

# Seminar on Vacuum Technology and Processes for Metallurgical Applications



By  
Prof. S. K. Karak



**A Short Term Course  
On Vacuum Technology and  
Process Applications  
on 11/11/11-21/11/11  
I.I.T Kharagpur**

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## What is Vacuum ?

The atmospheric air around us is said to contain nearly  $2.5 \times 10^{19}$  molecules for every cubic centimeter space. Any given space having molecular density less than this is said to be under "**Vacuum**" conditions.

**The technology dealing with the production of** such reduced-pressure environments using different scientific concepts is known as "**Vacuum Technology**".

Vacuum technology is fundamental to a range of scientific explorations and technological processes, extending from analyzing atomically clean surfaces at extremely low pressures of the order of  $10^{-11}$  Torr (**1 Torr = 1 mm Hg**) to **freeze drying** of foodstuffs at relatively high pressures in the range of  $10^{-1}$  Torr. Vacuum conditions between these extremes are required for TV tube production, vacuum metallurgy, vacuum coating, semiconductor processing, particle accelerators, space simulation, etc.

## Unit of Vacuum ?

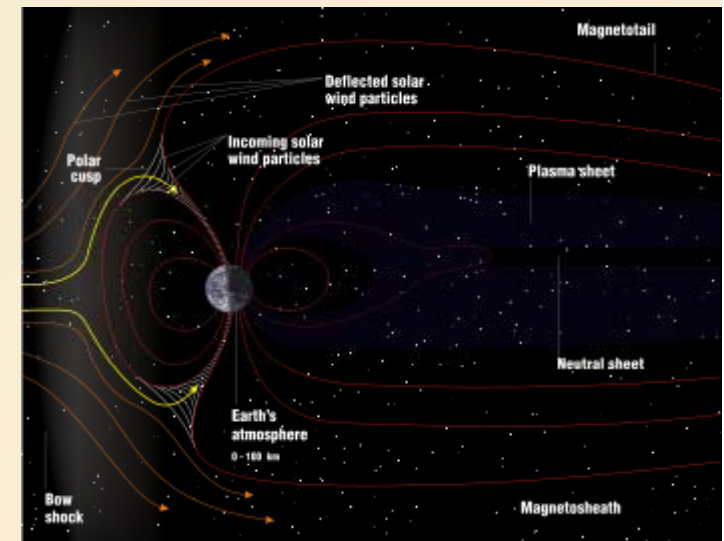
Vacuum is basically measured in pressure units. In a coherent unit system the pressure in a chamber is defined as the force acting on unit surface area and its dimensional formula is  $[L]^{-1} [M] [T]^{-2}$ , where L, M, and T represent the base units of length, mass and time respectively. In SI units, the unit of pressure is Newton/m<sup>2</sup> or Pascal, and in the CGS system of units it is dyne/cm<sup>2</sup>. The standard atmosphere is defined as the pressure exerted by a column of mercury 760 mm high. In recent scientific literature, this standard is often expressed in Torr and mbar, which are non-coherent pressure units, but widely used.

1 Torr = 1 mm Hg = 1/760 atm.

1 mbar = 100 Pascal = 1/1013 atm.

## Classification of Vacuum:

	Pressure (Torr)	Pressure (Pa)	Pressure (mbar)
Atmospheric pressure	760	101.3 kPa	1013 mbar
Low vacuum	760 to 25	100 kPa to 3 kPa	1000 – 30 mbar
Medium vacuum	25 to $1 \times 10^{-3}$	3 kPa to 100 mPa	30- $1 \times 10^{-3}$ mbar
High vacuum	$1 \times 10^{-3}$ to $1 \times 10^{-9}$	100 mPa to 100 nPa	$1 \times 10^{-3}$ to $1 \times 10^{-9}$ mbar
Ultra high vacuum	$1 \times 10^{-9}$ to $1 \times 10^{-12}$	100 nPa to 100 pPa	$1 \times 10^{-9}$ to $1 \times 10^{-12}$ mbar
Extremely high vacuum	$< 1 \times 10^{-12}$	$< 100$ pPa	$< 1 \times 10^{-12}$ mbar
Outer Space	$1 \times 10^{-6}$ to $< 3 \times 10^{-17}$	100 $\mu$ Pa to $< 3$ fPa	



Based on different physical characteristics of vacuum, we can achieve different industrial/scientific applications. For example, vacuum in a chamber allows us to have controlled sub atmospheric pressures, useful for several mechanical operations and controlled flow of gases in specified direction.

