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in

"Surface Treatments for Industrial Applications"

PROTOCOL FOR THE PREPARATION OF ALUMINA/METAL BRAZED JOINTS.

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ABSTRACT

Brazing is the joining of materials through the use of heat and a filler metal with a melting temperature above 450°C but below the melting point of the metals being joined. This technique joins parts by creating a metallurgical bond between the filler metal and the surfaces of the two materials joined.

There are several factors which have direct influence on the final quality of a brazed piece: the filler metal selection, the heating technique, the atmosphere in which the process is performed, the cleaning procedure, the assembly of pieces, etc.

In metal-ceramic junctions, ceramic surfaces, especially oxides, are very stable chemically, consequently, molten metals do not wet them well. There are several techniques that allow to overcome the chemical challenge.

In order to develop a procedure that ensures the conditions to achieve a successful brazing process, several techniques for metal-ceramic brazing in a controlled atmosphere (vacuum) were tested. Brazing using active filler metals and direct brazing using ceramic metallization by sputtering were studied in this work.

Alumina (Al_2O_3) was chosen as the ceramic part, whereas stainless steel 304 and Inconel were chosen as metallic components, cylindrical samples were assembled in lap configuration and were heated by IR lamps.

The pieces were cut and the cross-section was studied by Scanning Electron Microscope SEM and energy-dispersive X-ray spectroscopy (EDS, EDX) searching for evidence of filler metal uniform spread and diffusion.

The samples brazed using Pallabraze 850 disc of 0.01 mm of thickness for braze Inconel and Metallized alumina with 1μm of Titanium and using Argon atmosphere showed the more uniform distribution and diffusion.

To my mother Yajaira and my sisters and nephew Maria, Claudia and Alessandro. Family is always the most important thing in life.

To make you proud.

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1. INTRODUCTION

Over the years there has been an increase on the request of highly reliable products to be used in extremely demanding conditions, in environments which require performances that no single material can offer by itself. The assembly of two materials which can afford the mechanical, thermal and chemical requirement could be a solution.

The selection of materials to be joined was made in function of its application. To obtain the best performance in a corrosive environment at high temperature, the assembly of metal-ceramic is usually considered the best option.

Brazing is the joining of materials through the use of heat and a filler metal with a melting temperature above 450°C but below the melting point of the metals being joined. Brazing is a very versatile method of material joining. This technique joins parts by creating a metallurgical bond between the filler metal and the surfaces of the two materials joined.

There are several factors which have direct influence in the final quality of a brazed piece, the filler metal selection, the heating technique, the atmosphere in which is performed, the cleaning procedure, the assembly of pieces, etc.

In the case of the metal-ceramic junctions, ceramic surfaces, especially oxides, are very stable chemically. Consequently, molten metals do not wet them well because the energy of the molten metal-ceramic interface is greater than the surface energy of the bare ceramic surface. There are several techniques that allow overcome the chemical challenge, use reactive brazing alloys and metallized the ceramic are the most commonly used. Beside there is a mechanical challenge and is overcoming residual stresses caused by the physical property mismatch between the materials.

A protocol of preparation for metal-ceramic joints is necessary in order to take into account these factors and develop a procedure that ensures the conditions for achieve a successful brazing process.

This work is focus in the study of several techniques for metal-ceramic brazing in a controlled atmosphere (vacuum). Active filler metals are used and several methods of ceramic metallization are studied in order to use no-active brazing filler metals.

The **Alumina** (Al_2O_3) is chosen as the ceramic component of the assembly, whereas **stainless steel 304**, the most versatile and commonly used kind of stainless steel, and **INCONEL**, a family of nickel-chromium based super alloys, were chosen as metallic component.

The pieces characterizing are going to be made by scanning electron microscope SEM and energy-dispersive X-ray spectroscopy (EDS, EDX) searching for evidence of filler metal uniform spread and diffusion.

2. THEORETICAL BASES

The following chapter presents an overview of brazing theory and other relevant concept that support this research. Fundamentals of brazing, metal-ceramics brazing, surface treatments, joint design, heating methods are issues that will be discussed in this chapter.

2.1. Fundamental of Brazing

The term brazing comprises a group of welding processes that produce coalescence of materials by heating them to the brazing temperature in the presence of a filler metal having a liquidus above 450 °C (890 °F) and below the solidus of the base material. The brazing filler metal turns liquid and is distributed between the closely fitted faying surfaces of the joint by capillary action then creates an alloy bond with the faying surfaces. Brazed joint, could be strong as or stronger than the base materials, this depends of the design of the joint and the filler metal.

Use brazing technique brings the following advantages:

- Economical fabrication of complex and multicomponent assemblies.
- Simple method to obtain extensive joint area or joint length.
- Excellent stress distribution and heat-transfer properties
- Ability to join nonmetals to metals.
- Ability to join dissimilar metals.
- Capability for precision production tolerance.
- Reproducible and reliable quality-control technique.

Strong, uniform, leak-proof joints can be made rapidly, inexpensively, and even simultaneously. Joints that are inaccessible and parts that may not be joinable at all by other methods often can be joined by brazing.

There are several physical principles which are important for the brazing process. Capillary flow is the dominant physical phenomenon that ensures good brazements when both faying surfaces to be joined are wet by the molten filler metal**.** The capillary action as a result of the relative attraction of the molecules of liquid to each other and those of the solid influence the brazing filler metal flow, the joint must be properly spaced to permit efficient capillary action and coalescence. In actual practice, brazing filler metal flow characteristic are also influenced by dynamic considerations involving fluidity, viscosity, vapor pressure, gravity, and, especially, by the effect of any metallurgical reactions between the filler metal and the base material [1]**.**

The ability of a liquid to maintain contact with a solid surface is called **wetting**, this is the result of intermolecular interactions between them (solid and liquid). **Wettability** describes the trend of a solid to be in contact with a liquid rather than another. A drop of a wetting fluid has a tendency to **spread** over the surface, increasing its contact area; on the other hand, a non-wetting fluid has a tendency to compress itself, decreasing the contact area.

The phenomena of wetting and spreading are very important to the formation of brazed joint, a good wettability condition guarantees that liquid brazing filler metal is going to adhere to the surface of the material in solid state and, when cooled below its solidus temperature, to make a strong bond with this material. Wetting is a function not only of the nature of the filler metal, but the degree of interaction between materials to be joined. There is considerable evidence that in order to wet well, a molten metal must be capable of dissolving or alloying with, some of the metals through which flows [2]. Another factor that affect wetting is the cleanliness of the surfaces to be wetted. Oxide layers inhibit wetting and spreading, as do grease dirt, and other contaminants, that prevent good contact between the brazing filler metal and the base materials.

A very simple way to describe the diffusion is the transport of mass by atomic motion, this phenomenon occurs when a system is not in equilibrium, diffusion has two primary properties it is random in nature, and transport is from region of high concentration to low concentration.

Base material and filler metal interact through diffusion, the atoms of the filler metal in liquid state diffuses into the base material forming diffusion-bonds. Once formed, diffusion-bonded joints are stable to high temperatures so that the service temperature of the assembly can actually exceed the peak temperature of the joining process without risk of the joint remelting. [3]

The rate of diffusion is proportional to the temperature. At brazing temperatures, the possibility of diffusion is very high. Diffusion of the filler metal into the base metal should be at a minimum for the best joint quality, and diffusion can be minimized by minimizing the heat input at brazing temperature [4].

In a properly designed brazing process, an oxide-grease-free surface promote wetting and spreading of the filler metal, with the suitable joint design, the molten filler metal is drawn completely through the joint area without any voids or gap, at the right temperature and time, the diffusion occurs, and the filler metal diffuse into the base material forming bond which will keep the junction.

2.1.1. Base-Material characteristics:

The base metal has an important effect on joint strength; a high-strength base metal produces joints of greater strength than those made with softer base metals, also the larger number of complex metallurgical reactions in hardness metals can cause changes in the base-metal hardenability and residual stress, therefore the joint strength become less predictable.

The coefficient of thermal expansion (CTE's) is another important parameter because differences on CTE's generate the residual stress that may produce distortion, depending on the joint design and can affect the strength of the joint.

There are several metallurgical phenomena that influence the behavior of brazed joints. Among these base-metal effect are: alloying, carbide precipitation, stress cracking, hydrogen and oxide stability.

The brazing cycle by itself can affect the properties of the base-metal, if the brazing-process temperature and time are in the annealing range of a fortified by cold work alloy, this will be annealed and the joint strength reduced.

One of the advantages of brazing process is the possibility of joint dissimilar materials; some applications involve the junction of a metal and a ceramic. These inorganic and non-metal materials have very different properties from metals, normally, higher melting temperature, lower thermal expansion coefficient. The oxide in its surface avoids the wetting by most of fillers metals. In this case the designers have to select the right filler metal, the right method and consider the difference of the properties between the materials in order to avoid the residual stress.

2.1.2. Filler-Metal characteristics:

Most of the brazing filler are alloys which melt in a range of temperature and depending of the base-metal properties can react or not during the brazing process. Several metal-filler base materials are describes in table N°2.1.

Base Material	Nickel (Ni)	Cobalt (Co)	Silver (Ag)	Gold (Au)	Aluminum (Al)	Copper (Cu)
ASTM Designation	BNi	BCo	BAg	BAu	BAlSi	BCu
Braze Range (°C)	927-1205	1175-1245	620–980	890-1230	570–620	705–1150
Max Service Temperature (°C)	980	1040	370	800	150	370
Applications	Alloy steels Carbon Steels Copper Alloys Stainless Steel Nickel-Cobalt alloys	Cobalt alloys	Alloy steels Carbon steels Cast iron Copper alloys Nickel alloys Stainless steel Tool steels	Alloy steels Carbon steels Cooper alloys Nickel-cobalt alloys Stainless steels Tool steels	Aluminum alloys	Alloy steels Carbon steels Cast iron Cooper alloys Nickel alloys Stainless steels Tool steels

Table 2.1. Meta-Filler base materials for brazing.

A specific filler metal cannot be chosen to produce a specific joint strength. Actually, brazing can provide strong joints with almost any good commercial filler metal if the brazing methods and joint design are selected and applied correctly.

Several characteristics desirable in a filler-metal are:

- Proper fluidity at the brazing temperature to ensure flow by capillarity action and provide full alloy distribution. A high liquid surface tension, low contact angle, and low viscosity are desirable for promoting filler-metal flow.
- Ability to alloy or combine with parent metal to form an alloy with higher melting temperature.
- Stability to avoid premature release of low-melting-point elements for filler metals at brazing temperature.
- Ability to wet the base-metal joint surface, a low contact angle, which implies wetting, is necessary but is not a sufficient condition for flow, viscosity is also important.

The joint strength depends on many factor as joint design, state of stress, brazing temperature, amount of filler metal applied, location and method of application, heating rate, holding time at the peak temperature, and many other.

Diffusion is an essential part of the metallurgical process, through diffusion the filler metals penetrate and alloy with base metals during brazing; it is generally good practice to select a filler metal that diffuses readily and alloys with base metal.

The melting method used and working temperature in brazing is related directly to the strength of the brazed-joint. Filler metals in which the solidus and liquidus are close together do not usually exhibit a strong tendency to separate and they are relatively fluid, figure N° 2.1 presents typical brazing temperature ranges for various filler metals.

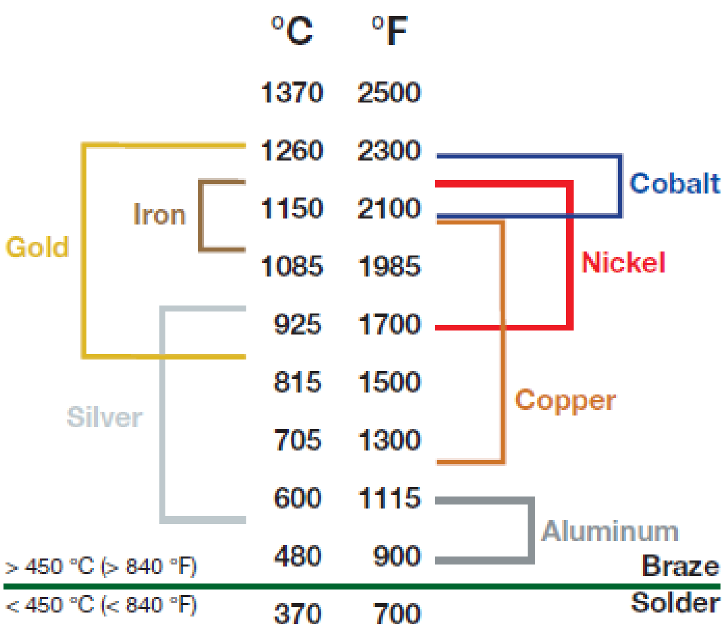


Figure 2.1. Typical brazing temperature ranges for various filler metals.

Depending on the application other important filler-metal properties are: corrosion resistance, such as oxidation and galvanic corrosion with other part of the assembly and service environment, color match to base metal, electrical and thermal conductivity, hardness and machinability, ductility, etc.

The filler-metal must be able to form a strong and permanent bond with the base materials, but this can be difficult to achieve when oxides are present in the brazing joint. Brazing ceramics, such as alumina, are examples of this king of process, the amount of oxide in Alumina make difficult wetting over its surface; therefore the filler-metal does not diffuse correctly.

If the base materials are not wetted by “conventional” brazed, a reactive metal (usually Titanium) is added to metal-filler, the addition of reactive metal to several braze alloy compositions results in increased reactivity and considerable improvement in wetting behavior.

In ceramic brazing case, the reactive metal (titanium) act as strong “getter” of oxygen, that means joining process will depend very much on the ability of titanium in the filler to react at brazing temperature with these oxides that are present in the ceramic structure.

Since reactive metal (titanium) will react with any oxygen that it can, an early “reaction” of reactive metal, such as with free oxygen or water-vapor in the furnace atmosphere, or with metal-oxides on the metal surfaces during heat-up, would result in a considerable reduction of joining/bonding of ceramic-to-metal. To keep the reactive metal addition as "active" as possible during this high-temperature joining process, it is wise to use a good vacuum brazing furnace to exclude as much oxygen as possible from the furnace throughout the entire brazing cycle. Vacuum levels on the order of 10-5 to 10-6 Torr are often used, additionally; the vacuum furnace should be very clean, with no significant contamination on the inner cold-walls of the chamber.

2.1.3. Desires joint properties:

The main desirable features of a brazing joint are:

Shear Strength: The ability to resist the angular deformation, calculated as the sideways displacement of two adjacent planes divided by the distance between them.

Butt Tensile Strength: The ability to resist a force applied perpendicular to a given plane without rupturing.

Stress Rupture: A fracture caused as a result of repeated physical strain.

