ambient air.

1.2 Chapter Organization

Chapter 2 discusses the relevant concepts and principles behind the diamond amplified photocathode capsule. Detailed fabrication procedures are described for the low and high temperature capsules in Chapters 3 and 4, respectively. The capsule fabrication procedure must be compatible with cathode and diamond preparation procedures. These procedures include materials selection, sample preparation, joining techniques, and integrity checks. An example of how the gain measurement would be performed is detailed in Chapter 5. A discussion of the results and improvements in the fabrication procedure to achieve higher success of integrity checks in the future are described in Chapter 6.

2. Diamond Amplified Photocathode Capsule Principle

The diamond amplified photocathode capsule utilizes and expands upon the diamond amplifier recently developed [2],[3]. The primary electron beam from the photocathode is accelerated by a voltage bias across the encapsuled gap of the capsule. The primary electron beam is then amplified in the diamond, which causes the emission of secondary electrons from the hydrogenated diamond surface. The photocathode and secondary emitter are both encapsulated in vacuum to improve its lifetime and eliminate the risk of contamination.

2.1 Primary Electrons

An incident light source on the photocathode surface causes primary electrons to be emitted by the photoelectric effect. The primary electrons from the photocathode are accelerated by a ~10 kV bias towards the metalized diamond surface across the gap of the ceramic spacer. A 10 kV bias across the insulating spacer is ideal in order to have enough acceleration of the electrons to overcome an approximate 3 kV loss through the metalized surface of the diamond. The metallization on the diamond surface acts as a source for the replenishing current for secondary electron formation.

2.2 Secondary Electrons

Once the primary electron beam reaches the diamond surface, the high energy electrons collide with electrons in the diamond. This generates a cascade of secondary electrons through

an electron-hole formation process. An applied field on the diamond allows secondary electrons to drift to the opposite side of the diamond. A hydrogenation process creates a negative electron affinity (NEA) surface so that the secondary electrons are able to be emitted from the diamond.

2.2.1 Diamond Etching, Hydrogenation, and NEA

The surface of the diamond is prepared to facilitate the emission of secondary electrons. An etching procedure attaches a strong oxygen bond to the open carbon bond on the diamond surface. The hydrogenation process essentially replaces the oxygen bonds with hydrogen bonds and is shown in Figure 2.1.

	0-0-0-0	H-H-H-H-H
C-C-C-C-C	C-C-C-C	C-C-C-C-C
C-C-C-C-C	C-C-C-C-C	C-C-C-C-C
Diamond surface	Etching (adds O	Hydrogenation
before (open C	bond, strong)	(adds H, mildly
bonds on end)		strong)

Figure 2.1: Diamond Etching and Hydrogenation A representation of the diamond surface before and after the etching process as well as after hydrogenation.

The hydrogenation process creates an NEA surface on the diamond. The NEA surface lowers the required band gap energy for secondary electron emission. These secondary electrons can be emitted from the diamond surface with little energy loss or low thermal emittance. This causes a cascading effect in which approximately 100 secondary electrons are produced per primary electron, equating to a gain of two orders of magnitude. For gain measurement purposes, secondary electrons are accelerated towards an anode placed a few mm from the diamond surface with a separate voltage bias. When the hydrogenated surface is exposed to air, water vapor attaches itself to the surface, which can decrease photoemission; one solution is to reheat the capsule to ~350°C, discussed in Section 3.3.1. Impurities in the diamond can decrease the gain for this process. Figure 2.2 illustrates the entire secondary electron emission process.



Figure 2.2: Primary and Secondary Emission The diagram above illustrates the primary and secondary electron emission processes.

2.3 Materials Selection

Components for the capsule were mostly based on the dielectric and thermal properties for each material. Another factor that also contributed to the materials section was the feasibility to purchase or acquire the material commercially.

2.3.1 Cathode Material

For initial experimentation, a metal cathode is to be used. Metal cathodes generally have low quantum efficiency (QE) and require ultraviolet (UV) photons; however, for preliminary testing, a metal cathode is sufficient due to its inert and durable qualities. In general, the cathode material can be a thermionic emitter, photoemitter, or field emitter. Current and future plans involve a more dedicated use of semiconductor cathodes, such as gallium nitride (GaN) [6].

2.3.2 Diamond

Diamond is the ideal material to be used as an amplifier [4]. Diamond can easily form an NEA surface, which helps create high secondary electron yield (SEY) and narrow energy distribution of secondary electrons. The high breakdown field, mobility, and saturation velocity are also important in high field electron guns. Diamond has great mechanical strength, electrical properties, and thermal properties, which will be important during the fabrication procedures. Initial capsule fabrication processes used low optical quality, high-carbon diamonds to determine joining procedures. A single crystal, chemical vapor deposition (CVD) diamond is desirable to

have a minimal amount of impurities for maximum amplification and secondary electron emission. The dimensions of the diamond are limited by the feasibility of commercially available products, resulting in a 4 mm x 4 mm square. The diamond thickness is determined by the transport of electrons through diamond, requirements of the electron gun, and its mechanical strength and ability to dissipate heat. Table 2.1 illustrates the important properties of diamond [4] and a comparison to a similar material [7].

Properties	Diamond	Silicon
Mobility Electrons $\left[\frac{cm^2}{Vs}\right]$	≤ 2200	1600
Thermal Conductivity $\left[\frac{W}{cm \cdot ^{\circ}C}\right]$	6 – 20	1.48

Table 2.1: Diamond Properties

A table listing the most important properties of diamond to be used as an amplifier along with comparisons to silicon.

2.3.3 Ceramic Spacer

A spacer must be used to create an accelerating gap of a few millimeters between the cathode and diamond in order to minimize space charge effects on the primary electron beam. This spacer should be able to hold off up to at least 5 kV. There is a loss of approximately 3 kV which occurs through the metalized diamond layer and the resulting voltage must be high enough to accelerate the primary electrons to create secondary electrons. A 5 kV bias is the minimum voltage bias needed. Ideally, a 10 kV bias between the cathode and metalized diamond layer is sufficient to ensure high gain, especially a gain in the range of 1 - 2 orders of magnitude.

Alumina (Al_2O_3) was a strong choice due to its insulating capabilities [8] and is able to hold off > 10 kV. This material was also chosen for its thermal properties, allowing for an appropriate temperature gradient to exist across the alumina when heating from one end. The temperature gradient is important during the soldering process explained in Section 4.1.3.2. Table 2.2 lists the pertinent properties of alumina [9] and another similar material for comparison [10].

Properties	Alumina	Cordierite (silicate)
Compressive Strength [MPa]	3600	500
Thermal Conductivity @ 20°C [$\frac{W}{mK}$]	18	2.0
Dielectric Strength @ 20°C [$\frac{kV}{mm}$]	25	18

 Table 2.2: Comparing Alumina Properties

A table listing the most important properties of alumina for the diamond amplified photocathode capsule and a comparison to another option.

2.4 Electronic Field and Electron Trajectory Simulation

Using Opera 13.0 [11], a simulation was performed to determine if the proposed material and dimensions would interfere with a primary electron traveling from the cathode to the anode. Electrons should be emitted from the cathode, accelerated through the gap, amplified in the diamond, and accelerated toward the anode without any obstructions. Figure 2.3 shows the result of the simulation. A static field analysis was used to generate the equipotential lines (blue) around the capsule with input settings of 5,000 V on the cathode, -5,000 V on the anode, and ground at the diamond. A typical trajectory for an electron emitted from the photocathode

surface (purple) was also simulated with initial conditions of negligible initial energy and originating in the 0.5 mm on the x axis, 1.0 mm on the y axis. The simulation showed that the electron is able to be emitted from the cathode and travel to the anode successfully. As a result, the proposed materials and dimensions should not hinder the process.



Figure 2.3: Simulation

An equipotential lines (blue lines) and electron trajectory (purple line) simulation using Opera 13.0. The simulation shows an electron with negligible initial energy originating from (0.5,1.0) will pass through the diamond to the anode unobstructed.

3. Low Temperature Capsule

Many iterations and tests of various materials, construction methods, and procedures were performed throughout the development of the low temperature diamond amplified photocathode capsule. The final version of the low temperature capsule consists of indium cold-welded joints created at room temperature between a nickel or nickel-plated cathode, a nickel-plated alumina spacer, and a square diamond. Prototypes used for leak checking and voltage hold-off tests contain a nickel cathode with a hole throughout the center and a groove on the side. The indium cold welded capsule is particularly beneficial to more sensitive cathodes, such as CsK₂Sb, since the joining process is performed at room temperature. The material selections, joining procedures, and integrity checks will be described during the fabrication process in Section 3.1. New research with diamond amplifiers and hydrogenation and its impact on the indium cold welded capsule will be discussed at the conclusion of this chapter in Sections 3.2 and 3.3. All details and procedures outlined in this chapter are also disclosed in the non-provisional patent, "Vacuum Encapsulated, Hermetically Sealed Diamond Amplified Cathode Capsule and Method for Making Same" [12].