### **Industry Product of scope**

#### **1. Electrical/Electronics:**

Gas-filled tubes, electron tubes, TV picture tubes, X-ray tubes, capacitors and transistors, rectifier tubes, cathode ray tubes, fluorescent tubes, switching tubes, interrupters, photocells, gas discharge lamps, incandescent lamps.

#### **2. Scientific instruments:**

Maintaining the vacuum in electron microscopes **FESEM, SEM, TEM, HRTEM**, mass spectrometers, surface analysis systems, oscilloscopes, thermoflasks, and thermometers.

#### **3. Mechanical operations:**

Railway braking, industrial filtering, vacuum sniffers, vacuum cleaning, holding /lifting /transporting solids and liquids.

4. Refrigeration, cryogenic engineering Insulation of vessels and tanks for liquid petroleum gases, cryogenic liquids.

5. Automobile Filling of air-conditioning, cooling and servo systems.

6. Research and development **Space simulation chambers**, bubble chambers, fusion experiments, fusion reactors, particle accelerators, ion implantation systems, thermal insulation, cryogenic experimentation

# Applications in Vacuum Technology in Industry

Sl. No	Industry	Product of Scope
1	Mechanical operations	Railway braking, industrial filtering, vacuum snifters, vacuum cleaning, holding/lifting/transporting solids and liquids.
2	Refrigeration , cryogenic engineering	Insulation of vessels and tanks for liquid petroleum gases, cryogenic liquids.
3	Electrical/Electronics	Gas-filled tubes, electron tubes, TV picture tubes, X-ray tubes, capacitors and transistors, rectifier tubes, cathode ray tubes, fluorescent tubes, switching tubes, interrupters, photocells, gas discharge lamps, incandescent lamps.
4	Scientific instruments	Maintaining the vacuum in electron microscopes, mass spectrometers, surface analysis systems, oscilloscopes, thermoflasks, and thermometers.
5	Automobile	Filling of air-conditioning, cooling and servo systems.
26	Research and development	Space simulation chambers, bubble chambers, fusion experiments, fusion reactors, particle accelerators, ion implantation systems, thermal insulation, cryogenic experimentation.



## Applications in Metallurgy

- High vacuum treatment of metals and their alloys susceptible to oxidation and/or gas.
- Vacuum cast forging ingots. Vacuum degassed alloyed steels and vacuum degassed cast steel

### Production of Pure Metal and alloys

Production of pure metals such as bismuth, selenium, zinc, antimony, magnesium, zirconium and titanium

### Equipment s or Furnace Requirement

Vacuum Induction Melting and casting  
**(VIM/VID/VIDP)**

#### Casting and Coating:

Vacuum Induction Melting – Investment Casting (VIM-IC)

Vacuum turbine blade coating (EB/PVD)

#### Remelting or Secondary Processes:

Electro Slag Remelting (ESR)

Vacuum Arc Remelting (VAR)

Electron Beam melting (EB)

#### Special Furnaces:

Hot Isothermal Forging (HIF)

Vacuum Induction melting Gas Atomizer (VIGA)

Electrode Induction melting Gas Atomizer (EIGA)

Induction heated Quartz tube furnace (IWQ)

High vacuum resistance furnace (WI)

# Requirement of Vacuum Technology in Powder Metallurgy

Powder Metallurgy (PM) is the innovative and environmentally friendly process of producing component parts using metal powder.

## Production of Pure Metal and alloys

Manufacture of metal powders, such as **Ferrous, Bronze, Brass, Copper, Tungsten and Hard metal powders.**

