EML 4905 Senior Design Project

A.B.S THESIS

PREPARED IN PARTIAL FULFILLMENT OF THE

REQUIREMENT FOR THE DEGREE OF

BACHELOR OF SCIENCE

IN

MECHANICAL ENGINEERING

HIGH TEMPERATURE VACUUM SYSTEM

25% Final Report

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This B.S. thesis is written in partial fulfillment of the requirements in EML 4905. The contents represent the opinion of the authors and not the Department of Mechanical and Materials Engineering.

Ethics Statement and Signatures

The work submitted in this B.S. thesis is solely prepared by a team consisting of DARIESKY LINARES, and CHRISTOPHER SEQUERA and it is original. Excerpts from others' work have been clearly identified, their work acknowledged within the text and listed in the list of references. All of the engineering drawings, computer programs, formulations, design work, prototype development and testing reported in this document are also original and prepared by the same team of students.

Dariesky Linares	Christopher Sequera
Team Leader	Team Member

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Faculty Advisor

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Abstract

About two years ago, The Boston Retinal Implant Project and it associate came to Florida International University with a very interesting project: to develop the process for assembling the bio-compatible materials used for the high-density retinal prosthesis in the Retinal Project. This senior design project specifically focuses on the process of brazing the ceramic feed-through disc to the titanium ring that will be placed into the subretinal space of the eye. The assembly composed of 96% alumina ceramic and titanium metal used pure gold (99.99%) as a filler material in the brazing process. These materials were pre-determined and justifiable as they are biocompatible. The melting temperature of gold is 1065 ^{o}C, which is below the melting point of titanium. However, under high temperatures, titanium is highly susceptible to oxidation. Therefore, the entire brazing process had to be done in high vacuum to ensure the integrity of the titanium. Also, titanium changes from the Alpha phase to the Beta phase at around 850 ^{o}C if left at that temperature for too long. Thus, the process also was completed in less the 15 seconds in order to achieve desirable results. Given the two main constraints above, a two zone furnace was designed in order to heat the parts to two different temperatures utilizing heat radiation from Tungsten wire channeled through the inner surface of the furnace. The first zone heats the parts to 750 ^{o}C, which is safe enough to prevent the phase change in titanium. The second zone heats the parts to 1200 ^{o}C to archive the actual brazing process. Both zones were in high vacuum of approximately 10^{-6}Pa. The two zone design was beneficial by the following: the design gave the operator better control of the materials' temperatures, the time exposed to those temperatures, and a means of profiling the material. The two zones are separated by two Zrconia insulation cylinders. Tungsten wire was vertically looped around the inside of the cylindrical insulation and powered from a power supply to produce the radiation necessary to achieve 750 ^{o}C and 1200 ^{o}C for the bottom and top zones, respectively. A linear mechanism was also designed to move the product

from the first zone to the second zone through by means of levers and gears. To contain the system, a number of heat shields were also designed around the system to: (1) reflect any radiation back inwards toward the process to increase the heat transfer rates, allow any person to safely observe the process from a close distance, and to protect vacuum system itself from the exposed high temperatures. The existing high vacuum system in the AMERI lab was used for the above all the pre-described processes.

Introduction

Problem Statement

In a previous project, a successful attempt in vacuum brazing titanium to 96% alumina ceramic using pure gold as the filler metal was achieved by “resistance heating of C.P titanium inside a thermal evaporator using a Ta heating electrode.” However, due to significant changes in the original geometry of titanium ferrule, the process no longer achieves the desired temperatures and heat transfer rates necessary to braze the two materials. The original design of the electrode was optimized based on several ANSYS resistive heating simulations, which, in turn, is based on the materials, geometry, environment, tolerances, mesh size and so on. Although effective in intent, even the slightest deviation from the calculations or experimental data, leads to undesirable results. Thus, when the titanium ferrule was changed to include a small step on the bottom portion of the disk, the heat generated from the electrodes to the titanium could no longer melt the gold filter metal. This team proposes to continue the project by completely re-designing the heating process, furnace, vacuum mechanisms, and control system.

Just as in the previous project, similar problems arise such as the “poor wetting tendency of ceramic by liquid braze alloys,” the mismatch between the coefficient of thermal expansion of each material leading to stress build up at the surface, “interactions between the braze and the metals,” “the high melting point of gold which is above the allotropic transformation of Ti,” and the titanium and gold reactions forming “a series of intermetallic compounds (IMCs),” which are brittle and detrimental to overall performance. Furthermore, the most cumbersome problem arises from performing the process entire 15 second process in high vacuum. As Ti is very reactive a metal to air, nitrogen or hydrogen, the brazing must be performed in vacuum of the order of 5×10^{-6} Pascal inside a thermal evaporator. This environment does not allow for any type of convection; leaving only black body radiation or conduction as possible heat transfer methods.

Motivation

The Boston Retinal Implant Project is an organization dedicated to research and development of new technologies to help blind patients. As part of the development of this new technology Florida International University was asked to develop a procedure to braze alumina ceramic to titanium metal using gold as the filler material. This project was started by Mr. Mohammad S. Siddiqui, while working on his Doctor of Philosophy Degree in FIU; Mr. Siddiqui was able to successfully braze the two materials but the results were not consistent though-out a number of tries. The motivation is to be able to create this procedure and develop all the required components to archive the brazing with constant result and held Florida international University compromise in this important and groundbreaking project that will bring joy to many disabled Americans and people around the world.