3.1 Low Temperature Capsule Fabrication

Included in the low temperature fabrication section are the reasons for selecting the specific materials, the careful sample preparation process, and the cold welding technique.

3.1.1 Material Selection

Indium, Ni-plated alumina, diamond, and nickel were all specifically selected as the materials to be used in the low temperature capsule. This section lists all of the factors that were taken into consideration when selecting each material.

3.1.1.1 Indium

Indium (In) was chosen because of its ability to be used as a cold welding material. Due to its softness and malleability, indium easily deforms to fill in any gaps in the joining surfaces with little pressure [13]. This results in a hermetic seal between the two surfaces, which is required for the capsule. Its malleability is also critical when forming the material to the shape of the desired joint, which was important with indium ring formation in Section 3.1.2.1. Indium retains its malleability and joint reliability when cooled to cryogenic temperatures approaching absolute zero, temperatures which may be duplicated in operating RF injectors. An indium cold welded joint is able to deform in cryogenic conditions without joint failure. When considering ultra-high vacuum (UHV) applications, indium is a great choice due to its low vapor pressure. Indium is also known for its adherence properties to non-metals, notably diamond, which was used in the cold welding process [14].

3.1.1.2 Alumina

Adhering indium to bare alumina was difficult and shown to be unreliable when creating a hermetic seal. Alumina with a metal coating on the flat joining surfaces was chosen to help make a vacuum tight seal in the cold welding process. An industrial process [15] was used to metalize the alumina, which involved baking a moly-manganese paint onto the surface followed by metal plating. The metalized alumina was commercially purchased from CeramTec North America Corporation. The metal plate chosen was nickel due to its sealing compatibility with indium. The Ni plating has a ceramic-to-metal joint leak rate of $\leq 10^{-8} \frac{(mbar \times 1)}{s}$ and bond strength of $> 100 \frac{N}{mm^2}$ [15]. Figure 3.1 and Figure 3.2 are examples of the metalized alumina used in this process with dimensions of 6 mm outer diameter (OD), 3 mm inner diameter (ID), and 3.5 mm thickness.



Figure 3.1: Metalized Alumina An example of a metalized alumina spacer with 6 mm OD, 3 mm ID, and 3.5 mm thickness (height).



Figure 3.2: Metalized Alumina Specifications An AutoCAD drawing of the metalized alumina with specifications. All dimensions are in inches.

3.1.1.3 Diamond

As previously stated in Section 2.3.2, the square diamond chosen has dimensions approximately 4 mm x 4 mm with 381 µm thickness. For gain measurement purposes, a CVD grown single crystal diamond with less than 1 ppb nitrogen content and high gain should be used; however, for development phases, a poor quality black diamond was substituted. Figure 3.3 is an example of the lower quality black diamond used.



Figure 3.3: Low Quality Diamond An example of a lower quality square black diamond used in prototype capsules.

3.1.1.4 Nickel Cathode

A basic metal cathode with 6 mm OD and 1 mm thickness was used due to its low outgassing, stable surface which will be contained in the capsule. Nickel was chosen since it can easily form metal to metal cold welded bonds to form a hermetic seal with indium. Any metal cathode can be used with the requirement that the outside rim (6mm OD, 3mm ID) is sputter coated with at least 50 nm of Ni. Figure 3.4 illustrates a typical nickel cathode.



Figure 3.4: Nickel Cathode An example of a polished nickel cathode with 6 mm OD and 2 mm thickness.

Prototypes were constructed using nickel pieces with a hole of 3 mm ID throughout the center, whose purpose was to be able to verify that the seal was indeed leak-free (see Figure 3.5 and Figure 3.6). A groove on the outer diameter was used in the leak check process to hold down the sample, described in Section 3.2.1. The procedure used in this chapter will involve these nickel samples with the hole; however, the procedure can be modified slightly to include nickel cathodes with good QE to be used later for gain measurements.



Figure 3.5: Nickel Prototype Cathode Specifications An AutoCAD drawing of a nickel cathode prototype with an ID. All dimensions are in inches.



Figure 3.6: Nickel Prototype Cathode An actual nickel prototype cathode with a groove and ID.

3.1.2 Sample Preparations

All sample preparations were performed with contamination-free procedures suitable for UHV environments. This involved careful handling of all materials with gloves and cleaning methods. It is important to note that the sample preparation process must be strictly followed for the cold welding process to be successful.

3.1.2.1 Indium

The indium used was 0.5 mm diameter 99.999% pure indium wire (purchased from Indium Corporation of America, Indalloy #4). A ~1 inch segment was cleaned with acetone and dried with high purity gaseous N_2 . The wire was wrapped around a 3.81 mm diameter pin, which is demonstrated by Figure 3.7 and Figure 3.8.



Figure 3.7 (left): Indium Wire Wrapping A demonstration of wrapping indium wire around a pin to create an indium ring. Figure 3.8 (right): Indium Wire Wrapping A demonstration of wrapping indium wire around a pin to create an indium ring. The ends were cut on a diagonal with a clean razor blade shown in Figure 3.9. Indium's self cold welding ability allowed for the freshly cut ends to be pinched together with tweezers to form a continuous ring [16]. Figure 3.10 demonstrates the joining process. Another indium ring was constructed in a similar manner using a 3.175 mm diameter pin then forming the ring into an approximate 4 mm x 4 mm square. Figure 3.11 shows the two indium pieces after the joining process.



Figure 3.9: Indium Wire Cutting

A demonstration of cutting the indium wire with a clean razor blade to create an indium ring.



Figure 3.10: Indium Ring Joining A demonstration of joining the freshly cut indium to create a continuous ring by squeezing together the ends with tweezers.



Figure 3.11: Indium Ring and Square A picture of an indium ring (left) and indium square (right) after the joining process.

The indium ring and the indium square were both etched to remove surface impurities, which will allow indium to be able to be cold welded to nickel successfully [13],[14]. Each indium piece was placed in a 9 parts de-ionized (DI) water : 1 part HCl solution for 5 minutes. Figure 3.12 illustrates the indium etching process. Both indium pieces were immediately placed into a beaker of acetone following the etching procedure until they were ready for use. Approximately 65 out of 71 indium rings were formed and etched in this manner. Out of 65 rings fabricated, 36 rings were used to create partial prototype capsules and 6 were used to create complete prototype capsules. Partial prototype capsules do not contain all of the materials in Section 3.1.1 and contain substitutions, such as replacing the Ni-plated alumina for a solid Ni piece or diamond for glass. The partial prototype capsules were mainly used to check adhesion and concentricity problems.



Figure 3.12: Indium Etching An example of an indium ring during the etching process.

3.1.2.2 Alumina

The metalized alumina described and shown in Section 3.1.1.2 was used. Each metalized surface was circularly sanded with 600 grit SiC sandpaper to remove surface oxidation which then causes the metalized surfaces to appear bright. Only a few layers are removed from the surface as not to remove the metallization from the alumina. Nickel is best etched by first using an oxidizer then an acid [17] to roughen the surface and remove the surface oxidation. The metalized alumina was placed into a solution containing 9 parts DI water : 1 part HNO₃ for 1.5 minutes. The orientation of the alumina on its curved side allows for both metalized surfaces to be exposed to the solution during the etching process, illustrated in Figure 3.13. The metalized alumina was transferred to a solution of 4 parts DI water : 1 part HCl and positioned in the same orientation in the beaker for 5 minutes. The alumina was placed into an acetone bath immediately following the etching procedure and dried with high purity gaseous N₂ right before use. Approximately 12 out of 14 alumina pieces were prepared in this manner. 3 alumina pieces were used in complete prototype capsules.


Figure 3.13: Alumina Etching A picture of the alumina etching process, depicting the orientation of the alumina on its side.

3.1.2.3 Diamond

The diamond follows the same etching process as the alumina. If this diamond was a high quality diamond to be used in a gain measurement, it arrives pre-cleaned and only needs one side to undergo the hydrogenation procedure. 3 low quality diamonds were used to create complete prototype capsules.

3.1.2.4 Nickel

The prototype nickel cathode was polished with 600 grit SiC sandpaper to roughen the surface for better indium adhesion. One flat nickel surface was circularly sanded until deep scratches are removed from the surface. The gain measurement cathode was polished to

approximately 1 μ m. Both nickel cathodes were then etched in a 9 parts DI water : 1 part HNO₃ solution for 3 minutes with joining surface facing upwards in the beaker. The nickel cathodes were immediately transferred to a 4 parts DI water : 1 part HCl solution for 10 minutes with the same orientation. Figure 3.14 shows both the nickel cathode and the prototype cathode during the etching process. After etching, both nickel cathodes were immediately transferred to an acetone bath and dried with high purity gaseous N₂ until it is ready for use. The 3 mm diameter center of the gain measurement cathode would undergo laser cleaning if it were to be used for a gain measurement at a later point. Approximately 36 out of 42 Ni cathodes were prepared using this buffing and etching technique. 3 prototype Ni cathodes were used to create complete prototype capsules.