Hardness: The ability of a material to resist scratching, abrasion, indentation or machining, as measured by a specifically chosen method or standard.

Corrosion Resistance: The ability of a material to resist attack resulting from environmental, chemical or galvanic action.

Oxidation Resistance: The ability of a material, particularly a metal, to resist reaction with oxygen, which can cause a loss of structural integrity resulting from the formation of undesirable oxide compounds.

Microstructure: The composition and microscopic structure of a material, as studied using metallographic methods.

Joint Configuration: The design and shape of the joint chosen to join members that will meet or exceed structural requirements in service. Types of joint configurations include lap, butt, tee, tubing, tube plate and scarf (see section on Joint Configuration).

2.2.Metal-Ceramic Brazing:

The joining of dissimilar materials is often the only solution to fulfill the complex requirements of high technology applications. Metal-ceramic brazing is particularly useful for fabricating high-reliability devices such as those used in high-voltage applications or requiring hermetically sealed joints.

Most of the time, joining different materials is not an easy task. Atoms, ions, or molecules in materials of different classes are joined together in different ways, and therefore characterized by particular combinations of physical-chemical and mechanical properties.

Joining dissimilar materials implies a property mismatches and structure discontinuities. In metal-to-ceramic brazing, ceramics relies on wetting (spreading) of the ceramic surface by some kind of metal, which is often hindered by the covalent nature of the ceramics. Besides that, the differences in thermal expansion coefficient result in the development of residual stresses which can compromise the integrity and quality of the final piece.

Metal-to-ceramic brazing can be accomplished by several methods, among them the application of metallic layer onto the ceramic surfaces or brazing directly to the unmodified ceramic surface (oxide) using active brazing alloys.

There are several metallization methods, the most used for joining metal to ceramic are Molybdenum-Manganese/nickel plating and Physical-vapor deposition or thin-film method [5].

2.2.1. Molybdenum-Manganese/nickel plating Method:

Also known as moly-manganese (Mo-Mn) metallization, a coating of molybdenum and manganese particles mixed with glass additives and volatile carriers is applied to the ceramic surface to be brazed, after air drying; the coating is heated in a wet hydrogen environment (15°–30°C dew point) at 1450°–1600°C leaving a "glassy" metallic coating 300–500 micro-inches (7.6–12.7 μm) thick. The fired coating is subsequently plated with a 0.001–0.003 in. (25.4–76.2 μm) layer of nickel. The nickel plating is sinter-fired at 850°–950°C in a dry hydrogen (–50°C dew point or less) atmosphere leaving a finished metallic surface that can be readily brazed using standard braze filler metals [5].

The figure 2.2 summarizes the process of Mo-Mn metallization:

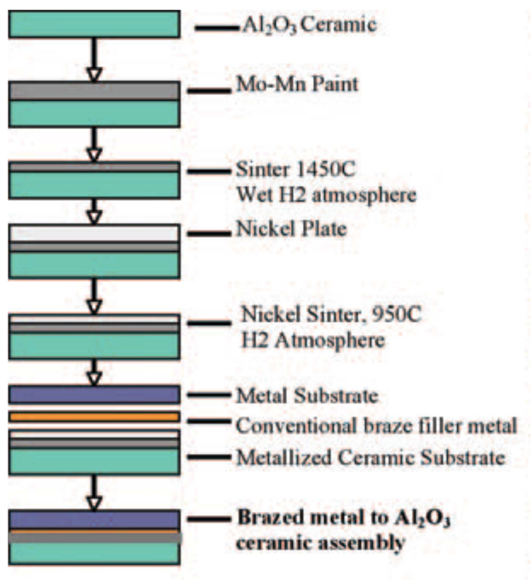


Figure 2.2: Moly-manganese metallization process.

2.2.2. Thin-film Method:

A combination of materials, usually two or three, are deposited onto the nonmetallic surface using a physical vapor deposition (PVD) method, such as evaporation or sputtering. The first layer deposited, often titanium, is typically 0.25-1 μm thick. Other oxygen-getter elements such as hafnium, zirconium, chromium, niobium, etc. may be chosen depending on the application and service temperature. Occasionally, an intermediate layer or layers are deposited to prevent unwanted metallurgical reactions between the initial metal layer and the braze filler metal. The top, or outer, layer is normally a noble metal such as gold, platinum, or palladium that is 0.25–1.0 μm thick. A noble metal is chosen in order to prevent the underlying layer from oxidizing and subsequently preventing proper brazes filler metal wetting and flow [5].

The figure 2.3 summarizes the process of thin-film metallization:

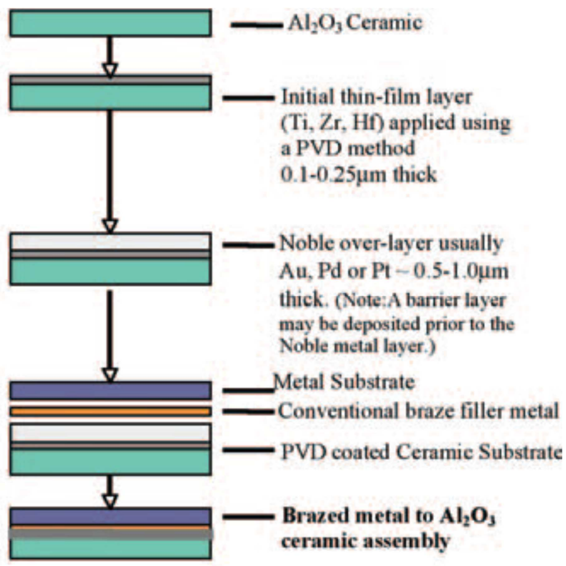


Figure 2.3: Thin-film metallization process.

2.2.3. Active filler-metal brazing:

Active filler metals display good wetting with most ceramic materials. Active filler metal brazing is a metal-ceramic joining method that permits the use of standard brazing techniques when making metal-to-ceramic brazing without the need to apply any metallization to the ceramic substrate, as shown in of Fig. 2.4, the metal and non-metal substrates are cleaned, and the active filler metal is applied between the faying surfaces of the brazements. The brazing operation is usually performed in an inert or ultrahigh vacuum environment, this because excessive oxygen in the atmosphere can react with the active element in the active braze filler metal and compromise joint strength and integrity [6]

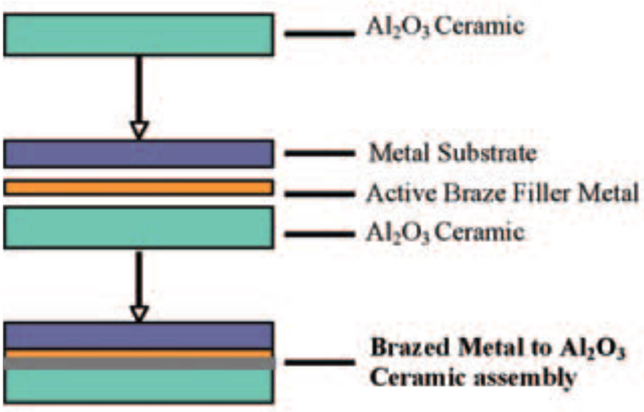


Figure 2.4: Active filler-metal brazing process.

2.3. Pre-brazing considerations:

Many factors can affect the quality of the brazing joint, among these, presence of oxide, grease or dirt, a wide clearance, extensive exposure to high temperature, etc. in order to avoid this factors some considerations have to be done before the process:

2.3.1. Surface Preparation:

In order to obtain uniform quality in brazing process is imperative to have a clean and nearly oxide-free surface, all grease, oil, wax, dirt, and nearly all oxide have to be carefully removed from the base and filler metals before brazing.

It is important to realize that metal surfaces actually consist of a thin layer of oxide crystals formed by reactions with other metals or with oxygen in the air. Only after penetration trough the metal oxide layer the atoms of metal itself are encountered and then the bond could be achieved.

Much of the effort expended in surface preparation is devoted to removing those materials. Moreover, brazing is recommended to be done as soon as possible after the parts have been cleaned [7].

Cleaning is commonly divided into two major categories: chemical and mechanical. Chemical cleaning is the most effective, means the removing all traces of oil or grease. Those vary from simple manual immersion to complex multistage operations. Chemical methods include alkaline cleaning, solvent cleaning, vapor degreasing, and acid pickling. The selection of the chemical cleaning agent depends on the nature of the contaminants, the base metal, the surface condition, and the joint design. Regardless the cleaning agent or the method used, it is important that all residue or surface film are removed from the cleaned parts by adequate rinsing to prevent the formation of other equally undesirable films on the faying surfaces.

Mechanical cleaning may be adequate, in cases that the design must permit it; the mechanical methods most used are dry and wet abrasive blast cleaning. The blasting technique is commonly used for surface preparation; the purpose of blasting is to remove all oxide film and to roughen the surfaces in order to increase the capillary attraction of the filler metal. The blasting material must be clean and must be of a type that does not leave on the surfaces to be joined any deposit.

Another technique in surface preparation is the use of solid and liquid fluxes; those compounds prevent, dissolve or facilitate removing of oxides and other undesirable surface substances.

2.3.2. Joint design and clearance:

The quality and strength of a brazing joint depends on many factors. Among them, the design of a brazed joint, this requires some special considerations, dictated by the nature of the joining process.

Because brazing depends on the principle of capillary attraction for distribution of the molten filler metal; joint clearance is a critical factor affecting the brazing process. Also, the application of the brazed piece has to be considered. It is preferred that any load on a brazed joint is transmitted as shear stress rather than tensile stress. Other factors to be considered are air flux and displacement, the composition and strength of the filler metal and the base metal.

The type and bonding area of the joint are important factors, as well clearance between members. Both of these affect not only the strength of the completed joint but also the ease of brazing [8]. There are basically only two types of brazed joints: butt and lap. All other joints are really only modifications of these two basic types.

The butt joint has the advantage of a single thickness at the joint, preparation is relatively simple, and the joint has sufficient strength for many applications. However, the strength of any joint depends on the bonding area available, and in a butt joint, this area is determined by the thinnest member of the joint. The thinnest member, therefore, dictates the maximum strength of the joint. Some advantages of the butt joint are ease of preparation and single thickness at the joint, which reduces stress concentrations.

The bonding area of a lap joint can be made larger than that of a butt joint. In fact, the area of overlap may be varied so that the joint is as strong as the weaker member, even when a lower-strength filler metal is used or when small defects are present in the final braze. The lap joint has a double thickness at the joint, but the load is transmitted primarily as shear stress, which is desirable.

Table 2.2 shows the common types of brazed joints.

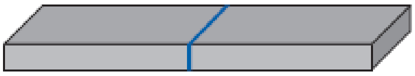
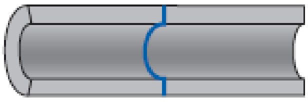
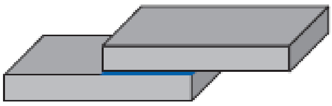
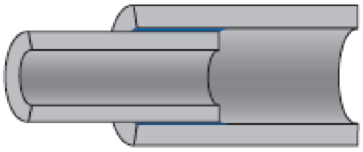
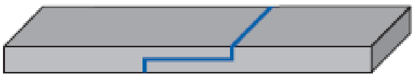
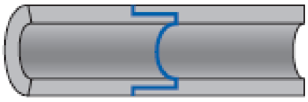
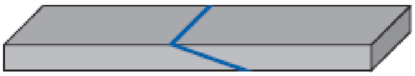
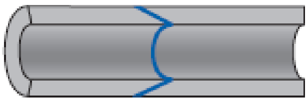
Joint Type	Flat Parts	Tubular Parts (Cutaway)
Butt Joint		
Lap Joint		
Butt-Lapp Joint		
Scarf Joint		

Table 2.2. Common types of brazed joints.

The butt-lap and the scarf joint are attempts to combine the advantage of a single thickness with maximum bonding area and strength. It requires more preparation than straight lap or butt joints and may not be applicable to thin members.

The Clearance, the distance between the faying surfaces to be joined, is the most important design consideration to achieve a good brazed joint. The clearance affects the mechanical performance of the final piece and is strongly related with the amount of intermetallic phases present in the layers, the possibility of voids, the Tensile strength of the final piece and the capillarity action which accounts for filler metal distribution.

The figure 2.5 shows the relation between the tensile strength and the joint clearance

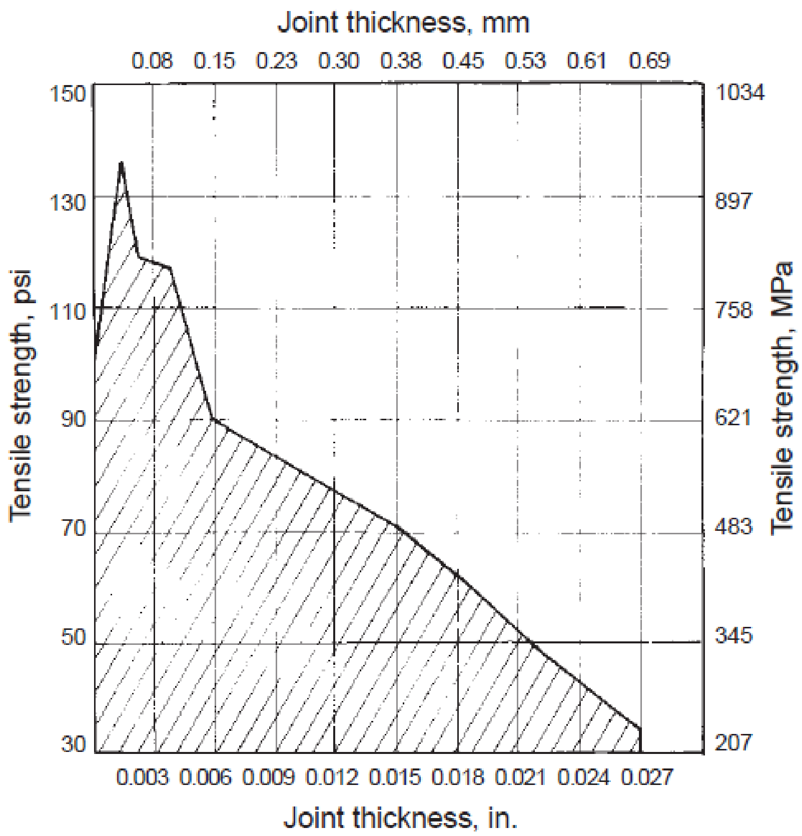


Figure 2.5 Tensile Strength versus Joint clearance [8].

The smaller is the clearance, in situations where there is not extensive alloying and erosion, the easier it is for capillarity to distribute the filler metal throughout the joint area, and there is less likelihood that voids or shrinkage cavities will form as the filler metal solidifies. Small clearances and correspondingly thin filler-metal films make stronger joints.