Literature Survey

Alumina Ceramic to Titanium Joining

Joining Alumina ceramic (Al_2O_3) to titanium (Ti) metal present a different sets of challenges that come from joining a ceramic to a metal material. Joining of metal and ceramic material is often used in designs, as high performance ceramic are sometimes use like metals. This kind of joints is used in many fields that include, airspace, electronic packaging, sensors, and biomedical. Many methods are used to archive the joint of metal and ceramic. Some of these methods include chemical, ultrasonic, microwave, brazing and others. The methods may change based on the properties of the materials being joint and the application of the product

Brazing

Brazing is a technique to joint reversal materials together that are close fitted to each other by capillarity. For this process, we require two different materials: the filler material and the base material. The base material is the one that is going to be jointed with another material and the filler material would as the glue holding the assembly together. To archive brazing some special characteristic are require from the two materials. The filler material has to have a lower melting temperature than that of the base material in order to archive brazing. This procedure is done by heating the filler material to the melting point and let the liquid metal flow on the base material, then joints the pieces together and let it cool to archive brazing, to successfully archive brazing all surfaces have to be clean and free of any contaminant that might be present on the base material or the filler material. Brazing and welding are somewhat similar process where a filler material is use to joint two or more material together, the difference is that brazing is done at a more height temperature. Figure 1 shows the schematics of brazing process:

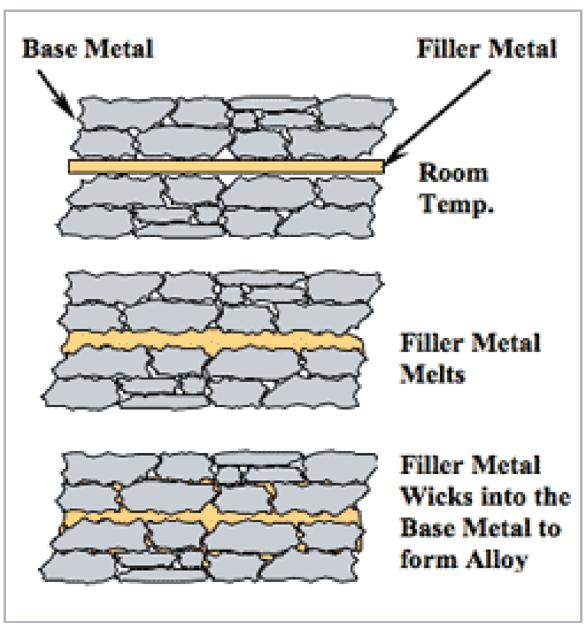

FIGURE 1 BRAZING SCHEMATIC [3]

Radiation Heat Transfer

The study of heat transfer is concerned with two main components is temperature and the flow of heat. Temperature signifies the quantity of thermal energy available. On the other hand, heat flow signifies the movement of thermal energy. Thermal energy stems from the kinetic energy of excited molecules setting a chain reaction with other molecules. The higher the kinetic energy of the material, the greater the temperature will be. Kinetic energy passes this energy to regions with less motion. Therefore, heat flows from higher to lower temperatures. Heat transfer between areas can be organized into three main categories - conduction, convection, and radiation. In conduction, collisions between atoms and molecules in the material transfer kinetic energy between atoms leading to increases in temperature. Different materials have different conduction rates measured in thermal conductivity. Convection is the concerted, collective movement of ensembles of molecules within a medium.

Project Objective

The objective is to design and manufacture a high temperature furnace to braze alumina ceramic to titanium metal using gold as the filler material. The brazing process has to be archive in a high vacuum environment of 10^{-6} Pascal, because of the material properties of titanium. A change in geometry of the Ti material from the previews design was one of the major factors for the inconsistences of the results of the previews furnace design that has to be corrected. The new design include a "step" in the Ti metal base were the gold sits, Figure 2. This step causes a major difference in the overall heat transfer for the whole system.