Figure 3.14: Nickel Cathode Etching The prototype nickel cathode (left) and nickel cathode (right) during the etching process.

3.1.3 Cold Welding Procedure

The cold welding procedure describes the procedure for construction of a prototype indium capsule. Suggestions were added for fabrication of a gain measurement indium capsule. Approximately 3 complete prototype cathodes were fabricated using this cold welding procedure.

3.1.3.1 Vacuum Press

The vacuum press is the vacuum chamber in which the capsule was cold welded to create a hermetic seal. The vacuum press consists of an anvil attached to the bottom port of a standard 2 ³/₄" Conflat (CF) cube. A holder for the samples attaches on top of the anvil and is fixed by a set screw. The sample holder also maintains concentricity of the capsule. A ram is attached to a modified right angle valve and sits on top of the cube. The ram is connected to a standard ¹/₂-13" jack screw with an external torque arm, which can be tightened to lower the ram. The ram should be lowered such that it meets up with the samples held on top of the anvil and exerts enough pressure for the cold welding process; however, too much or uneven pressure exerted or may result in a cracked diamond or improper seal. The vacuum press also contains ports for various vacuum pumps, including a scroll-turbo combination pump and ion pump. Figure 3.15 through Figure 3.19 illustrate the vacuum press and the components used in the process.



Figure 3.15: Modified Angle Valve

A picture of the modified angle value with $\frac{1}{2}$ -13" jack screw with ram attached.



Figure 3.16: Ram Housing

A picture of the housing in which the ram sits to ensure linearity of the ram's downward motion. This was constructed from a CF double blank and contains a linear ball bearing in the center.



Figure 3.17: Anvil in Cube A picture of the anvil inside the 2³/₄" cube. The modified angle valve sits at the top of the cube and the ram would directly line up with the center of the anvil.



Figure 3.18: Vacuum Press System A picture of the complete vacuum press system with turbo pump attached (left).



Figure 3.19: AutoCAD Vacuum Press System

An AutoCAD drawing of the main components used to construct the vacuum press. The ram is positioned such that it directly aligns with the anvil inside the cube.

If this capsule were to be used for a gain measurement, the ram would contain a mechanism that would grip the cathode and lower it when needed, similar to the mechanism utilized in Section 4.1.3.1.

3.1.3.2 Cold Welding

This cold welding process describes the procedure for fabrication of a prototype capsule. The procedure should begin immediately after each sample's etching process or preparation. Samples were stacked in the holder, shown in Figure 3.20, so that all pieces line up with the indium rings to form a complete seal. The square diamond was placed into the square recess containing a 0.7 mm deep counter bore, whose purpose is to provide a flat cold welding space and ensure the diamond's alignment. The indium square piece was placed directly on top of and aligned with the square diamond. The alumina spacer was placed on top of the indium square, followed by the indium ring and nickel piece with the hole in the center. Figure 3.21 demonstrates the stack of samples in the holder.



Figure 3.20: Square Recess Sample Holder The holder in which the samples were stacked. The three pegs were used for centering, along with the square recess for the diamond.



Figure 3.21: Components in Sample Holder An example of the stacked components in the holder.

The ram was then lowered by the attached torque arm. Approximately 1.0 Nm of torque was used to slowly compress the indium by the ram and form the capsule. A compression test was performed to determine the adequate torque needed to create a hermetic seal. An indium ring was compressed between a Ni prototype cathode and a Ni cathode. The compression was measured with a micrometer between a Ni prototype cathode and a Ni cathode for 0.5 Nm step increments of torque. Figure 3.22 shows the results of the compression test. The compression test showed that at least 1.0 ± 0.1 Nm was necessary to compress the indium approximately 0.480 \pm 0.001 mm. Compressing the indium less than 1.0 Nm did not apply enough pressure for the indium to form a seal whereas compressing the indium more than 1.0 Nm cracked the diamond.

Figure 3.23 and Figure 3.24 demonstrate the compression process. Figure 3.25 and Figure 3.26 illustrate a completed capsule after it is removed from the chamber for integrity checks.



Figure 3.22: Indium Compression Test

The plot shows the results of indium compression for varying values of torque from the torque arm on the modified angle valve. Uncertainties on compression are ± 0.001 mm and uncertainties on torque are ± 0.1 Nm.



Figure 3.23 (left): Before Indium Compression A demonstration before the compression process by the ram. Figure 3.24 (right): After Indium Compression After the compression process.



Figure 3.25 (left): Low Quality Diamond Prototype Capsule A complete indium cold welded prototype capsule with a black diamond. Figure 3.26 (right): Higher Quality Diamond Prototype Capsule A representation of what a complete indium cold welded prototype capsule with a higher quality diamond would appear as.

The cold welding procedure would differ slightly if a capsule were to be made for a gain measurement in order to encapsulate vacuum inside the capsule. The nickel cathode must be loaded onto a holding mechanism on the ram instead of being placed on the stack in the sample holder. The vacuum press chamber would then be sealed and evacuated. This would be accomplished by a scroll-turbo combination pump. To achieve a vacuum level of at least $10^{-9} - 10^{-10}$ Torr, a bake out of the chamber must be performed. The chamber should be heated between 100-140°C (just hot enough that the indium does not melt). The ion pump would be switched for the turbo pump when the current draw reads below 1.5 mA. The bake would be switched off when the pressure of the chamber is in the low- $10^{-8} - \text{mid}-10^{-9}$ Torr range. Once the chamber is cooled to room temperature (20° C) and the ultimate pressure of $10^{-9} - 10^{-10}$ Torr is reached, the cold welding process would begin. The ram would then compress the indium with 1.0 Nm of torque. Figure 3.27 illustrates the completed hermetically sealed capsule.



Figure 3.27: Hermetically Sealed, Indium Capsule A representation of what a hermetically sealed, indium capsule would appear as.

3.2 Integrity Checks

Checks were performed on many of the prototype indium cold welded capsules to ensure their ability to encapsulate vacuum and hold off significant applied voltage between the metalized side of the diamond and cathode. High success rates determined the suitable fabrication and cold welding procedure that would be implemented.

3.2.1 Leak Check

The leak check system shows the extent to which a capsule's indium cold welded joints can be pumped. In turn, this signifies if there is a leak or an opening in the joint, exposing the center of the capsule to atmosphere.

The leak check system consists of a modified 2 ³/₄" CF blank, which contains a groove for a 6.6 mm OD, 3.3 mm ID, and 1.8 mm thick o-ring and a 3.12 mm diameter hole through the center. Around the o-ring groove is four tapped places for standard 2-56 screws so that a clamping mechanism can be used to create a seal with the capsule. Figure 3.28 shows the modified CF blank with a representation of the prototype cathode clamped. A cross is connected to the modified CF blank which contains ports for an ion pump, micro ion gauge, convectron gauge, and adapters for a scroll-turbo combination pump. Figure 3.29 is a picture of the entire leak check system.



Figure 3.28: AutoCAD Modified Blank An AutoCAD representation of the prototype cathode clamped onto the modified CF blank in the leak check system.



Figure 3.29: Leak Check System A picture of the entire leak check system.

The purpose of the nickel piece containing a hole through the center is for leak checking. The groove in the side of the nickel piece fits into a slot of the clamping mechanism for the modified CF blank. Figure 3.30 shows how the capsule is situated into the clamping mechanism on the modified CF blank.



Figure 3.30: Capsule Leak Check A prototype capsule clamped onto the modified CF black of the leak check system.

The leak check system was pumped by a scroll-turbo combination pump followed by an ion pump. The pumping speed and pressure readings in a given amount of time were used as an indicator for the leak rate or size of the leak of the capsule. The leak check system, without a capsule attached, attained 10^{-6} Torr in 5 minutes after pumping with an 8 L/s scroll-turbo

combination pump. After pumping for 24 hours, the chamber would reach mid- 10^{-8} Torr. When pumped with a 6 L/s ion pump, the chamber would reach high 10^{-9} Torr in 1 hour. The addition of a successful capsule to the modified CF blank should not alter the pumping speed or time. Successfully sealed cold welded capsules able to reach an ultimate pressure of at least 10^{-9} Torr are considered to be leak free. Capsules with ultimate pressures > 10^{-9} Torr or altered pumping times after pumping with either the turbo or ion pump would be considered to have a leak. There was a 22.2% success rate (4 out of 18) for partial prototype capsules that did not follow all sample preparation procedures for each material used. Out of the 3 complete prototype capsules fabricated, a success rate of 66.7% was achieved. The 1 complete capsule that failed was due to uneven pressure from the torque wrench which cracked the diamond. Most partial prototype capsules failed due to a lack of concentricity. Other unsuccessful capsules could have resulted from failed adherence of indium to the nickel surfaces from improper surface cleaning or gaps in the indium joints.