Table 2.3 shows allowable joint clearances for various filler-metal systems.

Brazing filler-metal system	Joint clearance, mm (in.)
Al-Si alloys(a)	0.15–0.61 (0.006–0.024)
Mg alloys	0.10–0.25 (0.004–0.010)
Cu	0.00–0.05 (0.000–0.002)
Cu-P	0.03–0.13 (0.001–0.005)
Cu-Zn	0.05–0.13 (0.002–0.005)
Ag alloys	0.05–0.13 (0.002–0.005)
Au alloys	0.03–0.13 (0.001–0.005)
Ni-P alloys	0.00–0.03 (0.000–0.001)
Ni-Cr alloys(b)	0.03–0.61 (0.001–0.024)
Pd alloys	0.03–0.10 (0.001–0.004)

(a) If joint length is less than 6 mm (0.240 in.), gap is 0.12 to 0.75 mm (0.005 to 0.030 in.). If joint length exceeds 6 mm (0.240 in.), gap is 0.25 to 0.60 mm (0.010 to 0.024 in.). (b) Many different nickel brazing filler metals are available, and joint-gap requirements may vary greatly from one filler metal to another.

Table 2.3 Brazing joints clearances for different filler metals group and process [8].

Other factors influencing optimal joint gap with a specific filler metal are joint length, brazing temperature, and base-metal reactions.

In the design of a suitable joint. It is important to consider that an assembly expands during heating and that the joint gap may either widen or close by the time the filler metal starts to melt and move. Moreover, ensure that the filler metal amount is enough to absorb room-temperature tensile stresses in order to compensate for any reduction in joint gap.

2.3.3. Filler Metal selection:

The selection of the filler metal is one of the most important of the pre-brazing considerations. The selection of an unsuitable filler metal can lead to complete failure of the process.

When selecting a braze filler metal, there are important factors to consider:

- **The base metals to be joined:** Each of the braze filler metals is formulated for use with specific base metals and combinations of base metals. For example; the silver based fillers, one of the most versatile brazed filler, are used for joining most ferrous and non-ferrous metals except aluminum and magnesium alloys.

- **The brazing process to be used:** Depending on the filler metal composition a special brazing method can be necessary; some metal fillers react very quickly with the environment while others need a particular temperature thus the filler metal should be selected considering also the brazing method available.
- **The design of the joint and method to apply the braze filler metal:** There is a limited number of ways the braze filler metal can be introduced between the base metal faying surfaces. The filler may be pre-placed prior to heating in the joint, or manually face fed after heating. The joint design and brazing process will drive the selection of way to apply the filler metal.
- **The environment and the service of the joint:** The work temperature, corrosion environment, stress conditions, electrical and thermal conductivities, etc. are considerations to take into account when selecting the filler metal.

2.3.4. Heating Methods:

Effective capillary joining requires efficient transfer of heat from the heat source into the joint. The rate of heating, thermal gradients, cooling rates, the size of individual assemblies, and the rate of production are factors which influence in the selection of the heating method.

The joint can be heated in many ways, which are commonly categorized by the actual method of heating. There are six commonly used methods:

- **Torch Brazing:** Manual torch brazing is the method most frequently used because of its relatively low cost and portability, a torch is used to focus flame against the work at the joint. Normally a reducing flame is used to prevent the oxidation. The flame is generated by the combustion of a combination of oxygen and a fuel gas. The process itself give the chance to the use low-melting filler metals, which have excellent flow characteristics, in order to avoid the oxide on the surfaces to be brazed, specials fluxes are normally required to remove these contaminants.

- **Induction brazing:** uses electrical resistance of workpiece and high frequency current induced into the same as a source of heat generation. The parts are pre-loaded with the filler metal and placed in a high frequency AC field. The Frequencies ranging from 5 to 5000 kHz are used. High frequency power source provides surface heating; however, low frequency causes deeper heating into the workpieces. Low frequency current is recommended for heavier and big sections (workpieces). Any production rate, low to high, can be achieved by this process.
- **Resistance Brazing**: Resistance brazing is most applicable to relatively simple joints in metals that have high electrical conductivity. The workpiece, with filler metal preplaced, is part of an electric circuit, which means; heat to melt the filler metal is obtained by resistance to flow of electric current through the joint. Rapid heating cycles can be achieved in resistance Brazing.
- **Dip brazing:** In this case heating of the joint is done by immersing it into the molten soft bath or molten metal bath. In case of salt bath method, filler metal is pre-loaded into the joint and flux is contained inside the hot salt bath. The filler metal melts inside the joint when it is submerged into the hot bath. Its solidification and formation of the joint takes place after taking out the workpiece from the bath. In case of metal bath method, the bath contains molten filler metal. The joint is applied with flux and dipped to the bath. Molten filler metal fills the joint through capillary action. The joint forms during its solidification after taking it out from molten metal bath. Fast heating is possible in this case. It is recommended for making multiple joints in a single workpiece or joining multiple pairs of workpieces simultaneously.
- **Furnace brazing:** In this case, furnace is used to heat the workpieces to be joined by brazing operation. This is a medium- to high-volume production process for self-fixture assemblies with preplaced filler metal. The furnace is purged with a gaseous atmosphere or evacuated from air and heated to a temperature above the liquidus of the filler metal but, less than the melting point of the base metals. The brazements are then cooled or quenched by appropriate methods to minimize distortion and produce the required properties in the filler and base materials. This cycle is

designed to produce the required molten and solidification of the filler metal to join the components without molten or impairing the properties or shape of the base metals.

Figure 2.6 shows the typical braze cycle for furnace brazing; A brazing cycle consist of an initial pumpdown temperature, initial heating ramp, cement burn-off, stabilizing soak, heating ramp to brazing temperature, brazing soak and cooling down.

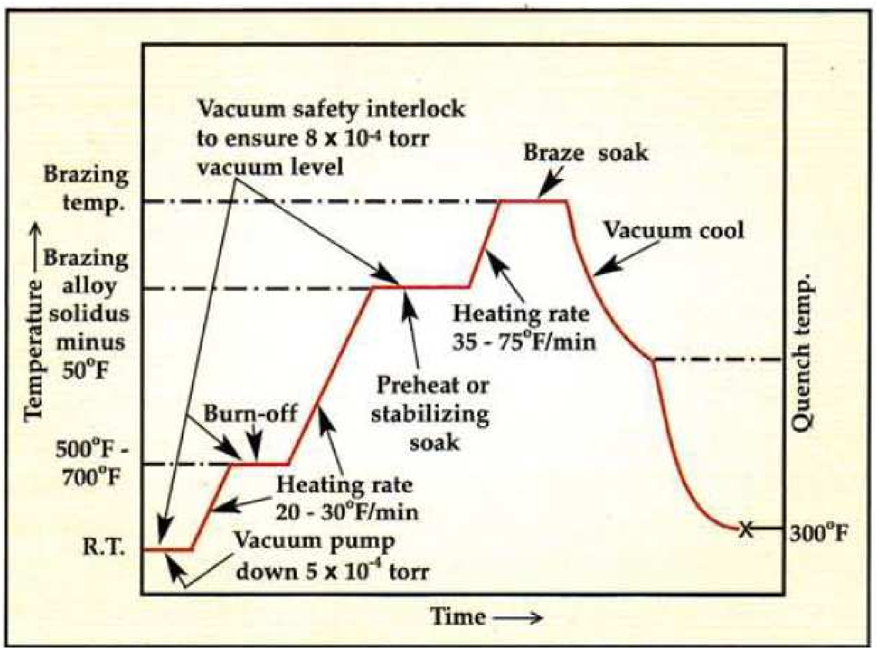


Figure 2.6 Typical braze cycle for furnace brazing.

The pumpdown temperature allows solvents or water in the paste or binder vehicle to outgas from the braze alloy deposit, helps to prevent eruptions (holes) in the braze deposit and restore the atmosphere quality which can degrade from gasses. The burn-off temperature allows the organics (not liquids) in the braze vehicle sufficient time to become gaseous and to be removed through the pumping system, beside allows time to achieve the suitable level of vacuum. The preheat or stabilizing soak allows the temperature throughout the load to equalize, so all parts in the load will reach brazing temperature at approximately the same time during the next heating cycle and it ensure that vacuum pressure levels are low enough before proceeding to brazing temperature. The braze soak allows sufficient time for the alloy to melt and flow into the joint, usually the lowest satisfactory brazing temperature are preferred in order to economize the heat energy required, minimize the heat effect on base metal, minimize the base-metal/filler-metal interactions.

2.4.Post-Brazing treatments and inspection:

Parts that are brazed in a suitable atmosphere should be bright and clean and should require no further processing. However, if flux or stop off material is present, it should be thoroughly removed. If the brazements require heat treatment, it must be done below the solidus or re-melt temperature of the filler metal.

A quality control system should be adequate for both general and critical applications. Inspection of finished brazed assemblies includes visual inspection, leak testing and radiographic examination. Visual inspection is the most widely used nondestructive method. Fluorescent penetrant inspection (FPI) is used for machined surfaces only; parts should be inspected for cracks or voids in brazed joint. Leak testing is most advantageous where gas or liquid tightness or brazed joints is required. Radiography (or ultrasonic inspection) should be used for braze joints to detect sub-surfaces or internal defects.

3. EXPERIMENTAL PROCESURE

3.1. Metal-Ceramic brazing process description:

3.1.1. Brazing Technique:

Depending on the heating method several brazing techniques were descripted in chapter III. Since the present of oxide in the faying surfaces to be joined is detrimental to brazing process and consequently to brazed piece quality, an oxygen-free environment is preferential for successful brazing. The active metals in the filler act like as “oxygen-getter” and an “oxygen-free” environment could be advantageous to improve the wettability of the ceramics.

There are two common ways to obtain the “oxygen-free” environment; use fluxes or a vacuum chamber. Fluxes prevent the formation of oxide on the faying surfaces to be joined but if the piece is exposed to high temperatures for a long time the flux could be evaporated and the process could be compromise.

The purity levels of atmosphere achieved in vacuum chamber are much higher than these obtained using fluxes. Also oxide layers on brazing parts are decomposed in a vacuum at high temperature, which improves base metal wetting resulting, in better joints properties as strength, minimum porosity, etc. [9]

Considering that a controllable oxygen-free environment helps to avoid the formation of oxide on the surface, increase the probability of “oxygen-getter” in the active filler react with the oxygen on the ceramics and not with oxygen in environment and consequently contribute to improve the joints properties the Vacuum Furnace was selected as brazing technique.

3.1.2. The Vacuum Chamber:

A multi-purpose chambers system is used to perform the furnace brazing process (figure 3.1.a). The system consists of four vacuum chambers connected through a central zone and separated one of another by pneumatic gates. A chamber is used to develop the experiments (figure 3.1b).

A working chamber vacuum pressure up to 10E-6 mbar is reached by a two stages pumping system, the Pfeiffer turbo molecular pump of 360 l/min and Varian Dry Scroll Pump 210 l/min as a primary pump. The entire system is controlled by a HMI (human-machine interface) (figure 3.1c).

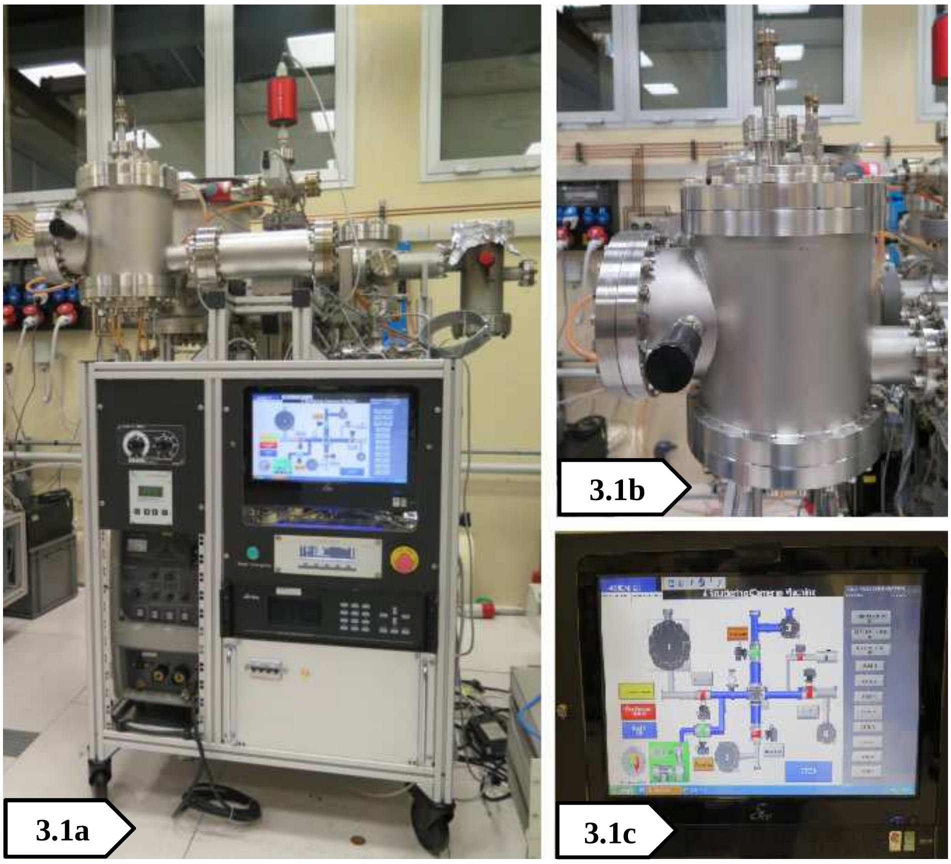


Figure 3.1: a) Multi-purpose chambers system for furnace brazing. b) Vacuum chamber for brazing experiments. c) HMI to control the entire system.

3.1.3. The Ovens:

To increase the temperature inside the vacuum chamber, there are two common options; heat through a set of resistances located inside the chamber or by inductive heating. Both methods are clean, accurate and easily automatized but using inductive heating some important parameters as the shape and dimensions of the coil, the penetration of the magnetic field and consequently the concentration of energy (heat) inside the sample depend on the shape and dimensions of the samples. It is a great disadvantage for experimental purposes. On the other hand, heating by a set of resistances represents a more versatile option.

The vacuum furnace temperature is increased by two ovens located on top and bottom of the vacuum chamber (figure 3.2). Each Oven (figure 3.3) consists of a sample holder, an IR lamp of 5000 W, a shield to concentrate the heat around the sample holder, a thermocouple of type K and the electrical connections for lamp and thermocouple.