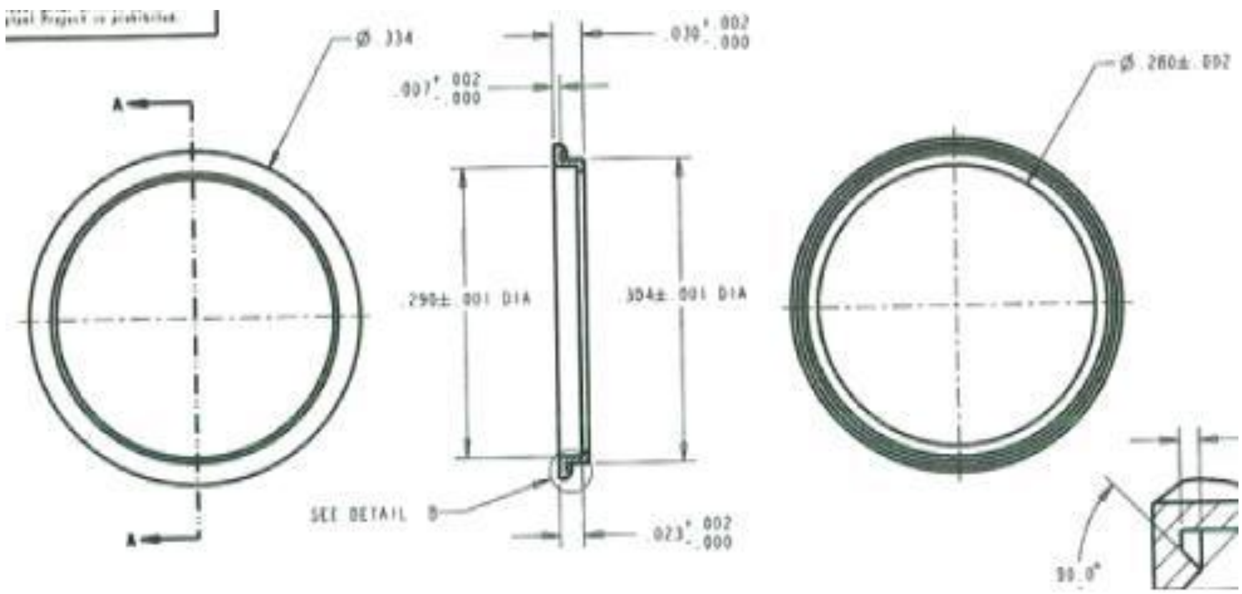

FIGURE 2 TOP VIEW, SECTION VIEW, BOTTOM VIEW OF TITANIUM ELEMENT [4]

Titanium is a reactive material and it has a high oxidation number, for this reason the brazing process has to be done inside high vacuum of 10^{-6} Pascal; also the brazing at these high temperatures causes another problem with the Ti metal. Titanium changes phase from alpha (α), closed pack hexagonal crystal phase, to beta (β), a cubic body center structure, at temperature of 885 ^{o}C; this change in phase creates a time restriction for the brazing of no more than 15 seconds at temperature of

1200 ^{o}C required to brazed gold to the titanium and alumina. Although there are several metal that could be used as stabilizer the used of those are restricted by the nature of the product being use for human implant. And the creation of some alloys may be harmful for the human organism.

Design Alternatives

Conceptual Design (Furnace)

The initial design of the furnace was made using SolidWoks, as shown in Figure 3. The concept for the initial design came from the previews project done by Mr. Siddiqui, this furnace is shown Figure 4. The furnace shown on Figure 3 was made using a .875 in outside diameter, and .125 in wall by 1.25 in tall alumina tube, the furnace was made using 100 microns diameter tungsten wire wrapped together on group of 16 in order to create a thicker profile and have more surface area of tungsten in order to reach the require temperature of 1200 ^{o}C.