3.2.2 Voltage Hold-off

The voltage hold-off system is used to determine if the capsule is able to hold off a significant voltage bias between the diamond and cathode. The system is designed to trip the high voltage supply when it cannot successfully hold off any more voltage.

The voltage hold off system consists of an insulated tube in which the capsule is placed and held in by springs. The springs attach to co-axial connectors which are attached to a high voltage power supply and ground. Figure 3.31 shows the voltage hold-off system with a capsule inserted. A cover is also placed on for safety reasons.



Figure 3.31: Capsule Voltage Hold off The voltage hold off test setup with a prototype low temperature capsule inserted.

A Bertan Associates, Inc. Model 380X 10 kV high voltage supply was used to apply the voltage. A trip current was set at 0.1 μ A so that the supply would turn off instead of allowing the capsule to arc. The voltage was slowly ramped up 200 $\frac{V}{\min}$ and the voltage was recorded right before the high voltage supply tripped. Successful capsules should be able to hold-off at least 5 kV. Approximately 9 partial and complete prototype capsules were used in voltage hold off measurement tests, achieving a 66.7% success rate. Out of the successful capsules, the mean

voltage hold off was 6073 V with a standard deviation of 1023 V. Figure 3.32 displays the results of the voltage hold off measurements as a histogram.



Figure 3.32: Voltage Hold Off Histogram A histogram displaying the results of the voltage hold off measurements with 9 low temperature capsules. Most fall within the 6000 V bin.

It is important to note that successful capsules also do not have any metal residue on the OD of the alumina, which would easily create a path for current to flow thereby holding off little to no voltage. Even though this does not hold off the 10 kV specified in Section 2.3.3, this test was not performed in a vacuum environment. The voltage hold off should significantly improve when tested under vacuum (see Section 4.2.2).

3.3 Limitations of Indium Cold Welded Capsule

A few drawbacks limit the use for the indium cold welded capsule. A possible solution includes opening the capsule while in the injector chamber with a retractable arm.

3.3.1 Hydrogenation Study

When the hydrogenated diamond is exposed to atmosphere, water molecules bond to the surface. E. Wang had shown that reheating of the diamond to 450°C after exposure to ambient air for 30 minutes restored the QE to 96% of the original [18]. The study produced a temperature curve (Figure 3.33) which showed that heating to at least 350°C restored approximately 85% of the QE while beyond 450°C degraded the QE. Since the diamond amplified photocathode capsule will be exposed to atmosphere during travel to the injector site, the injector should have the capability to reheat the diamond to at least 350°C. Indium's melting point at 157°C would limit the heating and ultimately, the gain.



Figure 3.33: Reheating Hydrogenated Diamond Study The Photocurrent vs. Temperature curve from "Systematic Study of Hydrogenation in a Diamond Amplifier." The dashed line is the photocurrent of a freshly hydrogenated diamond. The solid curve represents the photocurrent after heating the sample to the temperature indicated and allowing it to cool. The data points are current readings at various temperatures.

3.3.2 A Solution for the Indium Capsule

One possible solution to be able to use the indium cold welded capsule in an injector is to separate the capsule while in vacuum of the injector. With this idea, the capsule still retains its feasibility of transport to the injector site while protecting the sensitive cathode surface. The injector cavity would have to contain a heating element on or extending to the diamond. Another arm would hold the alumina attached to the cathode fixed. The diamond would be heated right below the melting point or the point at which the indium is soft enough for the diamond to be pulled off. The alumina would be able to hold off a temperature gradient such that the indium

attached to the diamond would only melt while the indium holding the cathode would remain solid. Now the diamond would be heated separately to remove the water molecules from the hydrogenated surface. Figure 3.34 and Figure 3.35 illustrate what this setup may look like. Another benefit to this active pumping method is that any out gassing from the cathode surface would be pumped away and not remain trapped inside the capsule, which would contaminate the sensitive cathode surface.



Figure 3.34: Before Indium Capsule Detach

In this diagram, a pincer mechanism (black) grips the diamond (pink) and heats it to $\sim 150^{\circ}$ C. When the indium is malleable enough, the mechanism slowly pulls the diamond off of the alumina (blue) which is still attached to the cathode (gray).



Figure 3.35: After Indium Capsule Detach

In this diagram, the diamond is separated from the capsule. Now, the cathode is actively pumped and the diamond can be fully heated to at least 350°C.

4. High Temperature Capsule

Due to the limitations of the indium cold welded capsule, a transition was made to focus on the development of a higher temperature diamond amplified photocathode capsule as a more feasible solution. As with the low temperature capsule, the high temperature capsule developed through many iterations, ideas, construction methods, and materials. The final version of the high temperature capsule consists of gold silicon (AuSi) soldered joints between a gallium nitride (GaN) cathode, a metalized alumina spacer, and a square diamond. In general, the high temperature capsule would be compatible with less sensitive cathode surfaces, such as metal cathodes. The fabrication process describes the material selections and joining procedures. Prototype capsules will also undergo integrity checks; however, the capsule constructed using an acceptable QE GaN and diamond can be used in gain measurements (Chapter 5). All details and procedures outlined in this chapter are also disclosed in the provisional patent, "Vacuum Encapsulated, High Temperature Diamond Amplified Cathode Capsule and Method for Making Same" [19].

4.1 Higher Temperature Capsule Fabrication

Included in the high temperature fabrication section are the reasons for selecting the specific materials, the careful sample preparation process, and the high temperature soldering technique.

4.1.1 Material Selection

AuSi, grooved Ni-plated alumina, diamond, and GaN were all specifically selected as the materials to be used in the high temperature capsule. This section lists all of the factors that were taken into consideration when selecting each material.

4.1.1.1 AuSi

Since the low melting point of indium limited the feasibility of the low temperature capsule, a new joining material was chosen for its ability to be heated to 350°C without melting during the reheating process for the hydrogenated diamond. The material chosen was 96.8% Au 3.2% Si, which is eutectic at 363°C [20]. This material must be soldered, not cold welded, which raises problems about outgassing causing contamination to the diamond and cathode surface (discussed further in Section 4.1.3.2); however, AuSi is still a good soldering choice for its ability to solder without flux in vacuum [21]. The AuSi soldered joints also exhibit good thermal properties and remain intact during temperature cycling.

4.1.1.2 Alumina

The alumina described in Section 3.1.1.2 was also used in the higher temperature diamond capsule. A groove of 13.7 mm thickness was added as a thermal break on the outside, which will be utilized in the soldering process (Section 4.1.3.2). The groove allows for a 1.52

mm thick side, which will be attached to the diamond, and a 0.81 mm thin side, which will be attached to the cathode. Figure 4.1 demonstrates the groove added to the metalized alumina.



Figure 4.1: Grooved Metalized Alumina An example of the grooved metalized alumina.

4.1.1.3 Diamond

Section 3.1.1.3, which described the characteristics of the diamond used for the low temperature capsule, also translates to the high temperature capsule. The capsule prototypes used for integrity testing contain a poor optical quality diamond, whereas the capsule which can be used for the gain measurement in Chapter 5 was a high quality diamond with significant QE. The high quality diamond used had dimensions of $4.148 \times 4.148 \times 0.290 \text{ cm}^3$.

4.1.1.4 Cathode

A cathode material that was chosen for prototype cathodes would be similar to the basic metal cathode chosen in Section 3.1.1.4; however, a more favorable metal to use would be

copper. The metal cathode surface is less sensitive to contamination and would be advantageous during the soldering process when gold particles will most likely be evaporated from the solder. Figure 4.2 shows a typical copper cathode.



Figure 4.2: Copper Cathode A typical copper cathode used in prototype capsules polished with a diamond suspension solution of 1 μ m grit size.

The cathode material that was chosen for gain measurement purposes was GaN, which was Mg doped, $p \sim 1 \ge 10^{19}$ cm⁻³ and has a band gap energy of approximately 3.5 eV. The dimensions used was a 1 cm x 1 cm square with a 0.1 µm thick GaN layer grown by molecular beam epitaxy on top of a 0.3 mm thick sapphire substrate. The mostly transparent GaN cathode will have benefits during the gain measurement, for the light source used will illuminate the capsule from the back through the GaN and emit electrons toward the diamond. Front illumination is difficult and requires optimal conditions. Using the equation:

$$I = \frac{QE \ x \ eP}{E\gamma}$$

in which I is the current, e is the charge of an electron, P is the transmitted power, and QE is the quantum efficiency of the material. Assuming the QE of a typical copper cathode $\sim 10^{-5}$, QE of hydrogenated diamond at 260 nm $\sim 10^{-4}$, and $\sim 10\%$ transmission through the 30 nm Pt layer on the diamond, front illumination would require a gain of ~ 100 to be able to detect any signal. Using back illumination will result in less of a loss than illuminating from the front of the diamond, since the sapphire is mostly transparent. One disadvantage of using GaN for the cathode material is the sensitive surface, which needs to undergo an extensive etching procedure and can be easily contaminated during the soldering procedure. Figure 4.3 is a picture of a GaN sample.