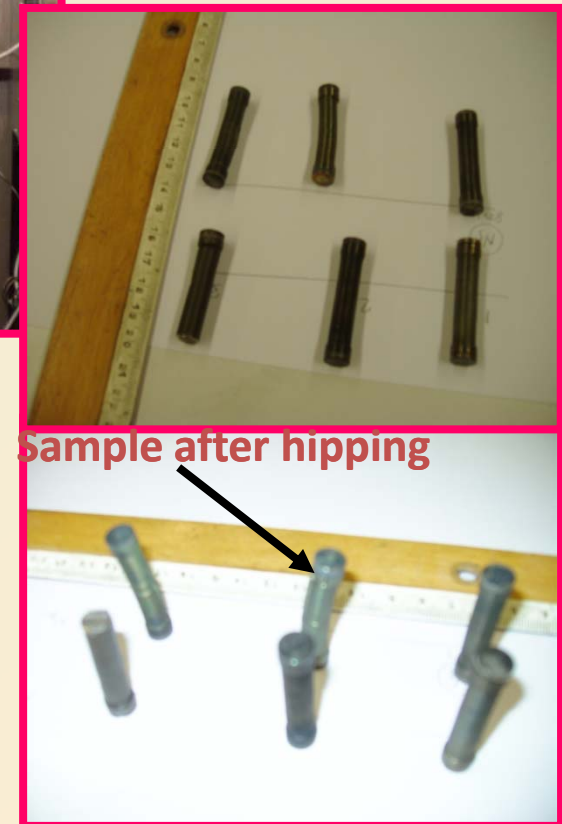
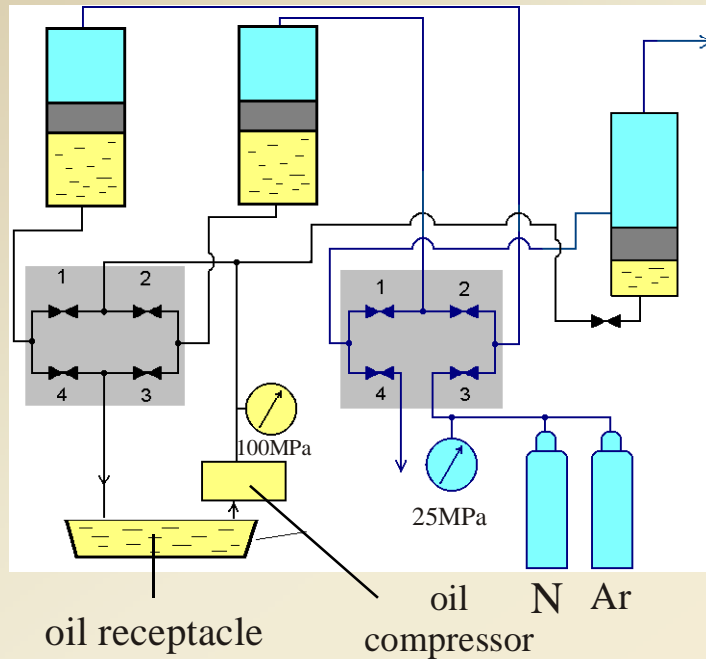
## Equipments , Pressing units or Furnace Requirement

Furnaces/sintering kilns, vacuum or atmospheres, **Hot Pressing, Hot Isostatic Pressing (HIP), Spark Plasma Sintering (SPS), Pulse Plasma Sintering(PPS)**, equipment and optional manufacturing steps, sizing, repressing, metal infiltration and optional finishing steps, heat treatment and plating.