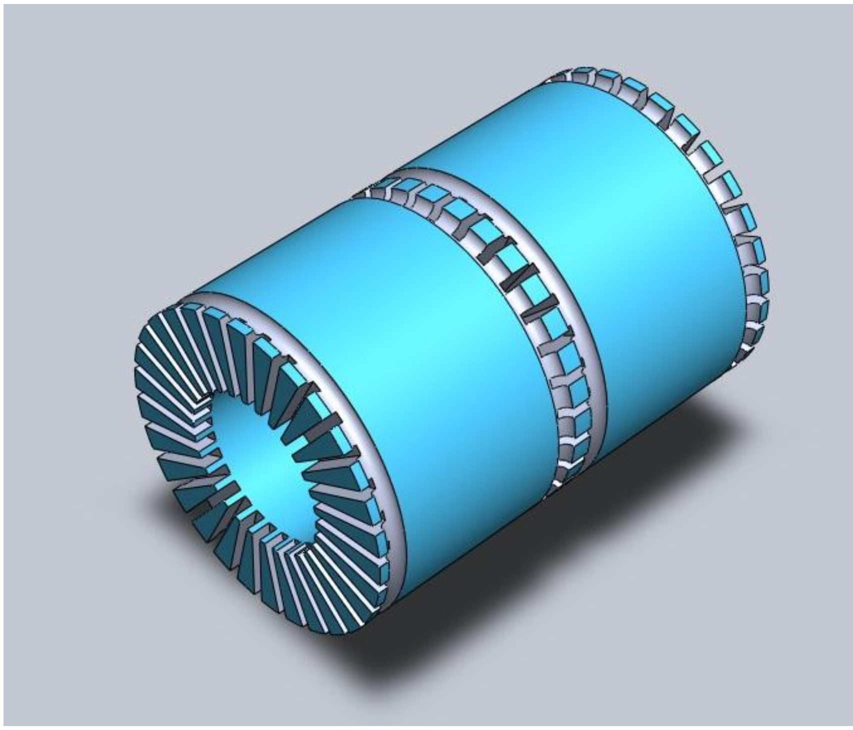

Figure 3 Initial Furnace Design

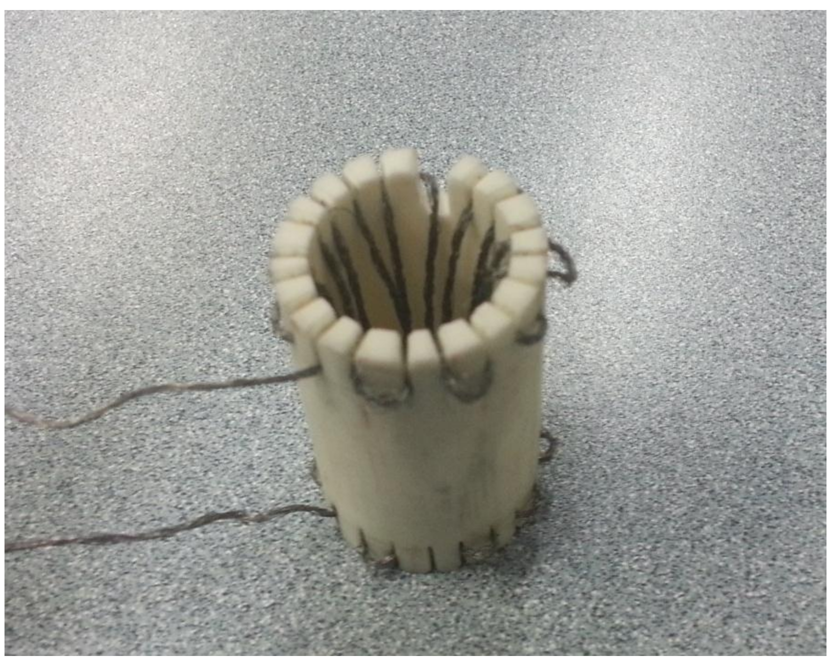

Figure 4 Original Furnace Design

The conceptual designs shown on Figure 3 differ in many forms from the original furnace on many aspects. The first difference between the two design is the material used, the conceptual design 1 was made using Zirconia Cylinders, properties and characteristics of zirconia are shown on Table 1.

Table 1 ZYC Zirconia Cylinders Properties and Characteristics [1]

Bulk Density, g/cc (pcf)	0.48 (30)
Porosity, %	91
Melting Point, °C (°F)	2200 (3992)
Continuous Maximum Use Temperature, °C (°F)	1650(3002)
Intermittent Maximum Use Temperature, °C (°F)	1700(3092)
Flexural Strength, (Parallel to thickness) MPa (psi)	0.55 (81)
Compressive Strength, (Parallel to thickness) @ 10% Compression MPa (psi)	0.21 (31)
Outgassing in Vacuum	Nil
Dilatometric Softening Temperature, °C (°F) at 10 psi	950 (1740)
Thermal Expansion Coefficient (Perpendicular to Thickness) Room Temperature to 1180 °C (2156 °F)	9 x 10-6/°C (5 x10-6/°F)
Linear Shrinkage (Perpendicular to Thickness) , %	
1 hour at 1650°C (3002°F)	2.5
24 hours. at 1650°C (3002°F)	4
Thermal Conductivity, (Parallel to thickness)	
W/mK (BTU/hr ft2 °F/inch) at 400°C (752°F)	0.08 (0.6)
W/mK (BTU/hr ft2 °F/inch) at 800°C (1472°F)	0.11 (0.8)
W/mK (BTU/hr ft2 °F/inch) at 1100°C (2012°F)	0.14 (1.0)
W/mK (BTU/hr ft2 °F/inch) at 1400°C (2552°F)	0.19 (1.3)
W/mK (BTU/hr ft2 °F/inch) at 1650°C (3002°F)	0.23 (1.6)

As shown on Table 1 zirconia is a great thermal insulator which is one of the characteristic needed for the purpose of the project, in this way no energy is wasted in heating the furnace and all the energy is use in heating the sample. Also another essential property need for this project is the ability of the material to be used in vacuum, as if pores materials were used vacuum impossible to archive. Zirconia can be machine using a regular power drilling or a milling machine.

This furnace was created with two different hot zones, one hot zone will reach the 1200 ^{o}C, the require temperature to archive brazing, the other hot zone heats to 750 ^{o}C, this temperature is high enough to begin the process of me brazing but will not cause a phase change from Alpha phase to Beta phase in the titanium material. Also, by creating a two phase furnace, the time of exposure of the titanium at high temperate was cut significantly. Power to the furnace was provided by a power supply with two different controllers, one for each hot zone, this gave more precise control over the temperature of each zone, and the ability to manipulate them separately. The furnace assembly was inserted inside a vacuum system shown in Figure 5 and power was provided via two rods.

FIGURE 5 GLASS DOME VACUUM [AMERI LAB]

Conceptual Design (Heat Shields)

Another key part of the design is the heat shields. Heat shields are used to keep the heat from leaving the atmosphere of the furnace. Shields can work in many different forms, dissipating the heat, reflecting the heat, or absorbing the heat. The most effective shield for the design is the reflecting heat shields.

One of the most common heat shields for vacuum furnaces is the all metal shields. All metal shields consist of several layers of metal surrounding the furnace with a space or gab between them. Figure 6 shows an example of an all metal heat shields for a vacuum furnace. Heat shield can be made out of several metal depending on the working temperature of the furnace, these material could be, tungsten, tantalum, molybdenum, stainless steel, and also some graphite base material as well as some carbon base materials. In many cases the heat shields are composed of different material, this is done to keep the cost down. A widely use combination of material for the heat shields are two or three layers of molybdenum follow by several layers of stainless steel, the molybdenum layers can be change to tungsten or tantalum depending on the working temperature of the furnace.

The process of brazing can take a toll on the heat shields, as shown on Figure 7 the brazing process can deposit residue of material on the shield compromising the radiation heat transfer of the different materials. In time this will caused the total heat deflected by the shields will not be enough to keep an adequate temperature in the outside of the vacuum.