Figure 4.3: GaN A 1 cm x 1 cm square GaN sample.

4.1.2 Sample Preparations

As with the low temperature capsule, all sample preparations were performed with contamination-free procedures suitable for UHV environments. This involved careful handling of all materials with gloves and cleaning methods. The AuSi soldering is less sensitive than In cold welding and the AuSi does not require any chemical etching processes.

4.1.2.1 AuSi

The 98.6% Au 3.2% Si solder used was purchased in ribbon form, 25.4 mm wide x 0.51 mm thick (purchased from Indium Corporation of America, Indalloy #184). Two pieces were made from this ribbon: a ring, for the joint between the GaN and alumina, and a square, for the joint between the diamond and alumina. The AuSi ring was made by precision punching a 6.35 mm OD centered around a 3.175 mm ID. The AuSi square was constructed by manually cutting a 4 mm x 4 mm square with a razor blade around a precision punched 3.175 mm ID in the center. Both faces of the AuSi ring and square pieces were lightly buffed with 600 grit SiC paper until the oxidized layer had been removed and appeared bright. The pieces were rinsed thoroughly in an acetone bath to remove sanded particles and dried with high purity gaseous N₂ right before use. Approximately 22 AuSi rings were created and prepared in this manner, in which 14 were used for partial prototypes, 6 for complete prototypes, and 2 for a gain measurement capsule. As with the low temperature capsule, partial prototype high temperature capsules do not contain all of the materials in Section 4.1.1 and contain substitutions, such as replacing the GaN for a Ni or Cu cathode or diamond for glass or a Cu square. The partial prototype capsules were mainly

used to check adhesion and concentricity problems. Figure 4.4 is a picture of the AuSi ring and square pieces.



Figure 4.4: AuSi Ring and Square An example of an AuSi ring (left) and AuSi square (right).

4.1.2.2 Alumina

The metalized alumina endured the same buffing and etching procedure described in Section 3.1.2.2 for the low temperature capsule. After the etching procedure, both metalized faces of the alumina were sputtered with 50 nm of Au. It is important to mask the inner and outer surfaces so that voltage hold-off properties are not compromised during the gain measurement. Approximately 11 alumina pieces were prepared in this manner, in which 7 were used for partial prototypes, 3 used for prototypes, and 1 for a gain measurement capsule. Figure 4.5 and Figure 4.6 are pictures of the alumina with masks before the sputtering process. Figure 4.7 shows the grooved alumina after the sputtering process.



Figure 4.5 (left): Overhead Masked Alumina An overhead view of the masked alumina. The two alumina were masked with a cut segment of 3 mm thick viton o-ring to protect the ID. The OD of the alumina were masked with two 5.6 mm ID o-rings. Figure 4.6 (right): Profile Masked Alumina A profile view of the masked alumina.



Figure 4.7: Sputtered Alumina Examples of the grooved alumina after the masked sputtering process.

4.1.2.3 Diamond

One face of the diamond was metalized with a 3 mm diameter circle of 30 nm of Pt. Also, 50 nm of Au is sputtered from the 3 mm diameter of the Pt section to the edges of the diamond. The opposite face of the diamond was hydrogenated in a 3 mm diameter circle in the center, while also careful not to scratch off the metallization. Even though a mask was used to only hydrogenate the 3 mm diameter center of the diamond, hydrogen can get in between the mask and the diamond surface, also hydrogenating the other portion of the surface. Figure 4.8 shows how the diamond was prepared and masked off for the sputtering process. Figure 4.9 is a picture of the diamond metallization after the sputtering process.



Figure 4.8: Masked Diamond

An example of a diamond prepared for the Au sputtering process. The 3 mm diameter mask was placed over the center of the diamond and held by thin wire and Kapton tape on a microscope slide after it was sputtered with 30 nm Pt.



Figure 4.9: Sputtered Diamond The diamond after the sputtering process with both Au and Pt.

After the diamond was sputtered, it was placed onto a button heater, metalized side on the button heater surface, and set up in the hydrogenation system. Figure 4.10 shows the diamond as it was clamped onto the button heater for the hydrogenation process. Before the hydrogenation process, a bake of the chamber was performed and elements of the hydrogenation system were degassed. This included the button heater, which was heated to 830°C, the hydrogen cracker, which was operated at 55 W, a residual gas analyzer (RGA) filament, and an ion pump. After degassing, hydrogen was leaked into the chamber at a rate 20 - 30 times more than any background element determined by the RGA. The button heater was reduced to 300°C and the hydrogen cylinder was turned off. In situ measurements of the photo yield of the hydrogenation diamond were conducted before the diamond was removed from the chamber and is discussed in Section 5.1.2. Approximately 9 low quality diamonds were used to construct 6 partial prototype

capsules and 3 prototype capsules, none of which were hydrogenated. 1 high quality diamond was sputtered and hydrogenated and used in the gain measurement capsule.



Figure 4.10: Hydrogenation Preparation The diamond positioned on the substrate heater, which will be inserted into the vacuum chamber for hydrogenation. A mask was placed over the diamond, resulting in only a 3 mm diameter section undergoing hydrogenation.

4.1.2.4 Cathode

Preparations for the copper cathode used for prototype cathodes involve manually step polishing the surface to 1 micron grit size with a water based diamond suspension solution. A 3 mm diameter portion in the center of the copper was masked off with a 3 mm diameter macor disc. Another section of the copper was masked off with a 18.8 mm OD, 7.4 mm ID washer. This resulted in sputtering Au a 7.4 mm OD, 3 mm ID ring on the copper cathode. The GaN must undergo an etching and heating procedure, which removes surface oxides, to be a functional cathode with QE greater than 10^{-3} . The procedure started with placing the GaN in a beaker of acetone for 10 minutes, while the acetone was constantly swirled either manually or by an ultrasonic machine. The GaN was rinsed thoroughly with constantly flowing DI water for 5 minutes. The piranha etching solution—a 3:1 ratio of H₂SO₄:H₂O₂—was heated to approximately 90°C and the GaN was etched for 10 minutes. Another rinsing of the GaN with constantly flowing DI water for 5 minutes for 5 minutes removed any residual acids on the surface. Figure 4.11 shows the rinsing stage of the etching process.



Figure 4.11: GaN Rinsing A representation of GaN rinsed with constantly flowing DI water.

Finally, the GaN was placed in a beaker of DI water while it was transported to the sputtering site. High purity gaseous N_2 was used to completely dry the GaN immediately before it was sputtered. As with the copper cathode, the GaN was also sputtered with a 7.4 mm OD, 3 mm ID ring on the GaN. The GaN must also undergo a heating cycle to further remove contaminants from the surface; however, this will be covered during the second step of the soldering process in Section 4.1.3.2. Approximately 5 GaN cathodes were prepared in this manner, in which 1 was used for a partial prototype, 3 were used for prototype capsules, and 1 was used for a gain measurement capsule. Figure 4.12 shows the setup in the sputtering apparatus while Figure 4.13 is a picture of the resulting sputtering process.



Figure 4.12: Masked GaN

A picture of the GaN setup for the sputter coating process. The two masks were placed on the GaN surface. Two 3 mm dia were stacked in the center while a 50 micron gold wire held it in place. The setup was taped using Kapton tape on a clean microscope slide.


Figure 4.13: Sputtered GaN A picture of the GaN after sputtering. The sputtered ring is off center in order to choose a pristine GaN surface.

4.1.3 Two Step Soldering Procedure

The following procedure describes the fabrication process for a gain measurement capsule. The soldering of the capsule took place in two stages: first, the diamond was soldered to the thick side of the alumina; second, the cathode was soldered to the thin side of the alumina. All soldering was performed in a specially designed vacuum chamber under vacuum conditions.