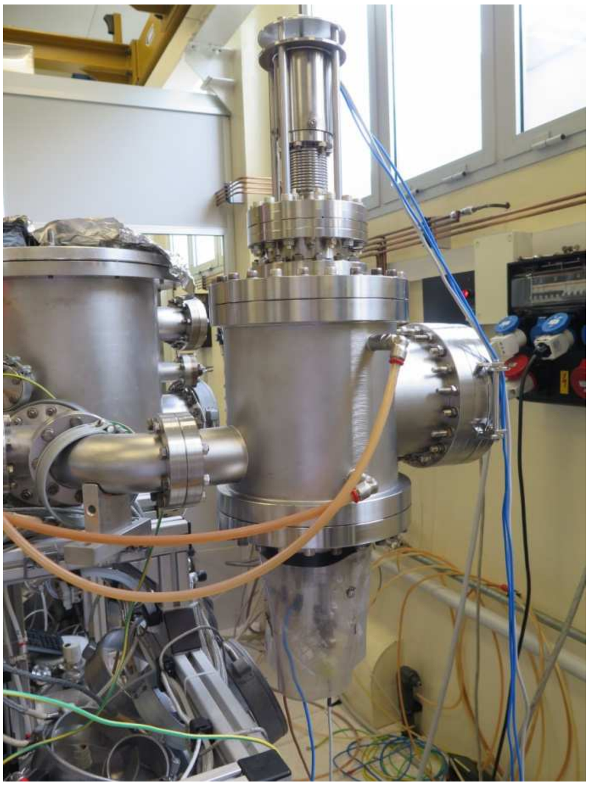


Figure 3.2: Top and bottom oven on the vacuum chamber.

The figure 3.3 shows the parts of the bottom oven.

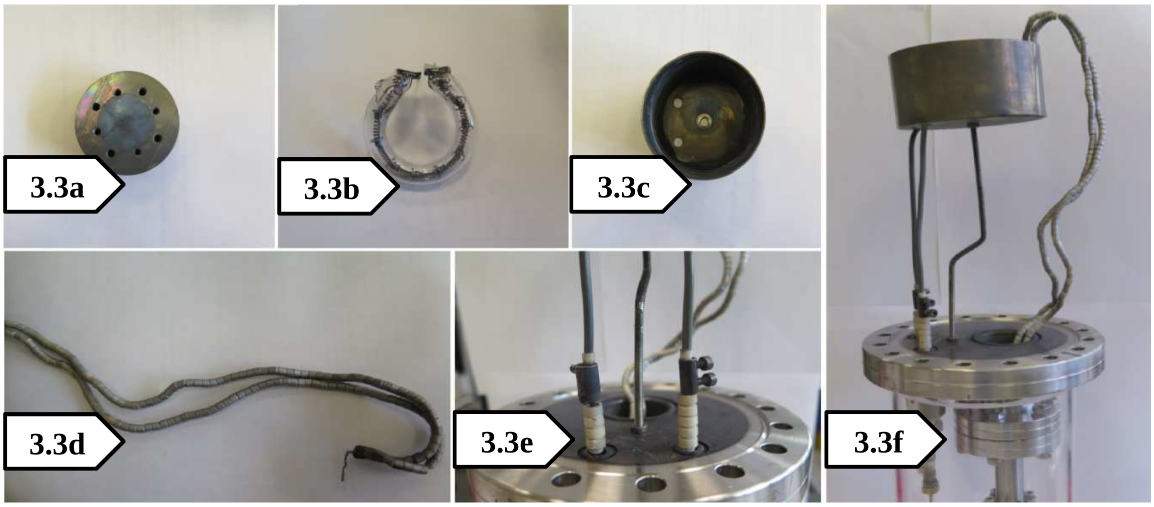


Figure 3.3: Parts of oven a) Sample holder. b) 5000 W IR lamp. c) Shield for lamp. d) Thermocouple K. e) Connections for thermocouple and lamp. f) Bottom oven assembled

The top oven has a special configuration which allows it to change the distance between the samples holders of each oven. Figure 3.4 shows an illustration of the top oven.

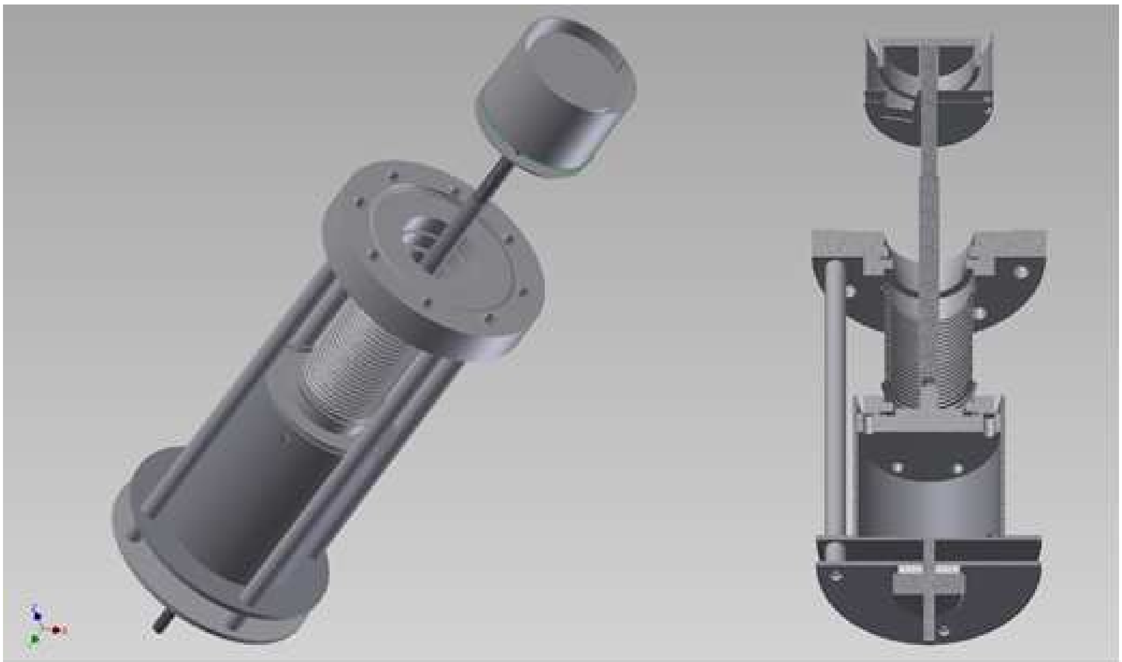


Figure 3.4: Illustration of the top oven.

3.1.4. The metallization of ceramics:

The ceramic samples were metallized by DC Magnetron sputtering technique. Deposition was carried out on a second chamber of the same vacuum system. Base pressure of $5x10^{-6}mbar$ was reached.

Figure 3.5 shows several magnetrons used on these experiments.

Figure 3.5: Magnetrons for DC Sputtering.

Table 3.1 summarizes the parameter used for these depositions:

Titanium Depositon		Titanium and Silver deposition.	
		Ti deposition	Ag deposition
Base pressure :	x10^{-6}mbars	x10^{-6}mbars	x$\mathbf{10^{-6}}$mbars
Argon flux:	6,4 sccm	6,4 sccm	6 sccm
Work pressure:	x10^{-3}mbars	x10^{-3}mbars	x10^{-3}mbars
Current level:	0.5 A	0.5 A	0.25 A
Sample/Magnetron distance	9 cm	9 cm	9 cm
Time	~10 min	~15 min	~120 min

Table 3.1 Parameter of magnetron sputtering for ceramic metallization.

There are two procedures for ceramics metallization:

- **Procedure 1:** The Ti metallization, a thin layer of Titanium (0.25-2μm) is deposited on the ceramic.
- **Procedure 2:** The Ti-Ag metallization, this is a two steps process, first a thin layer of Titanium (0.3-0.5 μm) is deposited on the ceramic. Then a thin layer of silver (10-15 μm) is deposited over the Titanium coating.

Figure 3.6 shows several ceramic samples metallized.

Figure 3.6: Alumina metallized samples.

3.1.5. The cleaning process:

Each sample was cleaned before brazing process following this procedure:

- Washing in ultrasonic bath into soap solution (Rodaclean) for 30 min at 40°C.
- Washing in ultrasonic bath into deionized water for 30 min at room temperature.
- Rinsing in Ethanol.
- Drying with nitrogen gas.

Figure 3.7 shows the process of cleaning of stainless steel sample.

Figure 3.7: Process of cleaning of stainless steel sample.

3.1.6. The Assembly of pieces and filler application:

The metallic and ceramic samples were assembled in lap-joint configuration (figure 3.8).

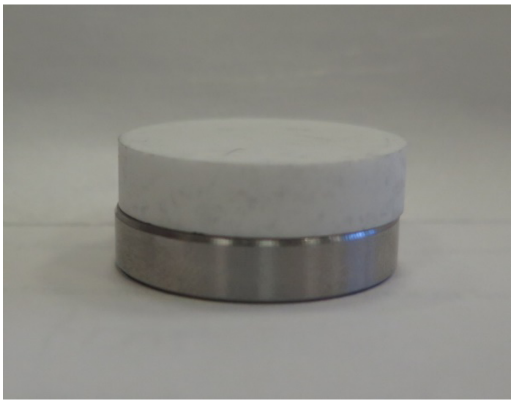


Figure 3.8: Metallic and ceramic samples on lap-joint configuration.

The application of the filler depends on its nature and shape; the foil filler metal is applied directly between the surfaces of the base materials. Figure 3.9 shows the assembly procedure of the pieces with foil filler. An Inconel ring and screws are used to fix and align the assembly.

Figure 3.9: Procedure to assembly the metallic and ceramic samples with foil filler.

In order to assembly the wire filler metal to the samples, a circular cavity was made over the metallic sample surface to fix the wire. Figure 3.10 shows the assembly procedure of the pieces with wire filler.

Figure 3.10: Procedure to assembly the metallic and ceramic samples with wire filler.

The assembly is fixed to the sample holder of the bottom oven by an Inconel ring in order to maintain the pieces aligned. Then the top oven was placed near to the assembly, in this way both ovens can irradiate heat to the piece.

Figure 3.11 shows the two ovens and the piece assembled.

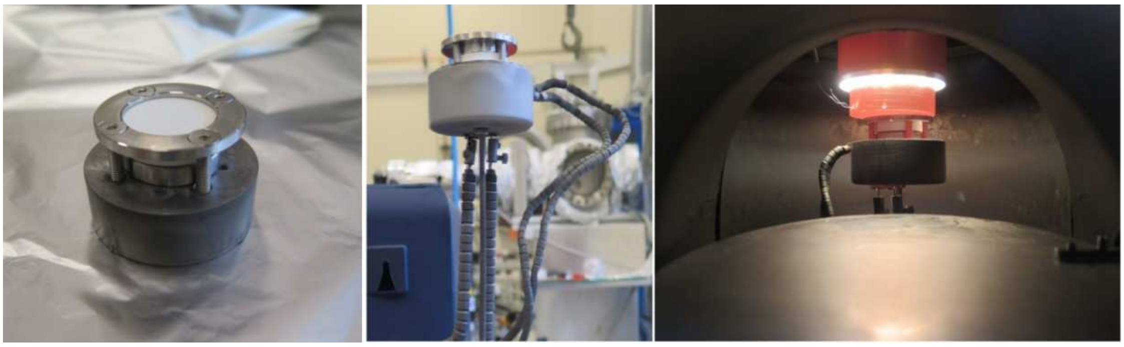


Figure 3.11: The piece assembled to the bottom oven and the top oven.

3.1.7. The heating process:

The vacuum chamber heating was carried out by IR lamps. A temperature control module (figure 3.12) based on a commercial temperature control device and a power controller device is used to perform the temperature profiles.

The thermocouples K close the control loop and thus the PID controller changes the power applied to IR lamp and the temperature in the chamber could be modified, this PID controller allows the user to perform gradients of heating and cooling in order to follow the brazing temperature profiles.

Figure 3.12: The temperature control module.

3.2.The materials description:

3.2.1. The base materials:

The hardness, high fusion temperature, chemical stability, availability on market and low cost made the Alumina (Al_2O_3) the most suitable options for the ceramic part of the junction.

An Alumina (Al_2O_3) disc with diameter of 28 mm and thickness of 6 mm with 99.99% of purity was used; the figure 3.13 shows the dimensions of the samples.

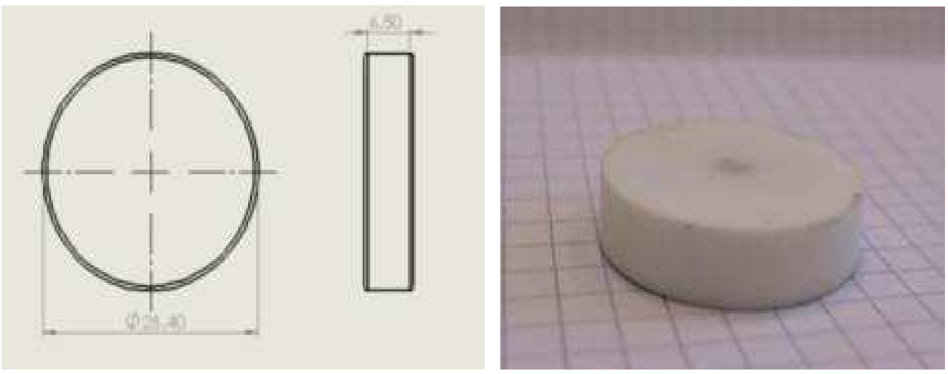


Figure 3.13: Dimensions of ceramic samples.

Stainless steel 304, the most versatile and commonly used kind of Stainless steel, was selected as the metallic component of the assembly.

INCONEL, a family of nickel-chromium based super alloys, has been chosen as additional variant of metallic component of assembly because of its relatively low thermal expansion coefficient (more close to the one of ceramic materials chosen).

A Stainless steel 304 and INCONEL discs with diameter of 28 mm and thickness of 5.5 mm were used; the figure 3.14 shows the dimensions of the samples.

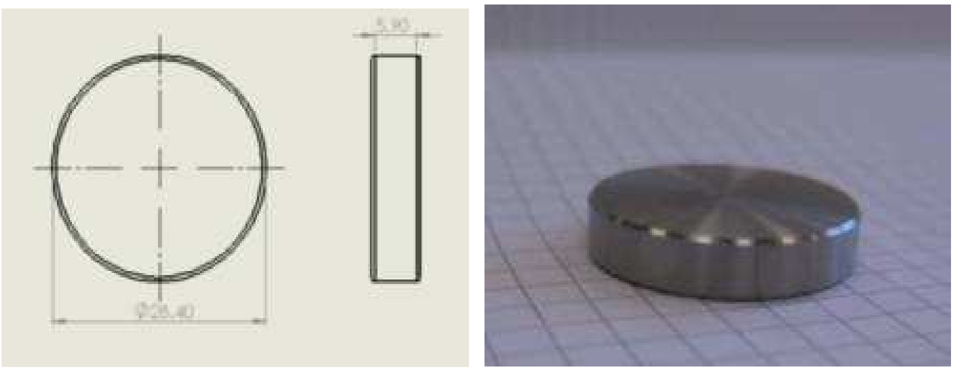


Figure 3.14: Dimensions of metallic samples.