# Hot Isostatic Pressure (HIP)

## Working Chamber

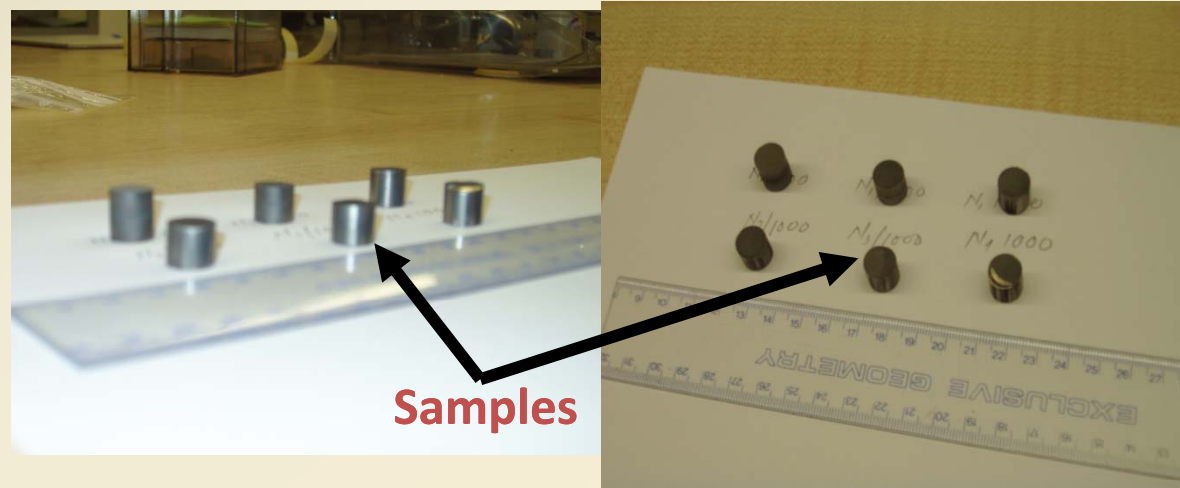
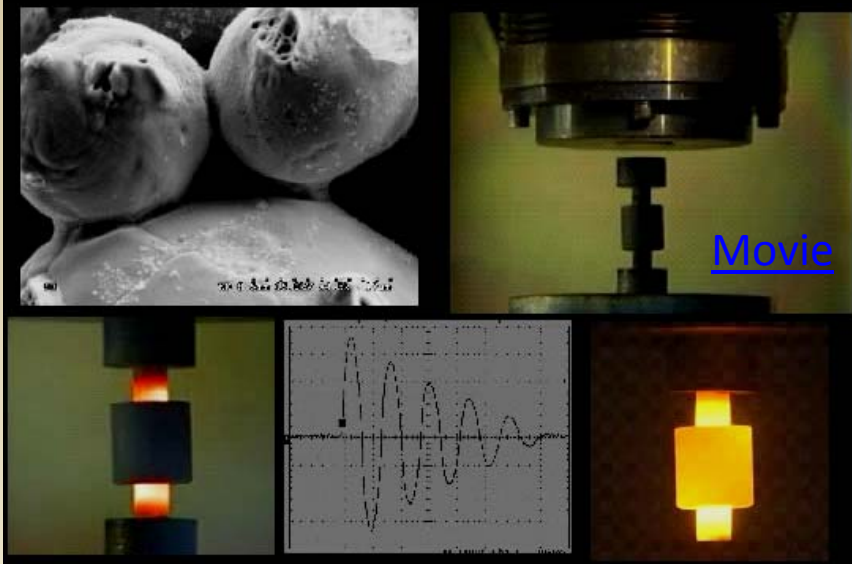


## HIP Parameters:

- Diameter,  $d = 10 \text{ mm}$ , Length  $l = 30 \text{ mm}$
- Pressure,  $P = 1.5 \text{ GPa}$
- Temperature,  $T = 600^\circ\text{C}, 800^\circ\text{C}, 1000^\circ\text{C}$
- Time,  $t = 30 \text{ min}$
- Atmospheres argon gas



# Pulse Plasma Sintering (PPS)



- Chamber 600x600x600 mm
- Vacuum  $10^{-6}$  mbar
- Hydraulic press :
- Pressure 50 kN
- Stroke of press 200 mm

## HIP Parameters:

- Diameter,  $d = 15$  mm, Height,  $h = 12$ mm
- Pressure,  $P = 50$  kN
- Temperature,  $T = 600^{\circ}\text{C}, 800^{\circ}\text{C}, 1000^{\circ}\text{C}$
- Time,  $t = 5$  min

# Requirement of Vacuum Technology for Brazing , Diffusion Bonding and Brazing Alloy Development

**Professional Experience  
In NFTDC,Hyderbad**

# ADVANCED JOINING TECHNOLOGY APPLICATION LABORATORY

AJTAL

## OUR GROUP MEMBERS

Swapan Kumar Karak

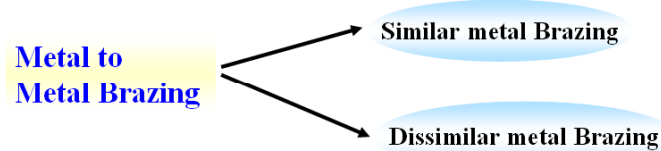
M. Govindaraju

*Non-Ferrous Materials Technology Development Centre  
Hyderabad, INDIA*

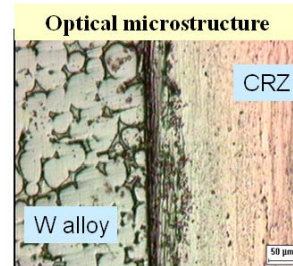
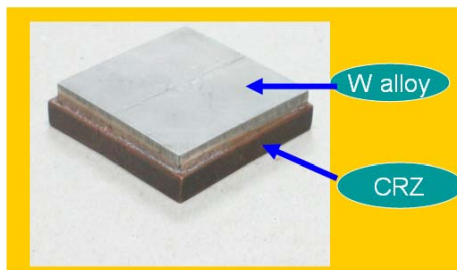
## FACILITIES