4.1.3.1 Brazing Chamber

A brazing chamber was constructed out of UHV components to solder the capsule together in two stages. A 1200°C button heater was mounted below a standard 2 ³/₄" cube so that the top surface of the button heater was in the center of the cube. This allowed samples to be easily mounted on top of the button heater, where the samples were heated to be soldered, through an available port or window on the front of the cube. At the top of the cube, opposite

from the button heater, sat a modified angle valve with a precision steel ram. A standard ¹/₂-13" jackscrew converted its rotary motion into the linear motion of the ram, allowing it to move vertically. A two stage spring lock mechanism was attached to the base of the ram, which is utilized in the second step of the soldering process. The spring lock mechanism was a device which held the alumina, with soldered diamond side upwards, by a choker style clamp heat sink on spring loaded threaded pins. When pressure was applied to the bottom of the alumina, the pins inside the device would shift upwards and spring loaded plungers on the side of the device would lock the pins from being able to return downwards again. This allows the diamond side of the alumina to be raised, locked, and sealed onto a Kalrez o-ring, which is mounted in a groove on the bottom center of the device. Kalrez was chosen due to its low out gassing properties in a UHV environment and high operation temperature [22]. This enabled the hydrogenated side of the diamond to be sealed off before the bake process; outgassing of the solder and chamber would most likely destroy the hydrogenated surface. Once the spring lock mechanism was locked, the entire unit can be vertically raised or lowered; however, the diamond could not be unsealed unless it was manually unlocked out of the vacuum chamber. Another port on the cube contained a Bayonet Neill-Concelman (BNC) connector with attached wire to make QE measurements of the cathode before it was soldered together. The brazing chamber also had the ability for pumping and measuring vacuum with an attached ion pump, micro ion gauge, and angle valve which connected to a scroll/turbo combination pump. A leak valve with capillary tube was added for the first step of the soldering procedure only. The leak valve was connected to a 6.35 mm stainless steel tubing and was used to leak in hydrogen. Hydrogen was necessary in the first step of the soldering procedure to protect the hydrogenated diamond surface from

contaminants, such as outgassing from the AuSi solder. Figure 4.14 through Figure 4.20 are pictures and AutoCAD drawings of the brazing chamber and spring lock mechanism.



Figure 4.14: Button Heater A picture of the button heater in the center of the cube of the brazing chamber.



Figure 4.15: First Setup Brazing Chamber The brazing chamber during the first step of the soldering procedure. Attached to the top of the cube is a leak valve, which flows in hydrogen.



Figure 4.16: Second Setup Brazing Chamber

The brazing chamber setup during the second step of the soldering procedure. The leak valve was replaced by the modified angle valve with spring lock mechanism and the CF blank to the left of the cube was replaced by a BNC connector.



Figure 4.17: Spring Lock Mechanism

The spring lock mechanism on the ram attached to the modified angle valve. The spring loaded plungers are held in by plates. The heat sink is held underneath by spring loaded pins. The center protrusion on the mechanism houses the Kalrez o-ring.



Figure 4.18: Heat Sink

The heat sink which holds the grooved alumina. The thicker section of the alumina above the groove will be clamped in the center of the heat sink. The tapped holes on both left and right sides connect the pins, which are also attached to the mechanism.









Figure 4.20: Spring Lock Mechanism Demonstration Before (left) and after (right) demonstrating how the locking mechanism works. When the ram is lowered by the angle valve and the alumina makes contact with the GaN, the springs on the pin (green) contract. As a result, the mechanism (blue) is lowered and the heat sink holding the alumina with diamond (blue, very bottom) raises to the o-ring and applies pressure on the diamond. The plunger in the mechanism (pink) slips underneath the head of the pin (teal) due to the pressure from the springs in the mechanism (small green springs). The pins are unable to move downward again.

4.1.3.2 Two Step Soldering Procedure

The soldering process described was the procedure used to fabricate a GaN cathode capsule which can be used in the gain measurement discussed in Chapter 5.

The first step of the soldering procedure required soldering the thick side of the Au sputtered alumina to the metalized side of the diamond. A clean 1 cm diameter diamond was

placed on top of the button heater, followed by the diamond with the hydrogenated side downwards and the center of the hydrogenated surface over the center of the 1 cm diamond. The AuSi square piece was lined up with and placed on top of the Au metalized side of the diamond. The thick metalized side of the alumina was then lined up with and placed on top of the Au square piece. Finally, a clean 50 g weight was placed on top of the alumina to apply good thermal contact and pressure for soldering. The chamber was evacuated with a scroll-turbo combination pump to at least ~10⁻⁷ Torr. Hydrogen was leaked in through the leak valve at a rate such that the pumping rate was in equilibrium with the hydrogen leak rate. This equated to a pressure on the order of 10^{-6} Torr. Once at equilibrium, the button heater was raised to 370° C with 3.3 A. After soaking for 60 ± 10 minutes, the current on the button heater was switched off and the chamber was allowed to cool to room temperature. Approximately 60 minutes was necessary because previous soldering trials had shown that soldering for less time did not allow the AuSi to adhere to the surface and soldering for more time evaporated the AuSi, creating a brittle bond. Figure 4.21 shows the arrangement of the first part of the soldering process.



Figure 4.21: First Step Soldering The stack for the first step of the soldering procedure. The stack was, from bottom to top on the button heater, the 1 cm diamond, square diamond (hydrogenated side down, metalized side up), AuSi square, grooved alumina (thick side down), and weight.

Once cooled to at least 30°C, the brazing chamber was opened and all samples on the button heater were removed. The alumina, diamond side upwards, was tightened into the choker clamp style heat sink. The pins were inserted through the spring lock mechanism device and screwed into the heat sink to the appropriate length with springs between the device and heat sink. The spring loaded plunger pins and outer plates were also set into place. A clean Kalrez o-ring was inserted into the groove in the device. Figure 4.22 and Figure 4.23 show the intermediate, out of vacuum setup process.



Figure 4.22: Diamond Soldered to Alumina Hydrogenated and metalized diamond soldered to Au sputtered alumina removed from brazing chamber after the first step of soldering procedure.



Figure 4.23: Alumina Clamped into Heat Sink The alumina with soldered diamond clamped into the choker style heat sink, which will be attached to the spring lock mechanism.

The second step of the soldering process started by attaching the angle valve with spring loaded mechanism setup on top of the cube of the brazing chamber. The spring mechanism was loaded into the chamber such that the diamond was not sealed off yet by the Kalrez o-ring. On the button heater, a clean 1 cm diameter diamond was placed, followed by the GaN cathode on top with the Au metalized surface upwards. The AuSi disc was placed on top of the Au metalized section of the cathode. The entire stack on the button heater must also align with the alumina, such that the Au metalized alumina can be lowered by the modified angle valve to meet directly with the AuSi disc on the cathode. The wire connected to a BNC from the side of the cube was placed on top of the GaN cathode, not touching the AuSi ring. The wire had a modified eye or ring connector to apply good, flat electrical contact for the QE measurement. Figure 4.24 shows the setup before the chamber was sealed for the second step of the soldering process.



Figure 4.24: Second Step Soldering

A picture of the alumina clamped into the spring lock mechanism positioned directly above the GaN with AuSi ring. The eye or ring connector was placed on a blank section of the GaN and did not interfere with the lowering of the alumina.

The brazing chamber was then evacuated by the scroll-turbo combination pump to $\sim 10^{-9}$ Torr. At this time, the ram was lowered to the cathode such that enough pressure was applied to the alumina to push the pins in the spring lock device upwards and lock the device in the position with the diamond sealed off. Figure 4.25 through Figure 4.27 illustrate the locking process. Once the diamond was sealed off by the Kalrez o-ring, the mechanism was raised from the cathode. Since the sensitive hydrogenated surface of the diamond was sealed off, the complete outgassing of the chamber and AuSi solder on the cathode took place. It was important to completely outgas the solder so that the best vacuum would be encapsuled in the interior of the capsule.



Figure 4.25: Before Sealing Diamond A picture of the spring lock mechanism before the diamond is sealed off.



Figure 4.26: Locking the Mechanism

A picture of the spring lock mechanism in which the modified angle valve was tightened, which caused the locking mechanism to be triggered. At this point, the diamond is sealed off by the Kalrez o-ring.



Figure 4.27: After Locking the Mechanism A picture of the spring lock mechanism after the ram is moved upwards with the diamond sealed off.

A bake of the brazing chamber was performed, heating the chamber to approximately 150°C with percentage controllers and keeping the button heater slightly warmer than the chamber at 200°C. The samples on the button heater were at a higher temperature than the chamber to prevent particles outgassing from the chamber and adhere to the cathode surface. The ion pump was also degassed at that time and the turbo pump was valved out when the chamber pressure was below the pumping limit of the turbo pump. After the ion pump, brazing chamber, and button heater were completely degassed, which takes approximately 1 - 2 days of baking, the percentage controllers on the brazing chamber and applied current to the button heater were turned off. The system was allowed to cool to 20°C and attain an ultimate pressure inside the chamber of ~10⁻¹⁰ torr. The button heater was applied with 3.3 A of current, heating

the samples to 370°C, the melting point of the AuSi soldering. After the solder melted and the pressure inside the chamber started to decrease, the applied current on the button heater was slightly lowered so the solder would resolidify. The brazing chamber was then allowed to pump to restore at least 10⁻⁹ Torr vacuum.