The table 3.2 shows the composition of the stainless steel 304 family, while the table 3.3 shows the composition of INCONEL family.

Grade		**C**	**Mn**	**Si**	**P**	**S**	**Cr**	**Ni**	**N**
304	max	-	-	-	-	-	18.0	8.0	-
	min	0.08	2.0	0.75	0.045	0.030	20	10.5	0.10
304L	max	-	-	-	-	-	18.0	8.0	-
	min	0.030	2.0	0.75	0.045	0.030	20.0	12.0	0.10
304H	max	0.04	-	-	-	-	18.0	8.0	-
	min	0.10	2.0	0.75	0.045	0.030	20.0	10.5	-

Table 3.2 – Composition of Stainless steel 304 family.

Inconel		Ni	Cr	Fe	Mo	Nb	Co	Mn	Cu	Al	Ti	Si	C	S	P	B
600	min	-	14.0	6.0	-	-	-	-	-	-	-	-	-	-	-	-
	max	72.0	17.0	10.0	-	-	-	1.0	0.5	-	-	0.5	0.15	0.015	-	-
617	min	44.2	20.0	-	8.0	-	10.0	-	-	0.8	-	-	-	-	-	-
	max	56.0	24.0	3.0	10.0	-	15.0	0.5	0.5	1.5	0.6	0.5	0.15	0.015	0.015	0.006
625	min	-	20.0	-	8.0	3.15	-	-	-	-	-	-	-	-	-	-
	max	58.0	23.0	5.0	10.0	4.15	1.0	0.5	-	0.4	0.4	0.5	0.1	0.015	0.015	-
690	min	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	max	59.5	30	9.2	-	-	-	0.35	0.01	0.02	-	0.35	0.019	0.003	-	-
718	min	50.0	17.0	balan	2.8	4.75	-	-	0.2	0.65	-	-	-	-	-	-
	max	55.0	21.0	ce	3.3	5.5	1.0	0.35	0.8	1.15	0.3	0.35	0.08	0.015	0.015	0.006
X-750	min	-	14.0	5.0	-	0.7	-	-	-	0.4	2.25	-	-	-	-	-
	max	70	17.0	9.0	-	1.2	1.0	1.0	0.5	1.0	2.75	0.5	0.08	0.01	-	-

Table 3.3 – Composition of INCONEL family.

3.2.2. The Filler Metals:

In order to select the suitable filler-metals these considerations were taken into account:

- The ability to "wet" the base materials.
- Suitable melting and flow properties to permit distribution by capillary attraction.
- The ability to make a strong bond to the base material.
- The filler-metal must be able to produce a braze joint that will meet the service requirements specified.

Considering these arguments the brazing filler metals on the table 3.4 and 3.5 were selected:

Brazing Filler-Metals	Manufacturer	Composition		
		Ag%	Cu%	Pd%
Pallabraze 850	Johnson Matthey	58.5	31.5	10
Copper	Good fellow	0	100	0

Table 3.4 - Non-active brazing filler-metals.

Active Brazing Alloys	Manufacturer	Composition		
		Ag%	Cu%	Ti%
Cusil ABA	WESGO metal	63	35.25	1.75

Table 3.5 - Active brazing filler-metals.

The most manufacturers or providers can supply five basic forms of filler-metals: wire, powder, foil, paste and performs. Taking into account factors as joint type, clearance between pieces, the filler-metal application mode and the brazing technique; wire, disc pre-form, and foils were chosen as filler metal forms for these experiments.

The wire selected has 0.8 mm of diameter; the disc of Pallabraze 850 has the same diameter of the samples and 0.3 mm of thickness; whereas the foil of CUSIL-ABA and copper has the same diameter of the samples and 8-10 μm of thickness, figure 3.15 shows the dimensions of some of these filler-metals forms.

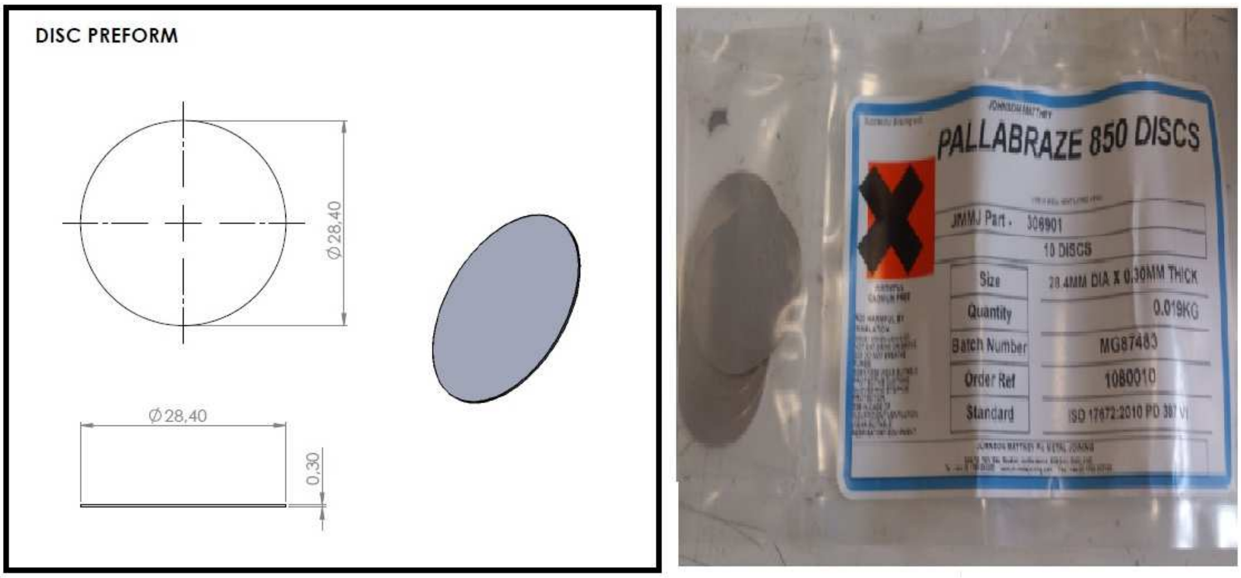


Figure 3.15: Dimensions and real pictures of pallabraze 850 discs.

3.3. The brazing experiments:

Depending on the base materials, the filler metal; composition (active or no active) and form (wire or disc) and the ceramic metallization technique, there are several brazing experiments, classified as:

3.3.1. Active Brazing experiments:

These include different combinations of base materials and different forms of CUSIL-ABA, resulting in the following experiments:

Denomination	Base materials	Filler Metal	
		Commercial name	Form
Experiment 1	Stainless Steel 303 -Alumina	CUSIL-ABA	Wire
Experiment 2	Inconel 600-Alumina	CUSIL-ABA	Wire
Experiment 3	Stainless Steel 304-Alumina	CUSIL-ABA	Foil
Experiment 4	Inconel 600-Alumina	CUSIL-ABA	Foil

Table 3.6 Active brazing experiments.

3.3.2. Non-active Brazing-metallized ceramic experiments:

These include different combinations of base materials, different forms of Pallabraze 850 and different methods of metallization; resulting in the following experiments:

Denomination	Base materials	Filler Metal	
		Commercial name	Form
Experiment 5	Stainless Steel 303 –Ti Alumina	Pallabraze 850	Foil
Experiment 6	Inconel 600-Ti Alumina	Pallabraze 850	Foil
Experiment 7	Stainless Steel 304- Ti&Ag Alumina	Copper	Foil
Experiment 8	Inconel 600- Ti&Ag Alumina	Copper	Foil

Table 3.6 Non-active brazing experiments.

3.4.Brazed samples characterization:

3.4.1. Cutting the brazed pieces:

The brazed pieces were cut by a combination of Electrical Discharge Machining (EDM) and a precision diamond wire saw machine. The EDM technique was used in order to cut the metallic component and the diamond saw was used to cut the ceramic.

Figure 3.16 shows the brazed sample been cut by diamond saw machine.

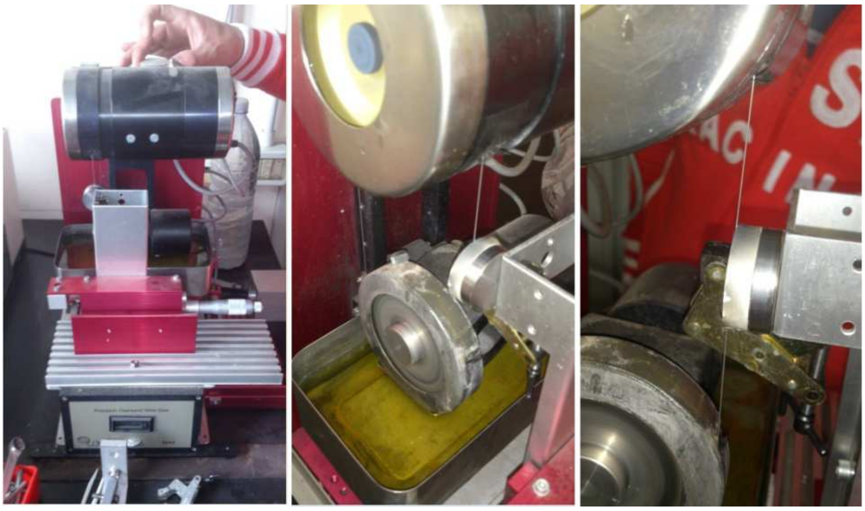


Figure 3.16: Brazed sample been cut by diamond wire saw machine.

3.4.2. SEM analysis:

FEI (ex Philips) Scanning Electron Microscope SEM XL-30 has been used to study the cross-section of the pieces with emphasis on the interlayer area, searching for evidence of filler metal diffusion into the base materials.

3.4.3. EDAX analysis:

Energy-dispersive X-ray spectroscopy (EDS, EDX) is an analytical technique used for the elemental analysis of a sample. EDX technique has been used for elemental analysis of the brazed pieces interlayers in order to identify elements of filler metal inside the base materials.

4. EXPERIMENTAL RESULTS AND ANALISYS

4.1. Active brazing experiments:

4.1.1. Experiment 1: stainless steel-Alumina with CUSIL-ABA wire:

Samples of stainless steel 304 and alumina were selected as base materials, a CUSIL-ABA wire with 0.8 mm of diameter and 16 mm of length was used as filler. Two rings were formed with the CUSIL-ABA wire and circular channels were carved over the metallic sample surface in order to fix these rings.

The pieces was cleaned and assembled following the procedures explained in section 3.1.5 and 3.1.6.

Figure 4.1 shows a general procedure to brazing process.

Figure 4.1: Stainless steel–Alumina procedure for brazing. a) Samples and filler metal cleaned. b) Assembly of filler metal rings. c) Heating in vacuum chamber.

The figure 4.2 shows the profile of temperature. A brazing temperature of 848 °C was reached and maintained for 15 min then a cooling down profile of 8-10°C min was applied.

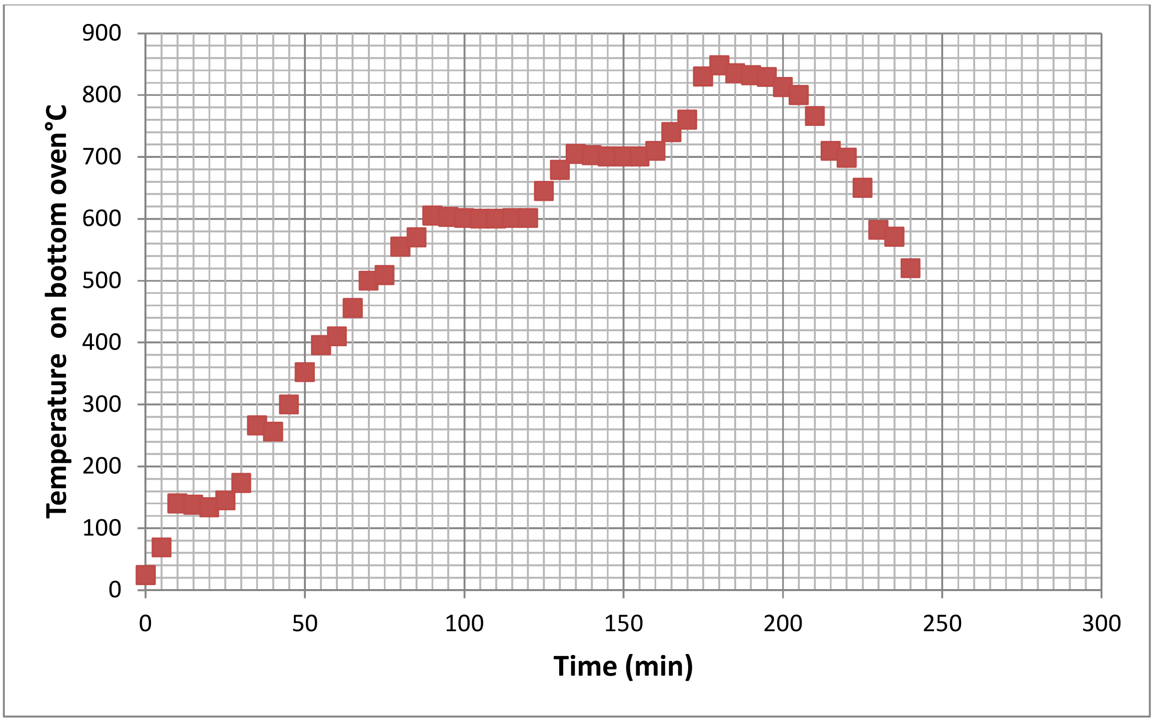


Figure 4.2: Profile of temperature in Stainless steel-Alumina brazing process with CUSIL-ABA.

Figure 4.3 shows the Stainless steel-Alumina assembly after brazing, several cracks could be observed on the ceramic component. When a minimal amount of force was applied to the joint area the ceramic was shattered.

Figure 4.3: Stainless steel-Alumina brazed with CUSIL-ABA wire.

4.1.2. Experiment 2: Inconel-Alumina with CUSIL-ABA wire:

Samples of inconel and alumina were selected as base materials, a CUSIL-ABA wire with 0.8 mm of diameter and 16 mm of length was used as filler. The wire was applied with spiral shape and a spiral cavity was carved over the metallic sample surface in order to fix the filler. The pieces was cleaned and assembled following the procedures explained in section 3.1.5 and 3.1.6.