	Max. Temp (°C)	Hot Zone dimension	Vac. Level (mbar)
<b>High Vacuum High Temperature Furnace</b>	1600 ± 0.3	300mm×300m m×300mm	10 <sup>-5</sup> 10 <sup>-6</sup>
<b>High Vacuum Annealing Furnace</b>	800 ± 5	Φ 300mm ×800mm length	10 <sup>-5</sup>
<b>High Vacuum Rapid Experimentation Furnace</b>	1100 ± 2	Φ 100mm ×150mm length	10 <sup>-5</sup>

## Vacuum Brazing

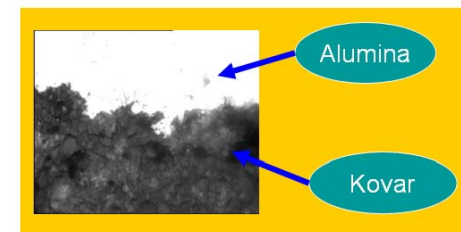


Brazed Materials: SS, CuCrZr, W alloy, Super alloy, PHSS etc.



## Active Brazing

Ceramic to Ceramic	Alumina-Alumina
Ceramic to Metal	Alumina-SS, Alumina-Kovar, Alumina-Cu, Alumina-Ti
Refractory metal to non-metal	Mo-Graphite
Non-metal to non-metal	Graphite-Graphite

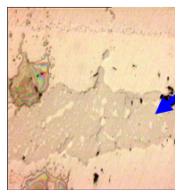


## Challenging Work

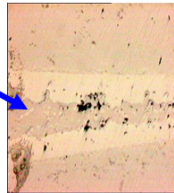


## Super alloy Brazing

Filler Material: *Microbraz* alloy



Filler



*Microstructure of the brazed joint*

High Temperature Tensile Testing Results

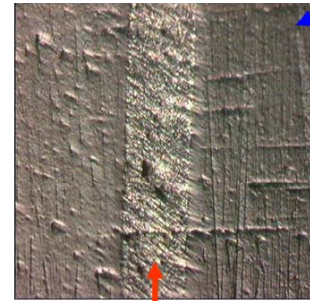
Testing Temperature: **870° C**

In Argon Atmosphere

UTS Value at brazed joint:  
**513MPa**

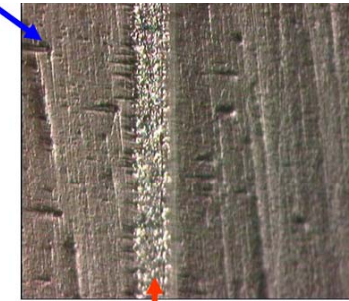
## Ti alloy Brazing for Cryogenic Application

Material: **Ti-5Al-2.5Sn**



Joint with **100 μm**  
thick Filler Material

Filler Material:  
*Silver based*



Joint with **50 μm**  
thick Filler Material

Microstructures of brazed Ti alloy joint

## Diffusion Bonding → Cu-SS and Super alloy

### Development of Materials

- ✓ Active Brazing Alloys (**Ag-Cu-Ti** and **Cu-Al-Si-Ti**)
- ✓ Vacuum Brazing Alloys
- ✓ UHV Components and Brazing Alloys