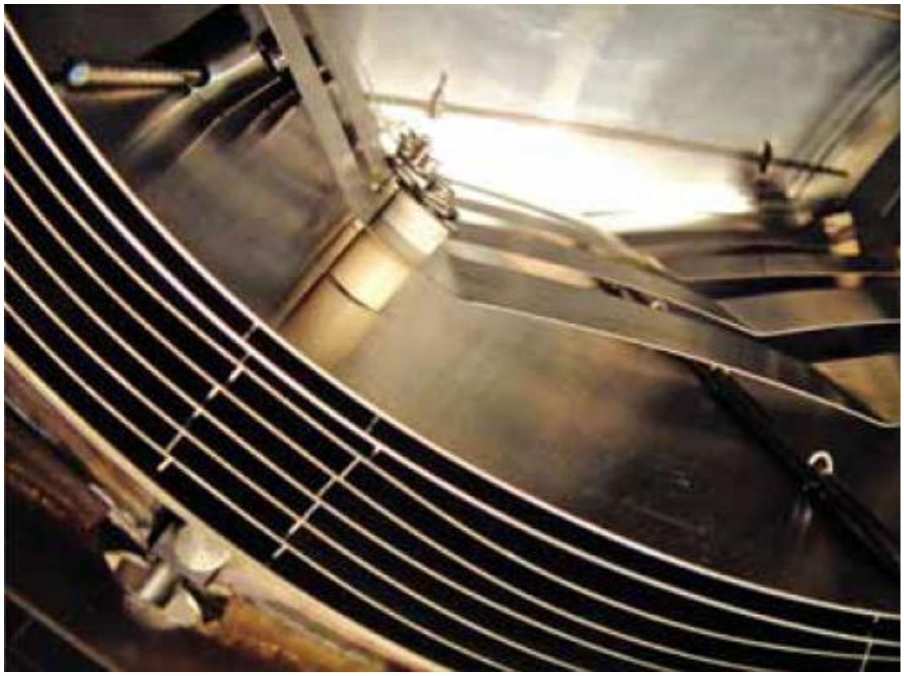

Figure 6 All Metal Heat Shields [5]

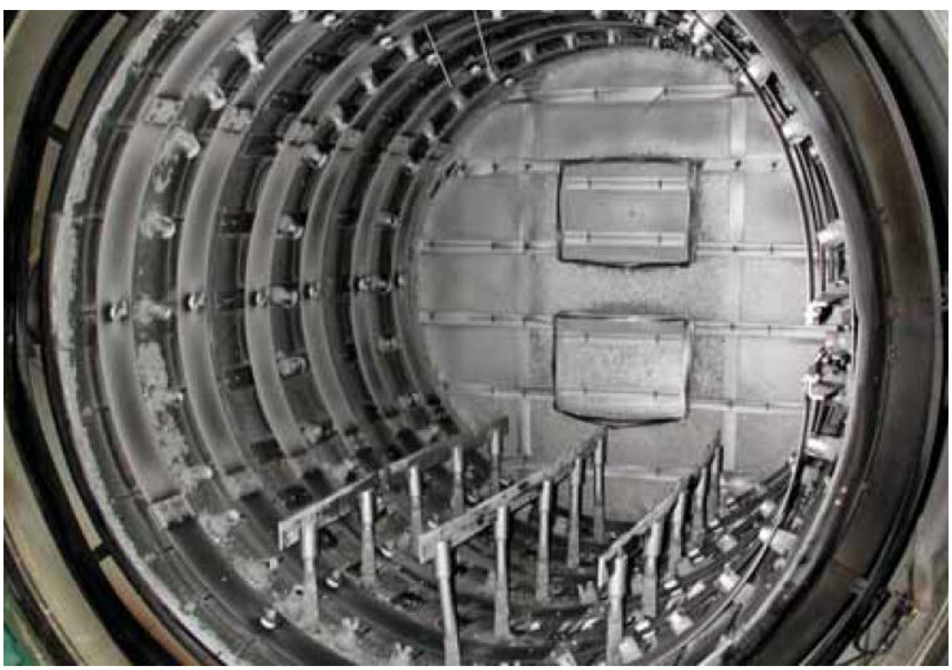

Figure 7 Heat Shields with Brazing Deposits [5]

Project Management

Timeline

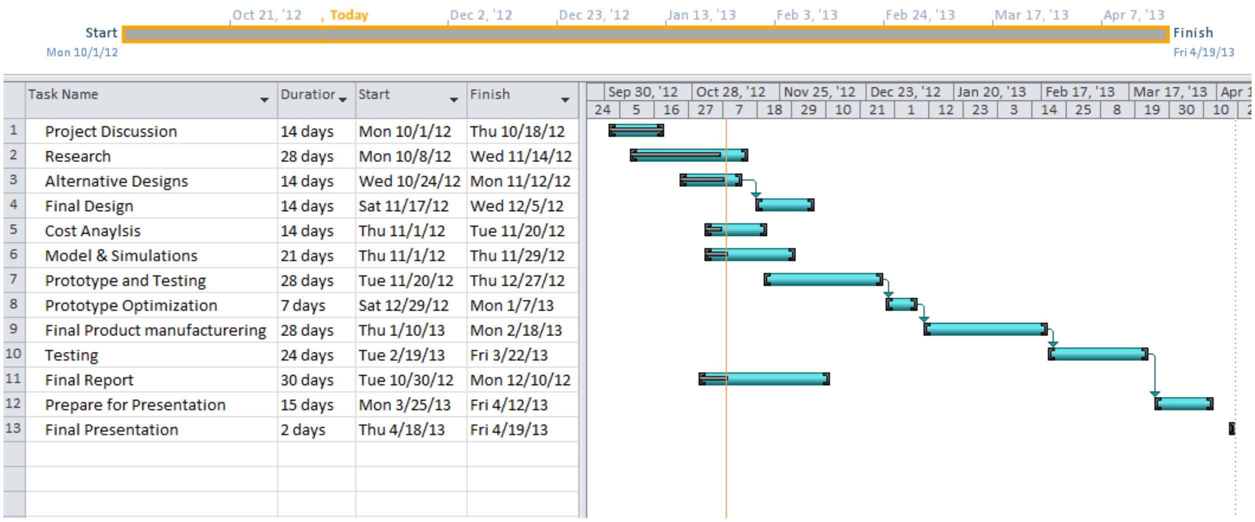

FIGURE 8 TIMELINE

Figure 8 shows a Gantt chart for the project, this table helps keep track of the project and give an idea of the work to be done. As shown on the chart research on the project was the first objective, and this was done by the entire team. A more accurate description of the time spend on the project is shown on Table 2 and Table 3 Time Spend on Project for Spring Semester