The figure 4.4 shows the profile of temperature measured on both ovens. A brazing temperature of 859 °C was reached and maintained for 15 min, for this experiment a more aggressive ramp of temperature was applied to the ceramic piece, on the other hand in cooling down a ramp of 5-8°C min was applied to the Inconel component whereas the temperature was decreased till 750 °C on the ceramic component and maintained for several minutes and then cooled down.

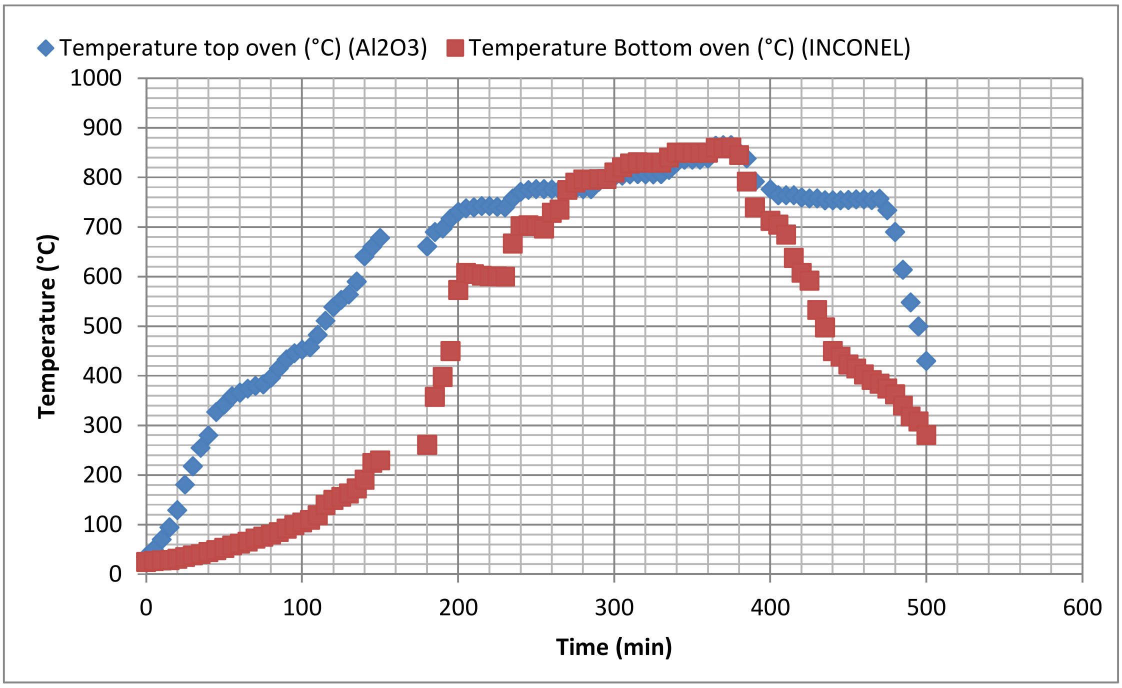


Figure 4.4: Profile of temperature in Inconel-Alumina brazing process with CUSIL-ABA.

Figure 4.5 shows the Inconel-Alumina assembly after brazing, in this case several cracks could be observed on the ceramic component too and when a minimal amount of force was applied to the joint area the ceramic was shattered. This behaviour in both experiments could be caused by the residual stress due the different of thermal expansion between the base materials.

Figure 4.5: Stainless steel-Alumina brazed with CUSIL-ABA wire.

4.1.3. Experiment 3: Stainless steel-Alumina with CUSIL-ABA foil:

Samples of stainless steel and alumina were selected as base materials, a CUSIL-ABA foils were used as filler. The foil was applied between the faying surfaces of the brazements.

The pieces were cleaned and assembled following the procedures explained in section 3.1.5 and 3.1.6.

The figure 4.6 shows the profile of temperature of a brazing process using a CUSIL-ABA disc of 28 mm of diameter and 8~10 μm of thickness. A brazing temperature of 865 °C was reached and maintained for 15 min, a cooling down a ramp of 3-5°C min was applied to the metallic component.

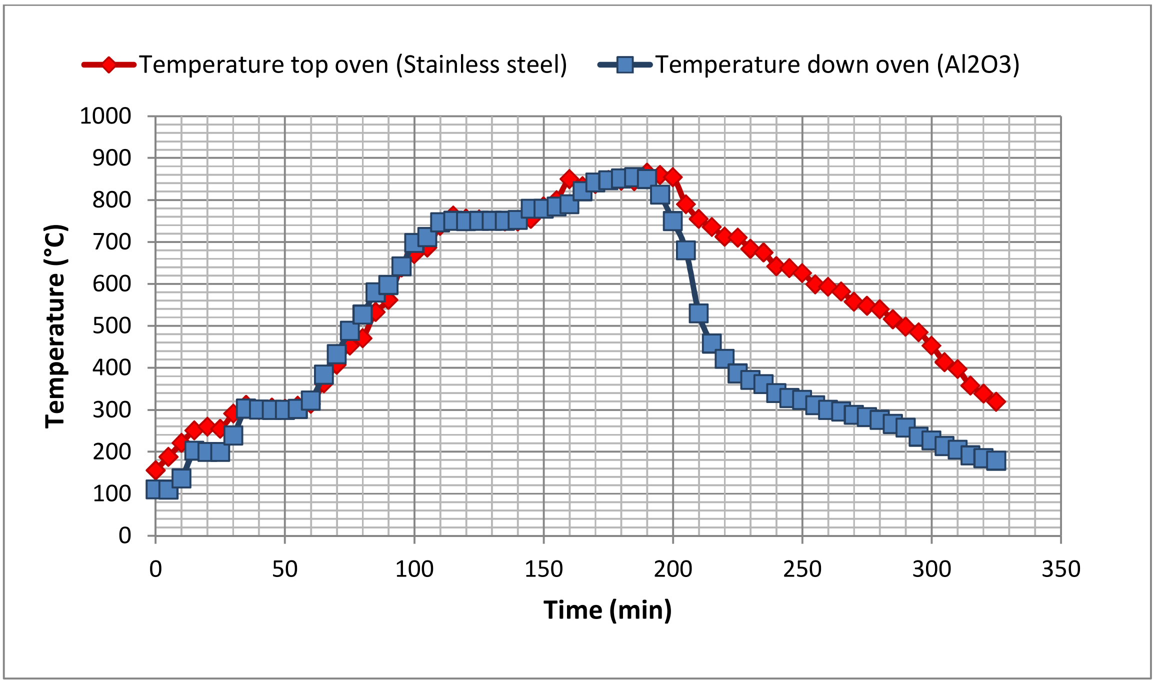


Figure 4.6: Profile of temperature in Stainless steel-Alumina brazing process with CUSIL-ABA.

Figure 4.7 shows the stainless steel-Alumina assembly after brazing, in this case no cracks were observed on ceramic component unlike the previous experiments.

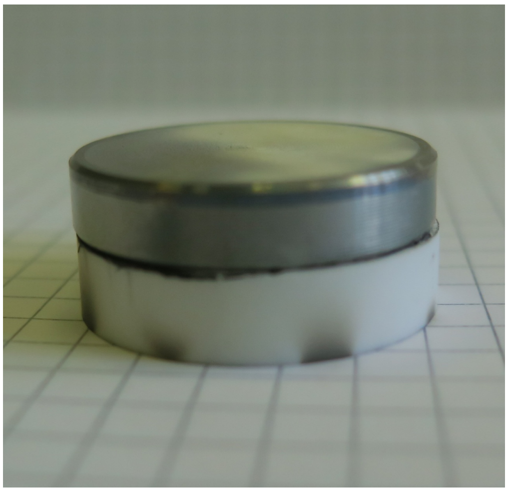


Figure 4.7: Stainless steel-Alumina brazed with CUSIL-ABA foil.

4.1.4. Experiment 4: Inconel-Alumina with CUSIL-ABA foil:

Samples of inconel and alumina were selected as base materials, a CUSIL-ABA foils of several dimensions were used as filler. The foil was applied between the faying surfaces of the brazements.

The pieces were cleaned and assembled following the procedures explained in section 3.1.5 and 3.1.6.

The figure 4.8 shows the profile of temperature of a brazing process using a CUSIL-ABA disc of 28 mm of diameter and 8~10 μm of thickness. A brazing temperature of 863 °C was reached and maintained for 10 min, a cooling down a ramp of 3-5°C min was applied.

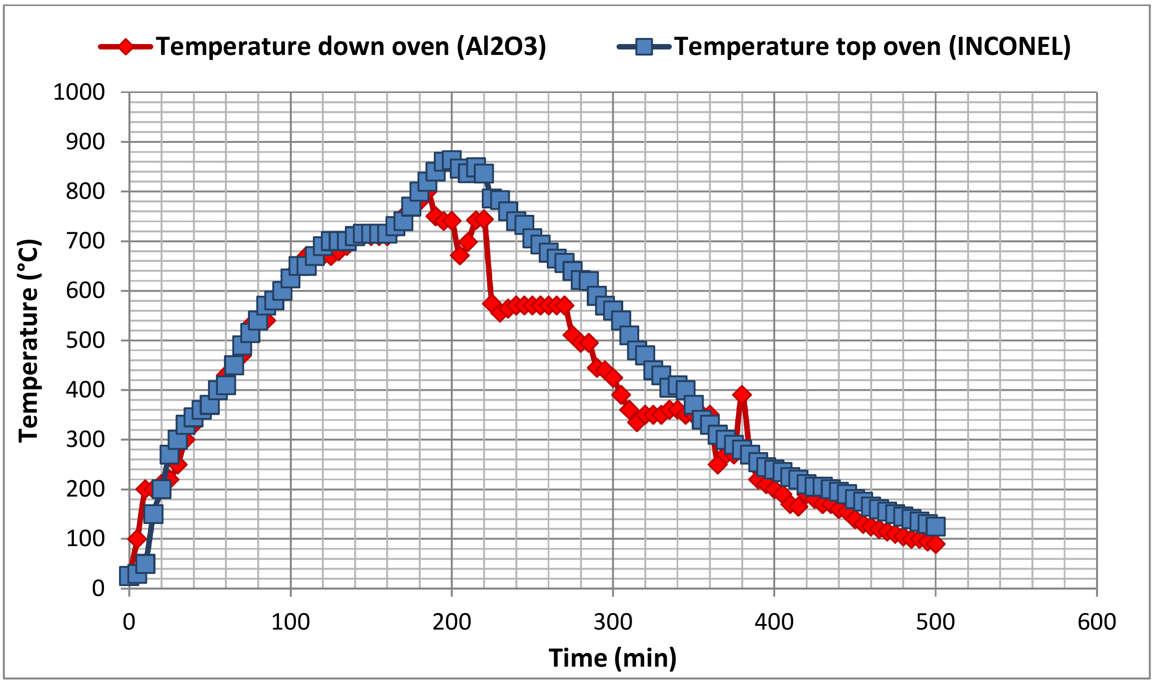


Figure 4.8: Profile of temperature in Inconel-Alumina brazing process with CUSIL-ABA.

Figure 4.9 shows a stainless steel-Alumina assembly after brazing, in this case no cracks were observed on ceramic component too. These results in both experiments could be consequence of a uniform distribution of residual stress due the uniform distribution of the filler on the lapping area, also due the application of a slower cooling down ramp in order to avoid the residual stress caused by different on thermal expansion.

Figure 4.9: Inconel-Alumina brazed with CUSIL-ABA foil.

A scanning electron microscope (SEM) image of the sample described above is shown on figure 4.10a. In the cross-section a several micro empty spaces could be observed in the interlayer or reaction zone, these empty spaces could be caused by some contamination on the assembled part surfaces, these impurities often could contain ingredients that tend to volatilize and outgas at the elevated temperatures involved in brazing [10], on the other hand, figure 4.10b and 4.10c show the cross-section of samples in which lowers amount of filler were used, a gap between the ceramic and the filler metal could be observe, this gap could be a consequence of a shortfall in the amount of filler metal.

An energy-dispersive X-ray spectroscopy (EDS-EDX) on the cross-section of the sample in figure 4.10a reveals elements of the filler metal as titanium, silver and copper on the base material nearby to the junction area, the presence of these elements could be evidence of filler metal diffusion in this area, otherwise EDS-EDX analysis reveals no signs of filler metal in the base materials nearby to the junction in figure 4.10b and 4.10c samples.

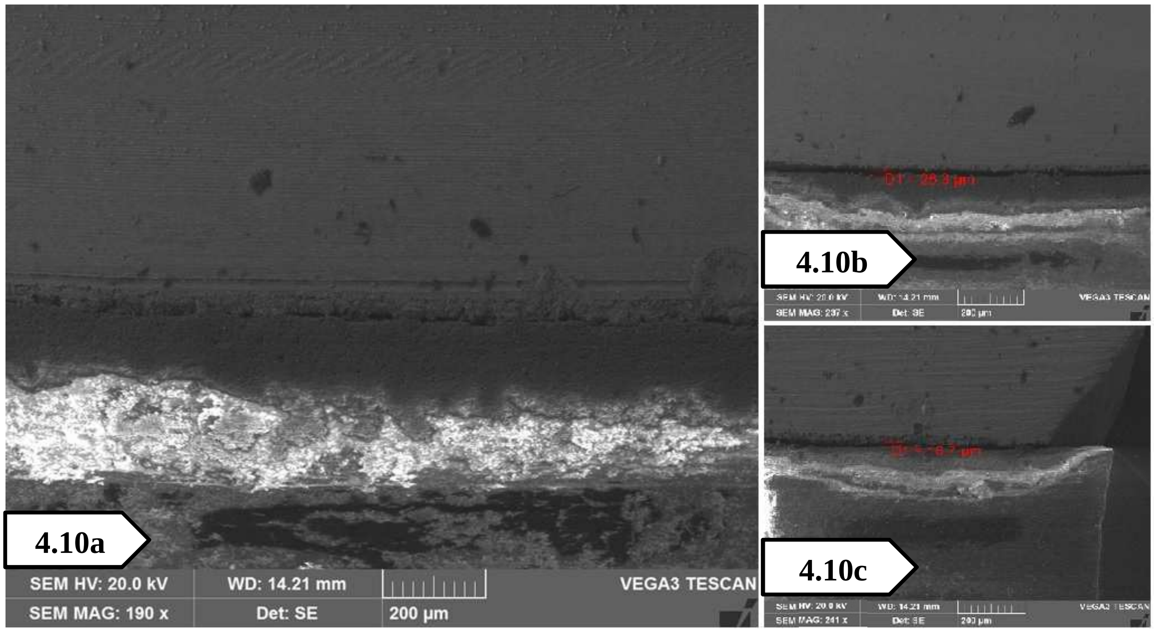


Figure 4.10: SEM image of Inconel-Alumina brazed cross-section with CUSIL-ABA foil.