### Development of Processes

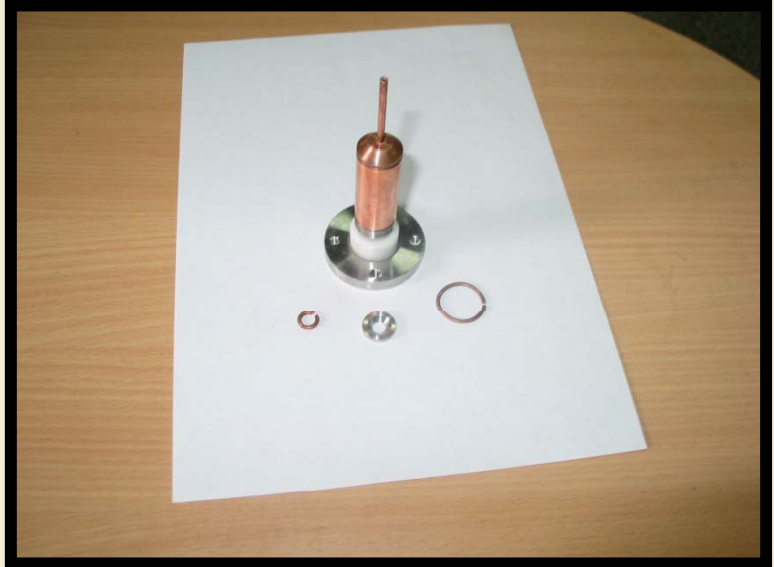
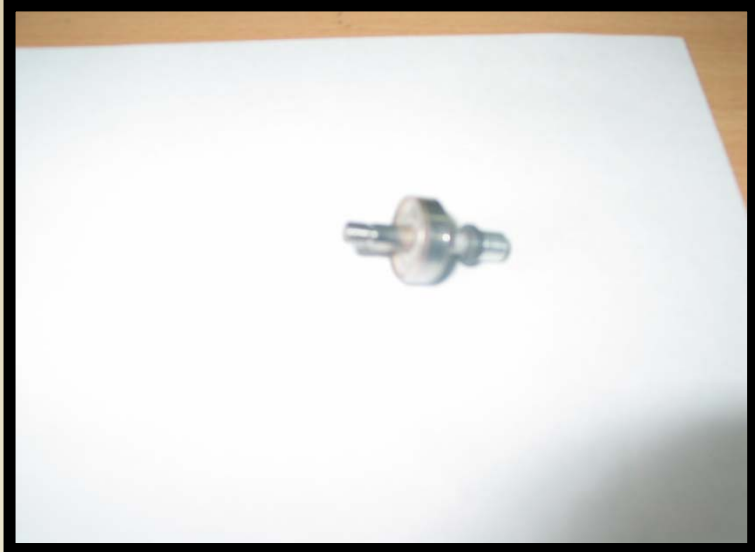
Heat Treatment, Thermal Treatment, Melting and Sintering

### Design Consultancy

- ✓ High Vacuum Quenching Blower Motors
- ✓ Materials for UHV Systems
- ✓ High Vacuum Furnaces
- ✓ Diffusion Bonding SET-UP