Breakdown of Roles and Responsibility

The responsibility for the project has been divided equally among the two members of the team. Several of the responsibilities had been share by both members while other responsibilities had been assigned to one specific member. The responsibilities had been assigned as follow:

Dariesky Linares: Initial project research, design and calculation for metal heat shields, manufacturing of heat shields, and helped in the manufacturing of furnace. Dariesky made SolidWoks and ANSYS computer models and ran heat transfer computer simulation for furnace and shields. He was also in charge of the linear mechanism to move the assembly through the furnace and overall project management.

Christopher Sequera: Initial project research, design and calculation of furnace, manufacturing of furnace, and help in the manufacturing of heat shields. Christopher assembled and programed the control systems and also responsible for wiring all thermocouples. He was in charge of the cost analysis and budget management of the project.

Time Spend on Project

TABLE 2 TIME SPEND ON PROJECT ON FALL SEMESTER

Time Spend on Project Fall Semester						
Dariesky Linares				Christopher Sequera		
Date	Hours	Description		Date	Hours	Description
10/7/2012	4.0	Research and Discussion		10/7/2012	4.0	Research and Discussion
10/8/2012	4.0	Research Boston Retinal Implant Project		10/8/2012	4.0	Research Boston Retinal Implant Project
10/13/2012	2.0	Meet with adviser		10/13/2012	2.0	Meet with adviser
10/15/2012	4.0	Research on materials		10/15/2012	4.0	Research on materials
10/20/2012	2.0	Meet with adviser		10/20/2012	2.0	Meet with adviser
10/22/2012	5.0	Research on new concept		10/22/2012	5.0	Research on new concept
10/23/2012	4.0	Poster soft copy		10/23/2012	4.0	Poster soft copy
10/25/2012	3.0	Meet with adviser		10/25/2012	3.0	Meet with adviser
10/29/2012	5.0	10% Report		10/29/2012	5.0	10% Report
10/30/2012	5.0	10% Report		10/31/2012	3.0	10% Report
10/31/2012	4.0	10% Report		11/1/2012	2.0	Meet with adviser
11/1/2012	2.0	Meet with adviser		11/2/2012	2.0	Furnace Calc.
11/2/2012	2.0	Heat Shields		11/3/2012	8.0	Shield and Power Cal.
11/3/2012	7.0	Shield and Power Cal.		11/5/2012	7.0	25% Report
11/5/2012	6.0	20% Report		11/7/2012	7.0	25% Report
11/7/2012	6.0	20% Report				
Total Hours	65.0			Total Hours	62.0	
Estimated number of Hours Required	120.0			Estimated number of Hours Required	120.0	

Total Hours Spend During Fall Semester	127.0

TABLE 3 TIME SPEND ON PROJECT FOR SPRING SEMESTER

Time Spend on Project Spring Semester						
Dariesky Linares				Christopher Sequera		
Date	Hours	Description		Date	Hours	Description
Total Hours	0.0			Total Hours	0.0	
Estimated number of Hours Required	300.0			Estimated number of Hours Required	300.0	
Total Hours Spend During Fall Semester						0.0

Analytical Analysis

The analytical analysis for the project requires a thorough understanding of radiation heat transfer as this is the only type of heat transfer found in vacuum systems.

In a real surface, the heat transfer depends on the spectral components of the irradiation, which can be reflected, absorbed, and/or transmitted.

EQUATION 1 IRRADIATION OF REAL SURFACE [6]

$$G_\lambda = G_{\lambda,ref} + G_{\lambda,abs} + G_{\lambda,tr}$$

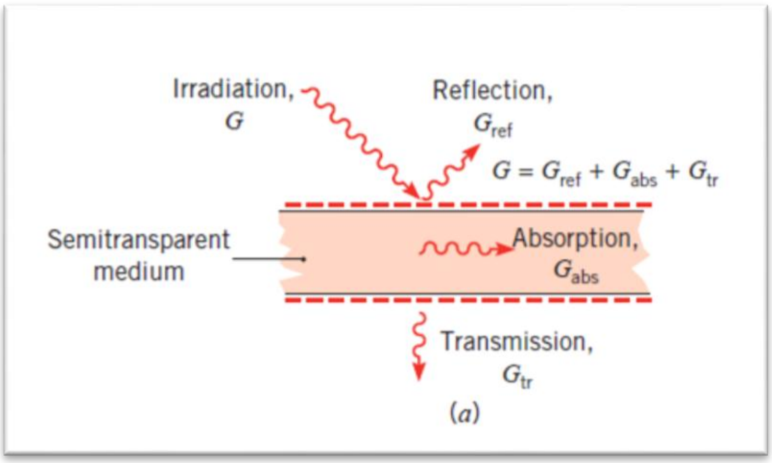

FIGURE 9 REFLECTION, ABSORPTION, AND TRANSMISSION OF IRRADIATION FOR A SEMITRANSPARENT MEDIUM [6]

The determination of these components are complex because of their dependence on the upper and lower surface conditions, the wavelength, temperature, volumetric effects, and the composition and thickness of the medium the radiation is penetration. Although, in our case, the heat transfer rates are in vacuum, the complexity comes from the rapid temperature changes across the materials, different material properties, varies surface finishes, and complex surface geometries between the titanium disk, tungsten wire, cylindrical insulation, and heating shields.