At this point, a QE measurement of the capsule was determined. The copper cathode was negatively biased by a 30 V battery. A fiber coupled deuterium white light source with an optics setup created a 2 mm diameter spot on the cathode. An electrometer was also used to measure the background or leakage current and photoemission from the cathode. Figure 4.28 illustrates a schematic diagram of the equipment used in this process. In a spectrum from 190 nm to 400 nm, the deuterium source yielded a maximum of 11.0 ± 0.5 pA with 0.3 ± 0.1 pA background at 200 nm. Using the equation:

$$QE = \frac{Photocurrent [A]}{Net power [W]} \times hv [eV]$$

the QE of GaN was calculated to be $0.50 \pm 0.05\%$. Uncertainty of the QE was determined using standard equations of error propagation. Only a cathode with measureable QE was allowed to continue with the remainder of the soldering process.



Figure 4.28: GaN QE Measurement

A schematic diagram of the equipment used in the GaN QE measurement. A negative voltage from the battery biases the GaN cathode so that electrons are released and absorbed by the chamber. The GaN cathode is electrically isolated from the chamber. The brazing chamber is grounded. An electrometer floats to both bias the voltage and measure the current. A box (green) utilizes a triaxial cable to bias the voltages.

Finally, the ram was lowered, applying good thermal contact between the alumina and cathode and the applied current to the button heater was raised to 3.3 A so that the AuSi solder would melt again. It is important to note that the previously soldered joint between the diamond and alumina should not melt again, for melting soldered joints multiple times usually weaken their integrity and bond strength. The heat sink was designed such that heat was evenly dissipated from the alumina. Also, the groove in the alumina acted as a thermal break, allowing the bottom to heat more quickly than the top. The Kalrez o-ring also played an important role at this point: the top of the alumina still reached temperatures of approximately 180°C, a temperature only Kalrez could successfully operate while under UHV conditions. The capsule

was allowed to soak at 370°C for 60 ± 10 minutes and then the applied current on the button heater was reduced to 0 A. Figure 4.29 through Figure 4.31 show different views of the completed capsule after it was removed from the brazing chamber after cooling to room temperature.



Figure 4.29: Complete Capsule A picture of the completed, hermetically sealed high temperature capsule.



Figure 4.30: Complete Capsule- Overhead An overhead view of the completed capsule.



Figure 4.31: Completed Capsule- Back A picture of the completed capsule from the back of the GaN, where the light during the gain measurement will enter.

4.2 Integrity Checks

As with the low temperature prototype capsules, high temperature prototype and gain measurement capsules also went through testing to ensure its vacuum encapsulating and voltage hold off abilities.

4.2.1 Leak Check

Prototype capsules containing GaN could not be tested in the same manner as the leak checks performed with low temperature capsules in Section 3.2.1, for the GaN does not contain an ID through the center. The GaN was a specially ordered material with limited quantities and

not able to be easily machined. As a result, a crude test to determine leak was to pressurize a vacuum chamber with a small molecule gas, such as helium, to 20 psig for 2 minutes. The chamber was then opened, placed in a beaker of ethanol, and watched for any emerging bubbles. 2 out of 3 complete prototype capsules successfully completed this leak check with no emerging bubbles. The 1 complete prototype capsule that failed the leak check was due to centering issues, in which the diamond was off-center and exposed the center of the capsule.

The gain measurement capsule was leak checked based on differences between pumping time and pressure in a vacuum chamber with and without the capsule inside. The gain measurement capsule was placed inside the brazing chamber, which was known to achieve 10^{-6} Torr after pumping 5 minutes and 10^{-7} Torr after pumping 1 hour with an 8 L/s turbo pump. With a 6 L/s ion pump, the brazing chamber pumped down to 10^{-8} Torr in 5 minutes. If capsule was not hermetically sealed, the center would be exposed to atmosphere and create a virtual leak when being pumped in the brazing chamber. There was no indication of a virtual leak and the pressure readings are summarized in Table 4.1.

Time (min)	Pump used	Pressure (Torr)
5	Turbo	8.1 x 10 ⁻⁶
60	Turbo	6.6 x 10 ⁻⁷
5	Ion	5.9 x 10 ⁻⁸

 Table 4.1: Gain Measurement Capsule Pressure Readings

 A summary of the pressure readings from an ion gauge attached to the brazing chamber for various times and pumps used.

4.2.2 Voltage Hold-off

A similar procedure was used as in Section 3.2.2 to determine the AuSi capsule's voltage hold off ability for prototype capsules only. Successful capsules should also hold off at least 5 kV. 2 out of 3 prototype capsules were successful in holding off at least 5 kV, with a maximum of 6.18 kV. The prototype capsule that only held off 500 V failed due to not masking off the alumina during the Au sputtering process.

Another method was used to determine the maximum voltage able to be held off in vacuum by the gain measurement capsule. A copper tab connected to a low voltage feed-through was placed and pushing down on the metalized alumina surface attached to the diamond. This tab was used to provide a ground plane and be used for measurement. The GaN was electrically connected to a high voltage feed through. Figure 4.32 shows the capsule situated into the testing chamber. The chamber designed with additional elements can also be used in the gain measurement and described in detail in Chapter 5.



Figure 4.32: Voltage Hold Off in Vacuum An example of a prototype capsule situated in the voltage hold off test figure in a vacuum chamber.

A Keithley 6485 picoammeter and a Bertan Associates, Inc. Model 380X 10 kV high voltage supply were connected between the GaN cathode and the tab which was connected to the metalized side of the diamond. The trip current on the high voltage supply was set to 0.1 μ A to prevent the capsule from arcing, which would damage its voltage hold off abilities for further testing. Test voltages were applied between 50 V – 8000 V and the current was measured on the picoammeter. When the data was plotted, it should follow a linear trend, behaving in accordance with Ohm's Law; however, when the device is approaching the range at which it cannot hold off anymore voltage, the trend should appear more exponential.



Figure 4.33: Voltage Hold off 50 V – 8000 V A plot of the data taken of the voltage hold off measurements of the gain measurement capsule from 50 V – 8000 V. The error bars on current are $\pm 0.25 \times 10^{-12}$ A and error bars on voltage are ± 1 V.

This experiment showed that the capsule could successfully hold off at least 7900 V during the gain measurement. From the data in Figure 4.33, it is shown that the trend was linear until the high voltage supply tripped right below 8000 V. The plot did not become exponential before the high voltage supply tripped. Immediately before the voltage supply tripped, the voltage readings from the supply flickered for a few seconds. The large error bars on current measurements were due to the difficulty of making such precise measurements in the 10^{-12} A

range. Current readings greatly fluctuated specifically in the range from 50 V - 1000 V. The equation of the best fit line was

$$y = 2.71 \times 10^{-16} x + 1.69 \times 10^{-12}$$

with a $\chi^2 = 1.155$. The inverse of the slope, which would be the resistance of the capsule, was calculated to be 3.70 x $10^{15}\Omega$, which is considerably larger than expected. The 1.69 x 10^{-12} A offset most likely represents the background noise from the picoammeter. The voltage hold off test was repeated to investigate the higher current readings above the best fit line from 1000 V – 4000 V and the resistance calculation.



Figure 4.34: Voltage Hold off 100 V - 5000 V

A plot of the data taken of the voltage hold off measurements of the gain measurement capsule from 100 V – 5000 V. The error bars on current are $\pm 0.25 \times 10^{-12}$ A and error bars on voltage are ± 1 V.

The same setup and measurements conditions were repeated as with the measurements previously taken. These results equation of the best fit line was

$$y = 3.88 \times 10^{-16} x + 1.94 \times 10^{-12}$$

with a $\chi^2 = 1.048$. The inverse of the slope, which would be the resistance of the capsule, was calculated to be 2.57 x $10^{15}\Omega$, which is again considerably larger than expected. The best fit line and resistance are both similar to the measurements between 50 V – 8000 V. The resistance has slightly degraded, which may suggest that arcing may have occurred in the previous test and somewhat lowered its voltage hold off ability. This voltage hold off test appears to be smoother than the previous test, suggesting that the sensitivity in current measurements may have caused the large fluctuations.

5. Gain Measurement

The higher temperature AuSi soldered diamond amplified GaN photocathode capsule constructed in Chapter 4 can be used to make this measurement. The QE's of both the hydrogenated diamond and GaN photocathode were separately tested prior to the capsule's construction. A test chamber was built to apply three biases between the GaN cathode, metalized diamond surface, and anode and provide back-illumination from a light source. A description of how the emission measurements and analysis could be performed to determine the gain concludes the chapter.

5.1 Individual QE Measurements

The QE of each component was separately tested to have a better estimate for what the ultimate gain of the capsule should be. It is also important to note that individual QE's will degrade somewhat during the capsule construction period, due to contamination from soldering and exposure to atmosphere.