4.2. Non-active brazing-metallized ceramic:

4.2.1. Experiment 5: Stainless Steel- Ti metallized Alumina with Pallabraze 850:

Samples of stainless steel and alumina were selected as base materials, Pallabraze 850 discs were used as filler. The foil was applied between the faying surfaces of the brazements.

A Titanium film was coated on the ceramic surface following the procedures 1 explained in section 3.1.4.

The metallic pieces and the filler disc were cleaned following the procedures explained in section 3.1.5. The ceramic was cleaned before the metallization. The pieces were assembled fallowing the procedure explained in section 3.1.6.

The figure 4.11 shows the profile of temperature of a brazing process using a Pallabraze 850 disc of 28 mm of diameter and 0.3 mm of thickness and a 0.25 μm layer of Titanium. A brazing temperature of 865 °C was reached and maintained for 10 min, a cooling down a ramp of 3-5°C min was applied.

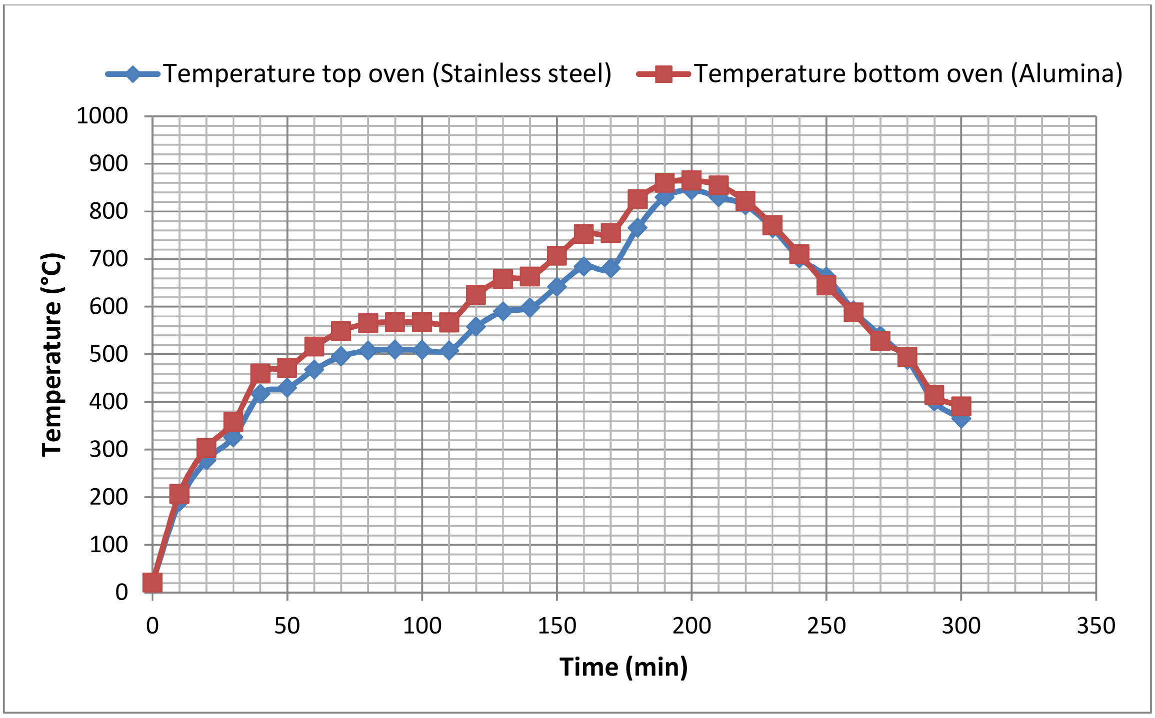


Figure 4.11: Profile of temperature in stainless steel – Ti metallized Alumina brazing process with Pallabraze 850.

Figure 4.12 shows a stainless steel- Ti metallized Alumina brazed.

Figure 4.12: Stainless steel- Ti metallized Alumina brazed with Pallabraze 850 foil.

A scanning electron microscope (SEM) image of the sample described above is shown on figure 4.13a. A uniform distribution of the filler metal could be seen; however as was observed on the experiment with CUSIL-ABA foils, several micro empty spaces appear again.

An energy-dispersive X-ray spectroscopy (EDS-EDX) on the cross-section of the sample in figure 4.13a reveals elements of the filler metal as palladium, silver and copper on the base material nearby to the junction area.

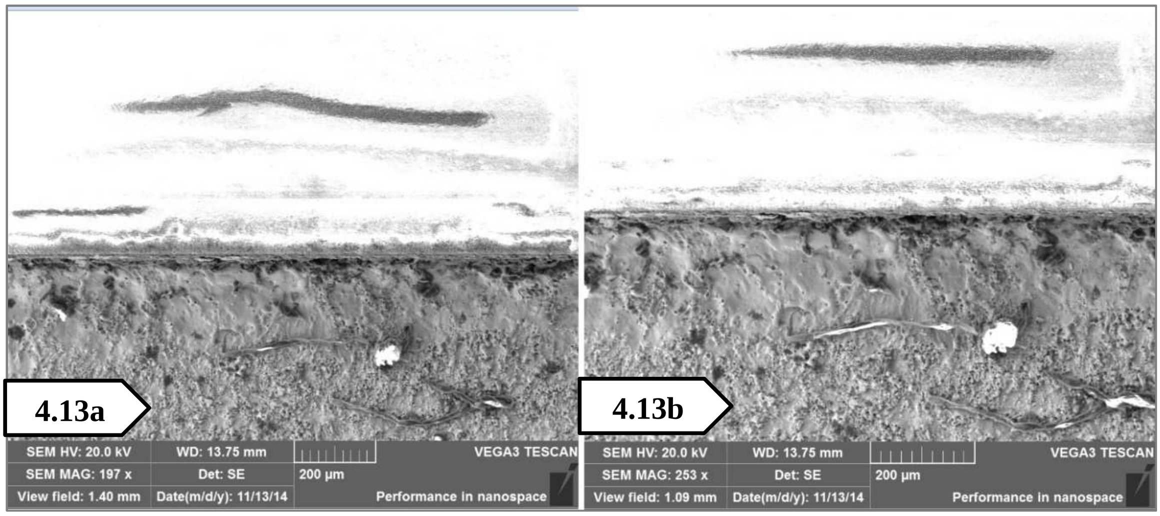


Figure 4.13: SEM image of stainless steel – Ti metallized alumina brazed cross-section with Pallabraze 850 foil.

4.2.2. Experiment 6: Inconel- Ti metallized Alumina with Pallabraze 850:

Samples of inconel and alumina were selected as base materials, Pallabraze 850 discs of several sizes were used as filler. The foil was applied between the faying surfaces of the brazements.

A Titanium film was coated on the ceramic surface following the procedures 1 explained in section 4.1.2. The metallic pieces and the filler disc were cleaned following the procedures explained in section 4.1.3. The ceramic was cleaned before the metallization. The pieces were assembled fallowing the procedure explained in section 4.1.4.

Figure 4.14 shows the profile of temperature of a brazing process using a Pallabraze 850 disc of 28 mm of diameter and 0.1 mm of thickness and a 1 μm layer of Titanium. The process started with a pressure of $2x10^{-5}mbar$, then the chamber was filled with argon gas till achieve a pressure of $3.2x10^{-3}mbar$. A brazing temperature of 851 °C was reached and maintained for 5 min, a cooling down a ramp of 3-5°C min was applied.

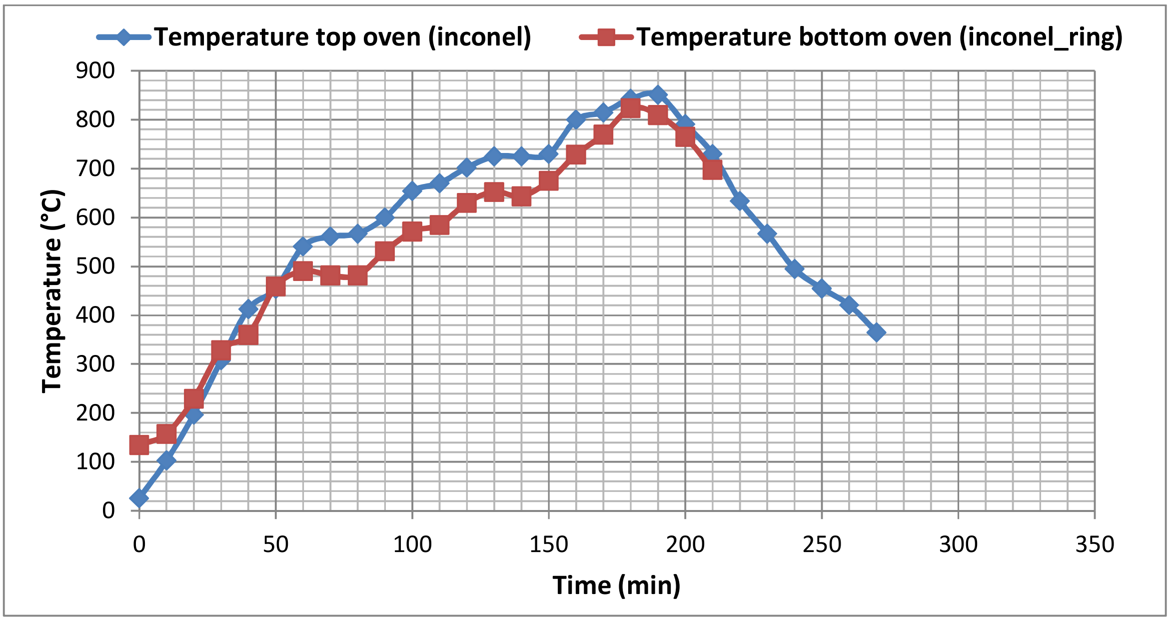


Figure 4.14: Profile of temperature in Inconel – Ti metallized Alumina brazing process with Pallabraze 850.

A scanning electron microscope (SEM) image of the sample described above is shown on figure 4.15a. In the cross-section could be seen a uniform distribution of the filler metal and no empty spaces could be observed. On the other hand, Figure 4.15b and 4.15c show the cross-section of samples in which a lower amount of filler metal and higher pressure were used, empty spaces and gaps could be observed.

An energy-dispersive X-ray spectroscopy (EDS-EDX) on the cross-section of the sample in figure 4.15a reveals elements of the filler metal as palladium, silver and copper on the

Inconel nearby to the junction area, the presence of these elements could be evidence of filler metal diffusion in this area, on the other hand EDS-EDX analysis on the samples in figure 4.15b and 4.15c reveals presence of these elements too but there are no adhesion to ceramic.

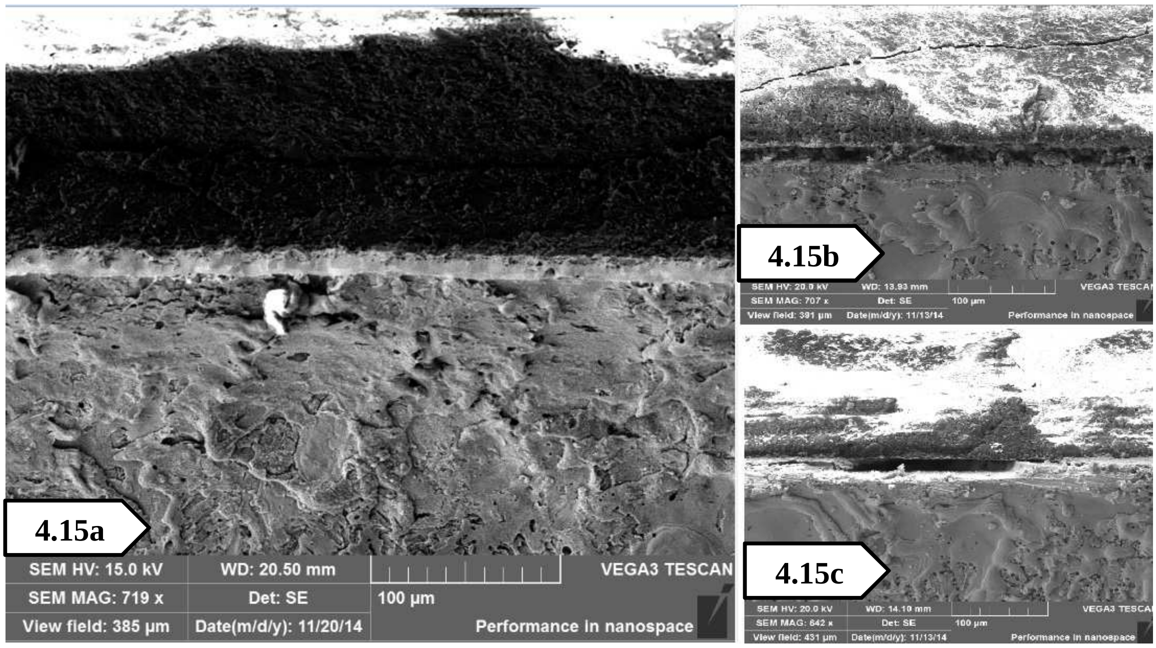


Figure 4.15: SEM image of several Inconel– Ti metallized alumina brazed cross-sections with Pallabraze 850 foil.

The presence of these element as Titanium, silver and copper detected on EDS-EDX analysis could be an evidence that it is diffusion of the filler metals inside the metal component of the junction but in the cases in which the pressure was higher than $x10^{-6}mbar$ a gap or groups or gaps were observed and these voids decrement the adhesion with the alumina, in the opposite case, when the chamber was refilled with argon gas and a pressure of $x10^{-3}mbar$ was achieved, the gap was not observed, this phenomenon could be caused because by increasing the pressure the vaporization temperature also increase [10].

4.2.3. Experiment 7: Stainless Steel- Ti&Ag metallized Alumina with copper:

Samples of stainless steel and alumina were selected as base materials, a copper disc of several dimensions were used as filler. The foil was applied between the faying surfaces of the brazements.

Two films one of titanium and other of silver were coated on ceramic surface following the procedures 2 explained in section 3.1.4. The metallic pieces and the copper disc were cleaned following the procedures explained in section 3.1.5. The ceramic was cleaned before the metallization. The pieces were assembled fallowing the procedure explained in section 3.1.6.

Figure 4.17 shows the profile of temperature of a brazing process using a copper foil with 28 mm of diameter and 8~10 μm of thickness, also the ceramic was metallized with 0.40 μm layer of Titanium and 15 μm layer of Silver. A brazing temperature of 830 °C was reached and maintained for 10 min, a cooling down a ramp of 3-5°C min was applied.