It is well known that the spectral irradiations for reflected, absorbed, and transmitted radiations can be obtained by finding the absorptivity, reflectivity, and transmissivity of the material, respectively. These properties are determined by the fraction of the irradiation reflected, absorbed, and/or transmitted by the surface. The complication comes from the fact that it is characterized by the directional, spectral dependence and, in our case, possible surface temperature dependence. The following equation is the general equation for obtain just the average spectral, hemispherical absorptivity,

EQUATION 2 SPECTRAL SPHERICAL ABSORPTIVITY [6]

$$\alpha_\lambda(\lambda) = \frac{\iint \alpha_{\lambda,\theta}(\lambda,\theta,\phi)\, cos\theta\ sin\theta\ \ d\theta d\phi}{\iint\ I_{\lambda,i}(\lambda,\theta,\phi)\, cos\theta\ sin\,\theta\ d\theta d\phi}$$

Where,

EQUATION 3 SPECTRAL DIRECTIONAL ABSORPTIVITY [6]

$$\alpha_{\lambda,\theta}(\lambda,\theta,\phi) = \frac{I_{\lambda,i,abs}(\lambda,\theta,\phi,T)}{I_{\lambda,i}(\lambda,\theta,\phi,T)}$$

The reflectivity and transmissivity follow a similar equation as the ones above.

In order to start the required calculations for the heat shields, some assumptions were necessary. The first assumption was to treat the furnace and the heat shields as diffuse gray surfaces; the second assumption was to assume the furnace and shields behave as long concentric cylinders. The gray surface assumption implies that the emissivity, ε, and the absorptivity, α, are independent of the wavelength over the spectral regions of our particular irradiation and surface emission. Gray surfaces can be assumed for surfaces under the condition of Figure 10:

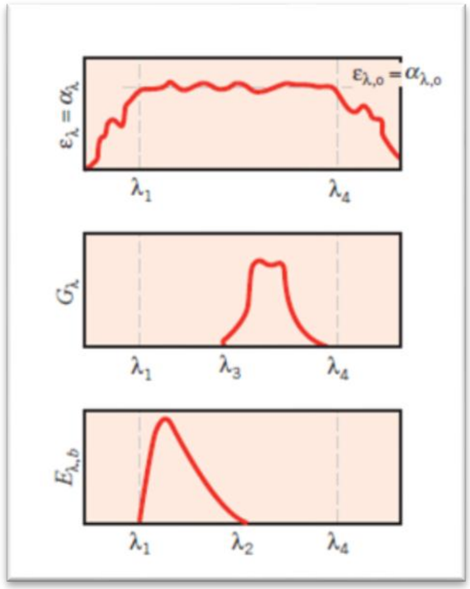

FIGURE 10 CONDITIONS FOR GRAY SURFACE RADIATION [6]

The assumption of gray surfaces greatly simplifies radiation calculations. This allows the total, hemispherical emissivity of the surface is equal to its total, hemispherical absorptivity.

Taking into consideration these assumptions we can obtain Equation 4 for one shield.

EQUATION 4 LONG (INFINITE) CONCENTRIC CYLINDERS [6]

$$q_{12} = \frac{\sigma A_1 (T_1^4 - T_2^4)}{\frac{1}{\varepsilon_1} + \frac{1 - \varepsilon_2}{\varepsilon_2} (\frac{r_1}{r_2})}$$

Cost and Budget

Overall Cost Analysis

The cost depends on a number of variables including materials, engineering labor, building labor, testing, and; with materials having the largest factor. When deciding on the material, the different cost factors taken in consideration include: the cost per unit of the material, the ease of obtaining these materials, the simplicity of manufacturing, the process these materials most undergo, the quality control of these materials, and the tolerances of the material.

Materials were only brought from reliable manufacturing companies with highly experienced staff to ensure any materials brought were dependable, warrantied, and any questions on the materials could be answered from the manufacturer. In Figure 11 is an example invoice of thin sheets of Tungsten and Molybdenum foil, obtain for the purpose of building the heat shields

TABLE 4 COST ANALYSIS

Materials	Diameter		Size			Weight	Price
	ID (in)	OD (in)	Thickness (in)	Width(in)	Height (in)		
Tungsten	N/A	N/A	0.005	12.5	24		$588.70
	N/A	N/A	0.003	6.5	24		$226.05
Molybdenum	N/A	N/A	0.004	12	24		$225.00
Zirconia	1	2	N/A	N/A	6		$225.00

Thermo Shield **Quote**

361 Grove Dr.
Portola Valley, CA 94028

Phone #	650-851-1859	sales@thermoshield.us
Fax #	650-851-2049	www.thermoshld.com

Date	Quote #
11/5/2012	7603

Name
Florida Int'l. Univ. Dariesky Linares 786.208.1996 dlina003@fiu.edu

Terms	FOB	Contact
Advise	Portola Vlly	mike

Item	Description	Qty	Price	U/M	Total
33001-0.004	Moly. Foil 0.004 thk x 12.00 x 24.00	1	225.00	ea	225.00T
88005-0.005	Tungsten Sheet 0.005 thk x 12.50 x 24.00	1	598.70	ea	598.70T
88001-0.003	Tungsten Foil 0.003 thk x 6.50 x 24.00	1	226.05	ea	226.05T
	All quoted items in stock and can cut/ship within a day of order. Thanks.				