# Brazing of Plasma Discharge Tube







**Vacuum annealing of Columbium**

**Temp-1225°C**

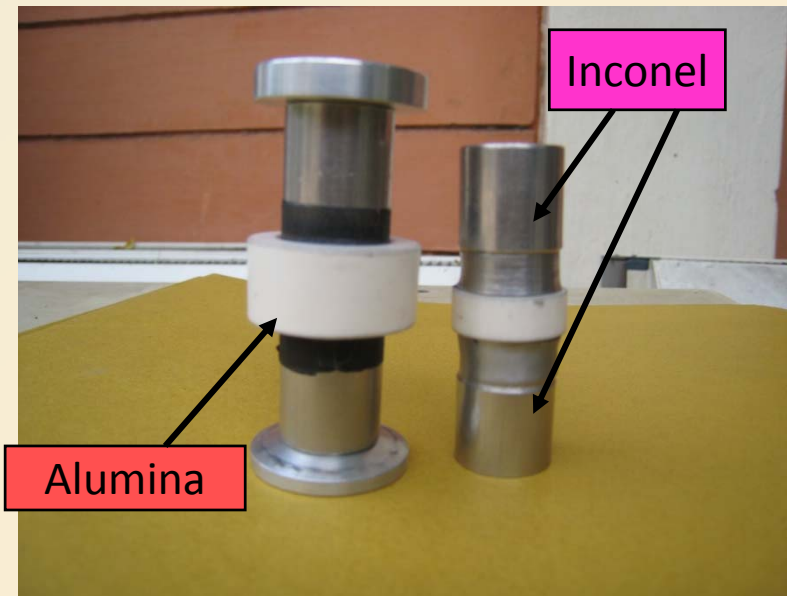
**Holding Time-2hrs**

**Brazing of Inconel with Alumina**

**Temp-1050 ° C**

**Time-25min**

**Filler Material-CuABA**

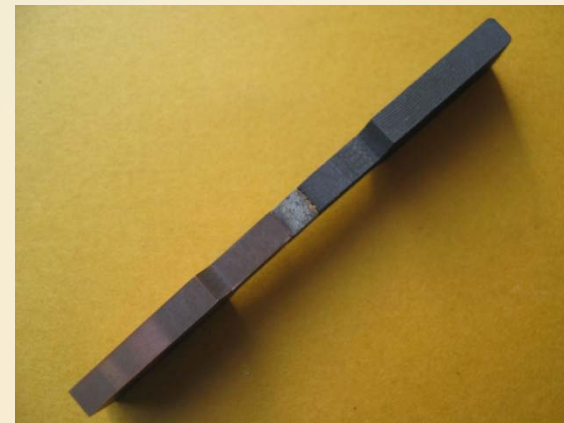
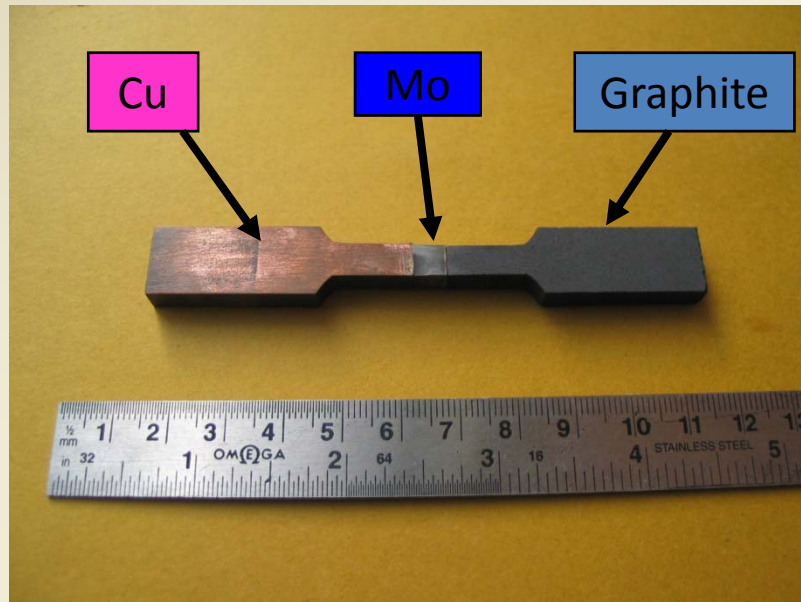


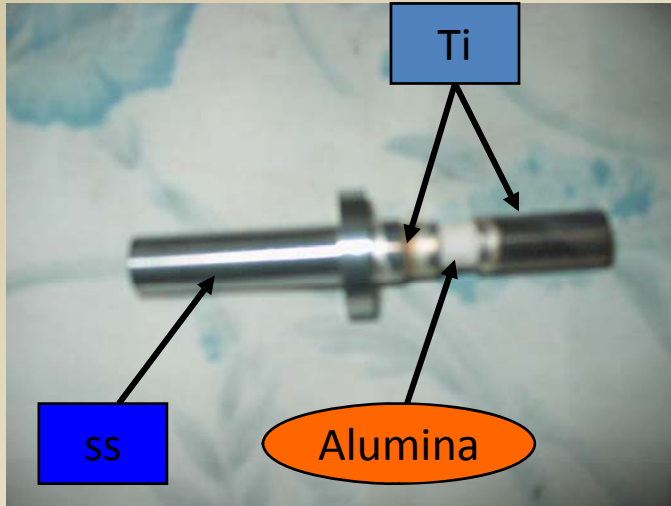
## Inter-Layer Brazing

Temp-975

Time-25min

Filler Materials-Cu-Mn-Ni





## Brazing of Alumina with Ti +SS

Temp-843.5

Time-2 min

Filler Material-Ag-Cu-Ti



## Major Developments:

- ❖ *Inter layer brazing of CRZ, Mo and Graphite.*
- ❖ *Brazing of Ti-Al<sub>2</sub>O<sub>3</sub>-SS component in Lab scale.*
- ❖ *Brazing of Ni based super alloy and high temperature Tensile at brazed joint.*
- ❖ *Brazing of CRZ with W alloy.*
- ❖ *Brazing of Kovar with Alumina.*
- ❖ *Brazing of Graphite with Mo.*
- ❖ *Brazing of Inconel with Alumina*
- ❖ *Development of filler materials-Ag-Cu eutectic, Ag-Cu-Ti alloy, Ag-Cu-Zn alloy, Pd-Ni alloy, Cu-Mn-Ni alloy*

Thank you