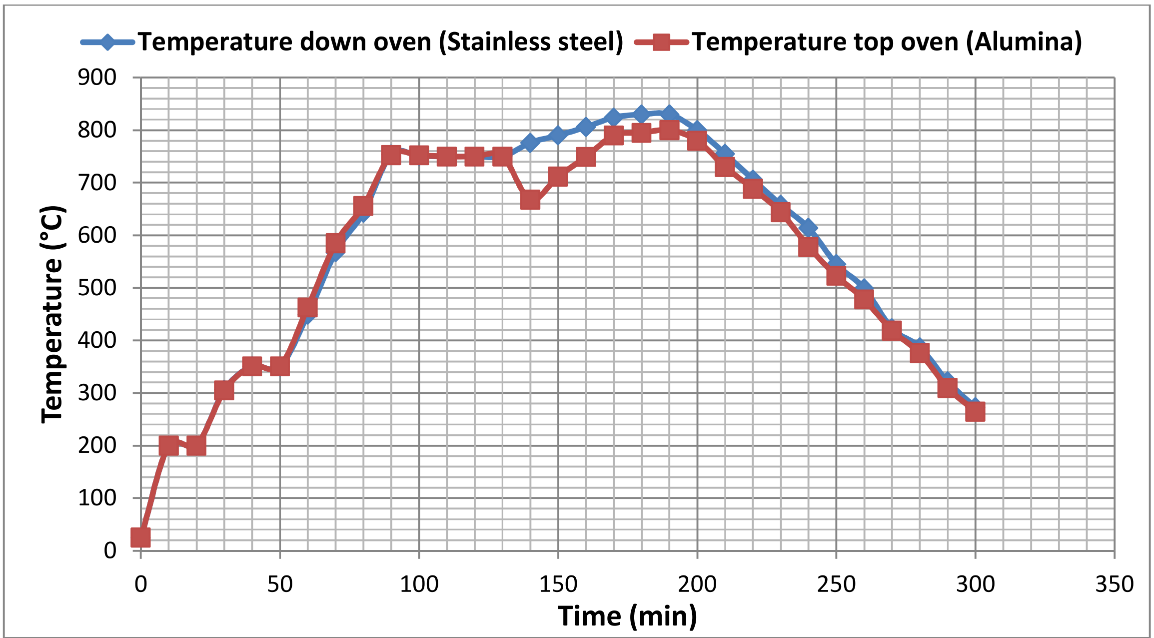


Figure 4.17: Profile of temperature in Stainless steel- Ti&Ag metallized Alumina brazing process with copper foil.

A scanning electron microscope (SEM) image of the sample described above is shown on figure 4.18a. A uniform distribution of the filler metal could be observed but empty spaces could be seen in the interlayer, on the other hand, figure 4.18b and 4.18c; show the cross-section of samples in which a lower amount of filler metal was used, empty spaces and gaps could be observed.

An EDS-EDX analysis on the cross-section of the sample in figure 4.18a reveals elements of the filler metal as Titanium, silver and copper on the metal nearby to the junction area on the cross-section on the samples in Figure 4.18b and 4.18c reveals no content of filler metals in the stainless steel nearby to the junction area, this fact could be evidence of poor diffusion in this area.

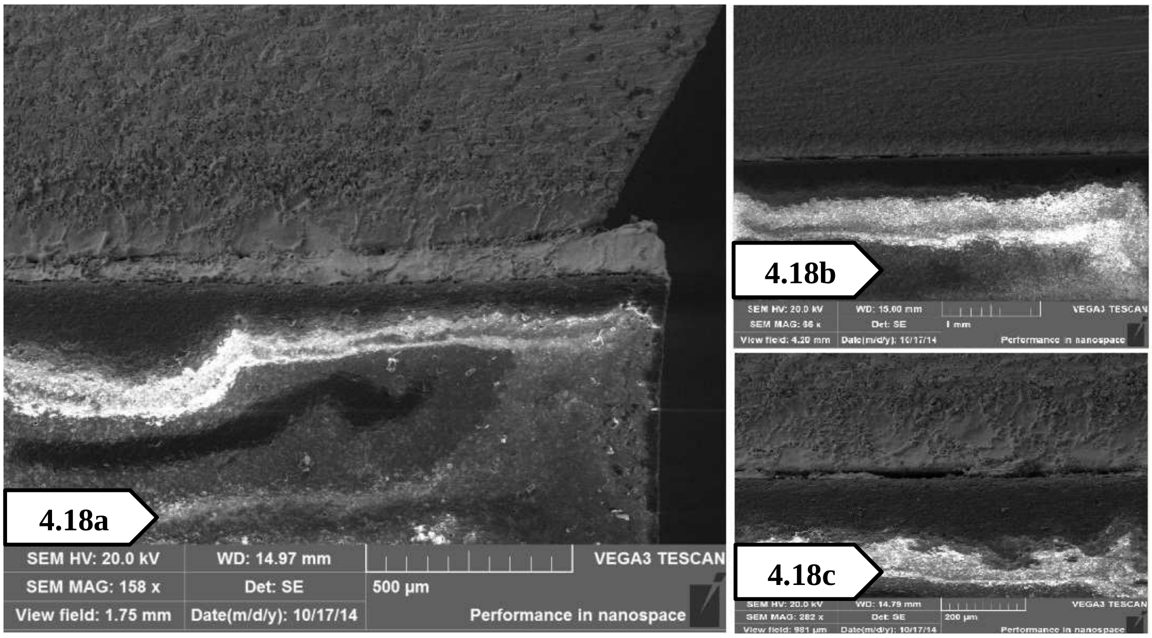


Figure 4.18: SEM image of stainless steel- Ti&Ag metallized alumina brazed cross-section with copper foil.

4.2.4. Experiment 8: Inconel- Ti&Ag metallized Alumina with copper:

Samples of Inconel and Alumina were selected as base materials; copper discs were used as filler. The foil was applied between the faying surfaces of the brazements.

Two films one of titanium and other of silver were coated on ceramic surface following the procedures 2 explained in section 3.1.4. The metallic pieces and the copper disc were cleaned following the procedures explained in section 3.1.5. The ceramic was cleaned before the metallization. The pieces were assembled fallowing the procedure explained in section 3.1.6.

The figure 4.19a shows the profile of temperature of a brazing process using a copper foil with 28 mm of diameter and 8~10 μm of thickness, also the ceramic was metallized with 0.40 μm layer of Titanium and 15 μm layer of Silver. The process started with a pressure of $6.2x10^{-6}mbar$, then the chamber was refilled with Argon gas till achieve a pressure of $4.2x10^{-3}mbar$. A brazing temperature of 893 °C was reached and maintained for 10 min, a cooling down a ramp of 10-15°C min was applied.

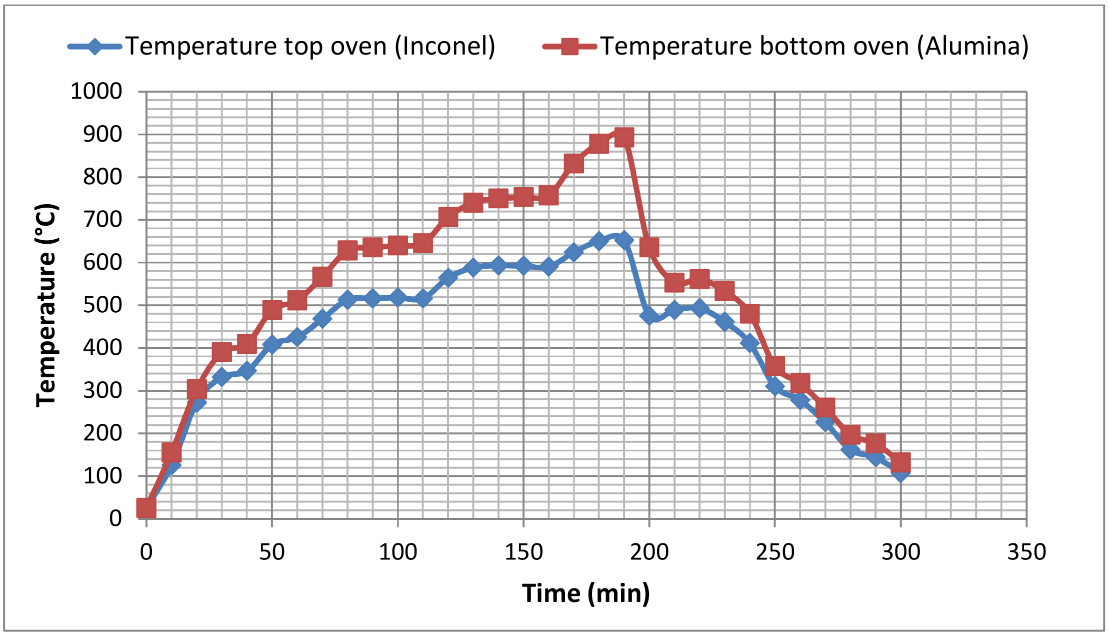


Figure 4.19: Profile of temperature in Inconel- Ti&Ag metallized Alumina brazing process with copper foil.

A scanning electron microscope (SEM) image of the sample described above is shown on figure 4.20a. In the cross-section could be seen a uniform distribution of the filler metal and no empty spaces could be observed. On the other hand, Figure 4.20b and 4.20c show the cross-section of samples in which higher pressure were used, empty spaces and gaps could be observed.

An EDS-EDX analysis on the cross-section of the sample in figure 4.20a reveals content of filler metals as titanium, silver or copper in the inconel nearby to the junction area.

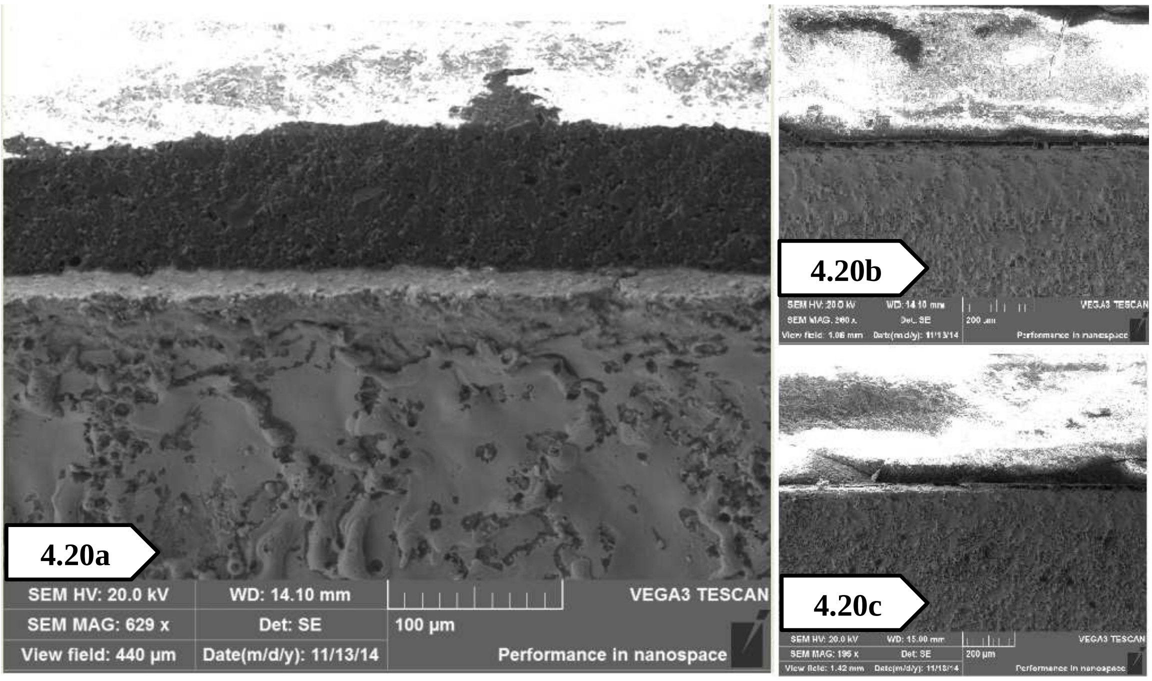


Figure 4.20: SEM image of inconel- Ti&Ag metallized alumina brazed cross-section with copper foil.

In these experiments we observed the same phenomenon in experiments with Pallabraze 850, there was presence of filler metal elements inside the metal component but in the cases in which the pressure was higher than $x10^{-6}mbar$ a gap or groups or gaps were observed, unlike the case when the chamber was refilled with argon gas and the gap was not observed.

5. Conclusions:

The aim of these studies was to obtain a procedure that leads to achieve a successful Metal-ceramic brazing process in a controlled environment, including the most important phases as the filler metal selection (active or no-active), filler metal shape (foil, disc or wire), the metallic component (Inconel or stainless steel), cleaning procedure, the heating source, etc.

For lap joints, the application of filler-metal on wire form do not seem like the most appropriate, due it is more difficult for the motel filler to spread over the complete interlayer surface, most of the residual stress, result of thermal expansion difference, tends to concentrated on the area near to wire location and consequently the ceramic pieces tends to break.

The amount of brazing filler metal has a key influence in the final brazed piece quality, it seems that using a brazing filler metal amount much lower than the necessary to achieve a successful brazed piece could cause the appearance of empty spaces in the interlayer, this could be a consequence of non-uniform distribution of the molten alloy due a weak capillarity action. On the other hand, an excess amount of filler can cause spillage of the same.

In order to avoid cracks on the ceramic, due residual stress, heating ramps of 10-15 °C /min and cooling down ramp of 3-5 °C/min showed the best results. In fact, when faster ramps of temperatures were applied some cracks were observed in the ceramics. Also the time on brazing temperature has an influence in this behavior, times of 10 min showed the best results.

The amount and size of empty spaces on the interlayer were significantly reduced when the chamber was filled with argon gas till achieve a pressure around $4x10^{-3} mbar$.

The most uniform distribution in the interlayer and diffusion of the filler metal into the brazed materials were observed when a Pallabraze 850 disc of 0.01 mm of thickness was used as filler metal and Inconel and Metallized alumina with 1μm of Titanium were selected as base materials for a brazing process with Argon atmosphere.

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ANNEXES

Material	CTE (mm/m/°C)		Young's Modulus (GPa)		0.2% YS (MPa)	
	20°C	800°C	20°C	800°C	20°C	800°C
Stainless steel 304	15	18	200	128	254	90
Inconel 600	13	15	214	164	280	100
Alumina	7	8.5	366	366^a	-	-

Physical properties of base materials at 20°C to 800°C.

Material	CTE (mm/m/°C)	Young's Modulus (GPa)	0.2% YS (MPa)	Density (g/cm^3)
Copper	19.4	125	69	8.9
CUSIL-ABA	18.5	83	271	9.8
Pallabraze 850	18.5	-	327	10

Physical properties of filler metals.