FIGURE 11 METAL FOIL QUOTE

Major Components

The major component in this project is the high vacuum system. In order for the brazing process to become successfully, the entire system must be in at 10^{-8} Torr or less in to inhabit Titanium oxidation on the wetted surface. For this purpose, a high vacuum system must be designed carefully. The different parts of a high vacuum system include the roughing pump, the diffusion pump, and the cold trap (See

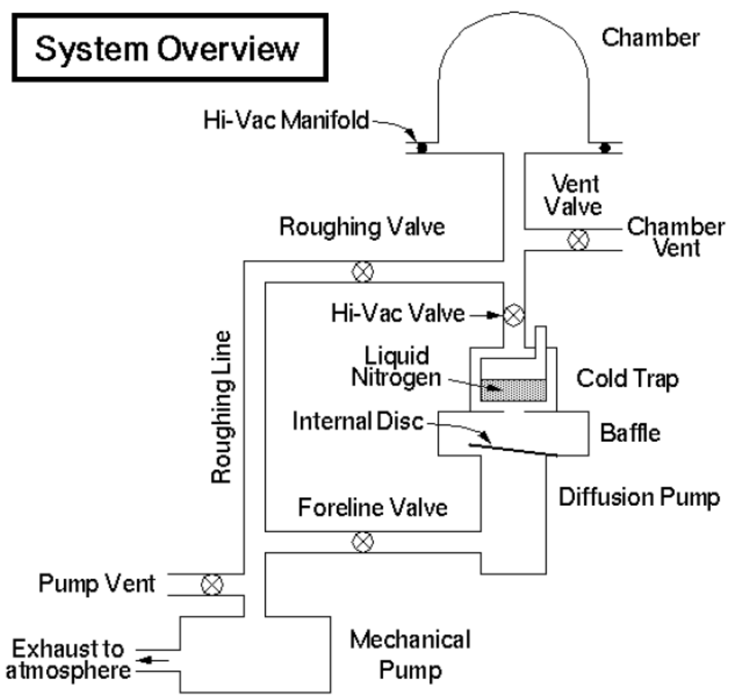

Figure 12.)

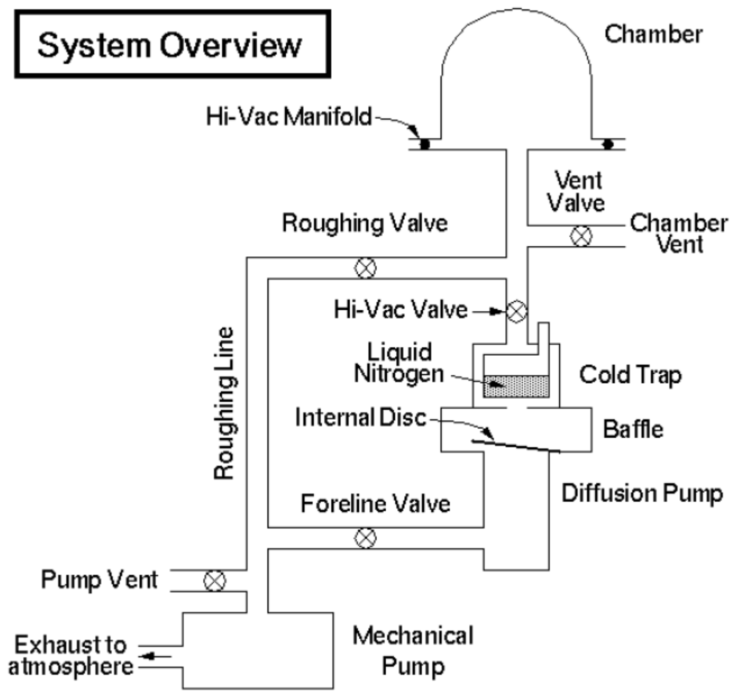

FIGURE 12 SYSTEM OVERVIEW [7]

With the roughing valve open, the mechanical pump is first turned on to remove the initial large capacity of atmosphere air in the vacuum chamber. The pump sometimes also serves as the backing pump, where the same pump is used to “back-up” the diffuse pump as the figure below shows. Once the chamber has been evacuated, the roughing valve is closed and diffusion pump is turned on with the fore line and Hi-Vac valve open. The vacuum chamber can now achieve levels of about 10^{-7} Torr with this arrangement. With the mechanical pump maintaining exhaust, fasting pumping can be achieved through the high speed jets of oil vapor, which help with compassing and directing the air into the mechanical pump. The oil pool at the bottom of the pump is heated, causing oil vapor to be forced up the jet stack.

Conclusions

The experience and knowledge obtained during the course of this project was substantial. The impact of our work was an important piece to the completion of the Boston Retinal Implant Project and its associates. Our work will be used as a reference for future development and a base for large scale manufacturing for mass production. The work presented will give future engineers a platform when it comes to brazing biomedical materials, specifically titanium to alumina. Due to the limited type of materials that can be used in the body without corrosion, this research is vital. All techniques used are repeatable with satisfactory results. The work also sheds light on the vacuum process and its challenges; from the design to the temperatures control system. Challenges such as no convection loads for cooling, using none porous material, maintaining the integrity of the system, staging the vacuum process correctly, and maintaining a high vacuum pressure throughout the brazing process.

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