5.1.1 GaN QE

The QE measurement of GaN was discussed in detail in Section 4.1.3.2. Immediately before soldering the capsule, the QE was determined to be $0.50 \pm 0.05\%$.

5.1.2 Diamond Photo Yield Measurement

This measurement was performed in-situ after the hydrogenation process described in Section 4.1.2.3. The setup was similar to that of the GaN QE measurement, in which a 50 V battery negatively biases the cathode so that electrons are emitted to the rest of the chamber, which is at ground. The measured current is the replenishing current supplying the electrons that had been emitted from the cathode. A deuterium lamp from a white light source coupled with a monochromator and optics was scanned over a spectrum ranging from 190 nm to 270 nm. The schematic diagram of the setup is similar to the GaN QE setup in Figure 4.28, in which the GaN and brazing chamber were replaced by the diamond and hydrogenation system, respectively.

Three measurements over the range between 190 nm and 270 nm were taken, which included photocurrent, total power, and background power. The optics setup was changed for the power measurements, in which a power meter was set up at the same distance as the diamond. A LabView program coupled to the electrometer or power meter recorded the data over the spectrum. The QE at each wavelength was determined using the equation for QE listed in Section 4.1.3.2. Figure 5.1 through Figure 5.3 show plots of photocurrent, net power, and calculated QE over the range 190 nm to 270 nm, respectively. All plots include uncertainties in precision of equipment and error propagation. The results showed that the diamond had a maximum QE of approximately $8.7 \pm 0.2\%$ at 218 ± 5 nm.



Figure 5.1: Photocurrent vs. Wavelength

A plot of measured photocurrent [A] vs. wavelength [nm]. The uncertainties in wavelength are from the slit in the monochromator used, which has a width of approximately 1 nm. The uncertainties in photocurrent were due to the precision of measurement from the electrometer.



Figure 5.2: Net Power vs. Wavelength

A plot of net power [W] vs. wavelength [nm]. The net power was determined by the difference between the total power and background. The uncertainties in power were due to the precision of measurement from the power meter.



Figure 5.3: QE vs. Wavelength

A plot of the calculated QE [%] vs. wavelength [nm]. Uncertainties in QE were determined through normal equations of error propagation. Data below ~215 nm was excluded due to low photon energy.

5.2 Gain Measurement

A specialized chamber was built, similar to the voltage test system in Section 4.2.2. Further research towards making gain measurement on the high temperature capsule can determine its functionality as an amplifier.

5.2.1 Testing Chamber

The gain measurement of the high temperature capsule should be performed in a separate vacuum chamber than that of the soldering. Since the capsule and hydrogenated surface was exposed to atmosphere, the testing chamber must have the ability to heat the capsule to approximately 350°C. The chamber must also have the ability to bias the cathode, metalized diamond layer, and anode at three separate voltages. Figure 5.4 illustrates the main ideas needed for the gain measurement.





A diagram of the main ideas needed for the gain measurement. Three voltage biases were placed on the GaN surface, metalized layer of the diamond, and anode. The replenishing current will be measured at the metalized diamond layer. The gap between the diamond and anode may vary between 0.3 - 2.0 mm based on the required field in the diamond.

The testing chamber designed clamped the capsule to a sample holder. Electrical contact was made to the GaN surface by a copper plate with projections, which screwed into the sample holder. The sample holder was also attached to a 200 W cartridge heater. Two 1 mm thick sapphire washers were connected by ceramic screws between the sample holder and heater, which electrically, not thermally, isolated the two elements. Figure 5.5 illustrates the high quality diamond capsule inserted into the sample holder. Figure 5.6 shows the setup from the anode side and Figure 5.7 shows the setup from the cathode side. The sample holder attached to

the heater was situated in a standard 2 $\frac{3}{4}$ " CF tee attached to one port of a standard 2 $\frac{3}{4}$ " CF cube. Another port contained a thermocouple to take temperature readings at the heater before the electrical isolation at the sample holder. Two high voltage feed-throughs were used: one connected to the anode and fixed 0.3 - 2.0 mm away from the hydrogenated diamond surface and the other attached to the copper plate with projections on the GaN surface. The low voltage feed-through with copper tab was also attached on the cube. Two 2 $\frac{3}{4}$ " CF sapphire windows were positioned 180° apart and parallel to the face of the hydrogenated diamond and GaN back surface. This allowed for a light source to shine a 2 mm spot size directly on the back of the GaN and pass through the capsule. The testing chamber also had to ability to be pumped by a scroll-turbo combination pump and ion pump. Figure 5.8 shows the testing chamber and components.



Figure 5.5: Capsule in Gain Measurement Chamber A picture of the high quality diamond capsule inserted into the chamber for the gain measurement.



Figure 5.6: Capsule in Gain Measurement Chamber- Anode A picture from the anode side of the high quality diamond capsule in the chamber for the gain measurement.


Figure 5.7: Capsule in Gain Measurement Chamber- Cathode A picture from the cathode (GaN) side of the high quality diamond capsule in the chamber for the gain measurement.



Figure 5.8: Gain Measurement Chamber A picture of the entire gain measurement chamber. The picture shows an ion pump (black, left), flange with two high voltage feed throughs (right, center), ground plane (top), roughing valve (bottom, right), and heater (bottom).

5.2.2 Emission Measurements

In order to make a measurement of the gain of the capsule, either a pulsed light source incident on the cathode or a pulsed high voltage source on the anode must be used. An example would be to use a deuterium lamp from previous QE measurements as the incident light source and use a 10 kV high voltage supply coupled with a pulse generator. The high voltage able to be applied on the cathode and anode was determined during voltage hold off testing. When making

the gain measurement, the signal from the deuterium lamp with the high voltage only applied to the cathode should be analyzed first. An oscilloscope should be attached to the connector leading to the metalized diamond face in order to analyze the primary current. When the high voltage on the anode is applied, the resulting signal or the secondary signal should inverse its sign. The ratio of the current of the secondary signal to the primary current is then analyzed. A gain > 1 would be achieved if the secondary signal is inverse in sign and greater in magnitude than the primary signal. If the secondary signal is equal to the primary signal, the gain = 1.

6. Conclusion

The purpose of this thesis was to develop a fabrication procedure for the diamond amplified cathode capsule. Prototypes of both the low and high temperature capsules have shown to be successful during the integrity checks. As a result, the In cold welding and AuSi soldering fabrication procedures are acceptable in creating a diamond amplified cathode capsule. The In cold welded capsule is more favorable for sensitive cathodes whereas the AuSi soldered capsule is more favorable for less sensitive cathodes.

6.1 Results

The number of capsules fabricated by both methods and their success during integrity checking is summarized in Table 6.1.

Fabrication Procedure	In Cold Welding		AuSi Soldering	
Type of Capsule Fabricated	Partial Prototype	Complete Prototype	Complete Prototype	Gain Measurement
Number Tested for Leak Check	18	3	3	1
Number Successful in Leak Check	4	2	2	1
Number Tested for Voltage Hold Off	6	3	3	1
Number Successful in Voltage Hold Off	3	2	2	1

 Table 6.1: Summary of Results

This table summarized the number of capsules that were tested for each integrity check for each fabrication method. The number of successful capsules are also recorded.

Successful results were achieved during both integrity checks for both of the fabrication procedures. On average, there was a 66.7% success rate for In cold welded and AuSi soldered prototype capsules that endured leak checking and voltage hold off measurements. Overall, the partial prototype capsules, which did not follow the exact sample preparation and fabrication procedures described in Sections 3.1 and 4.1, had a lower success rate. The one gain measurement capsule indicated no sign of a leak and was able to hold off 8000 V before tripping the high voltage supply. Most of the failed capsules resulted from concentricity problems and unsuccessful adhesion during fabrication and metal contamination on the alumina.

6.2 Improvements

Improvements will be focused in both of the fabrication procedures to achieve near 100% success rates during integrity checks. An improvement in the In cold welding procedure is reducing In overflow onto the alumina during compression, which would improve its voltage hold off ability. Improvements in the AuSi soldering procedure include creating a fixture to align the diamond on the alumina during soldering, masking off the alumina during soldering to eliminate the risk of Au evaporation adhering to the alumina, and further research into using GaN as a photocathode and improving its QE. An increased number of complete capsules should be fabricated in order to isolate potential problems and create a more fluid fabrication procedure. In particular, a focus should be on increased fabrication of gain measurement AuSi soldered capsules.

6.3 Future Research

Future research will be focused in making an emission measurement of the capsule and calculate the gain, which was briefly described in Section 5.2.2. Since a focus will be placed towards fabricating more gain measurement AuSi soldered capsules, improvements in the fabrication procedure will, in turn, improve the measured gain. Improving the gain can be accomplished by reducing contamination of the hydrogenated diamond surface, photocathode, and alumina.

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