CANDLE Synchrotron Research Institute

(Center for the Advancement of Natural Discoveries using Light Emission)

Vacuum Systems and Metal-Ceramic Joints in Advanced Accelerators

A Thesis
Submitted for the degree of Doctor of Philosophy (PhD) of Technical Sciences in division of
''Charged Particle Beam Physics and Accelerator Technology'' 01.04.20

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Scientific Supervisor – Doctor of Technical Sciences - Vardan Sh. Avagyan

Yerevan 26.10.2017

The modernity and importance of the problem:

The success of the design and creation of new generation accelerating technology mainly depends on the material choice, the technologies of basic joint generation, the exploitation features and the dependence of temperature-temporal characteristics connected to the charged particle beam power, etc.

For example, AREAL (Advanced Research Accelerator Laboratory) laser driven RF gun based linear accelerator was operated in Armenia. It provides 2-5 MeV energy and 10-100 pC bunch charge for electron impulses. For the creation of free electron laser in AREAL accelerator, it is planned to increase the beam energy up to 20-50 MeV.

Depending on the commissioning conditions of metals and metal-ceramic joints in accelerators high requirements are made for the following parameters: mechanical strength, vacuum tightness, reliability, temperature and radiation resistance, etc.

Purposes and main problems of the thesis:

The main purposes of this thesis are fabrication technology development for high quality vacuum tight ceramic-metal joints and system design for precise commissioning of accelerators under the condition of particle energy change in accelerators.

Main topics discussed in the thesis defence:

- Thermoregulation systems for electromagnetic vacuum systems of AREAL linear accelerator – thermoregulation system of RF gun, thermoregulation system of klystron and cooling system for solenoid magnet.
- New brazing method for difficult geometric shape ceramic and metal materials, based on inner and outer volume pressure differences of materials.
- New bonding method for bonding cylindrical ceramic and metal items based on local warming up and pressure effects.
- Vacuum RF window (brazing technologies) and RF window test stand design.

Scientific novelty :

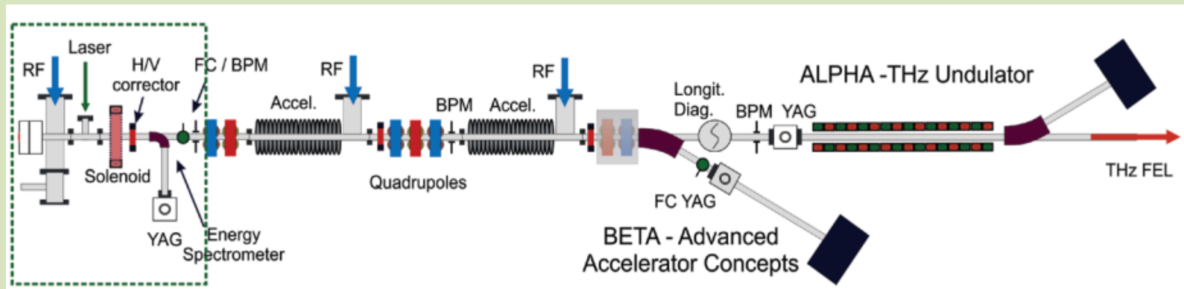
- New brazing method for difficult geometric ceramic and metal materials, based on inner and outer volume pressure differences of materials.
- New bonding method for bonding of cylindrical ceramic and metal items based on local warming up and pressure effects.

UHV and Thermoregulation systems of AREAL linear accelerator.

Electron Beam Energy

	Phase 1	Phase 2
Energy	5 MeV	50 MeV

Main scheme of AREAL Linear accelerator



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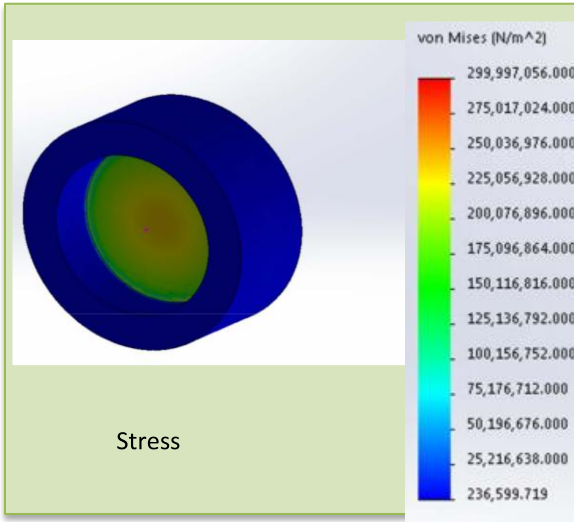
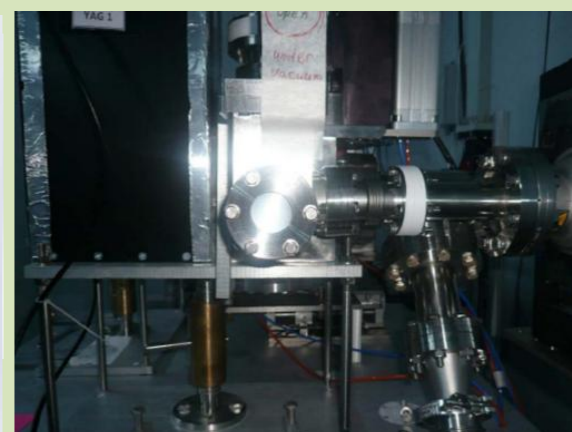
UHV System of AREAL

Achieved vacuum level – 2.3 10^{-9} Torr

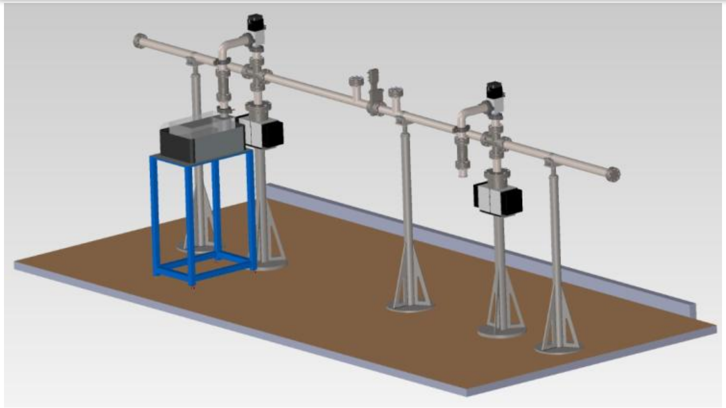


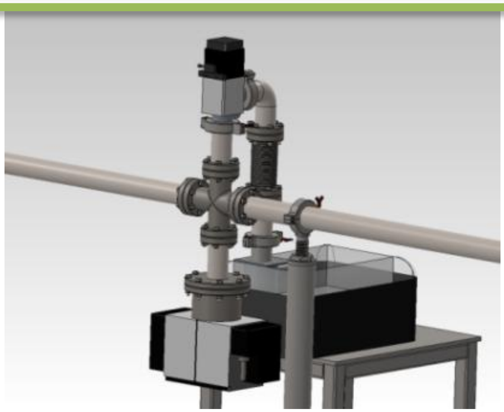
Stress



Ti E-beam windows

UHV test stand



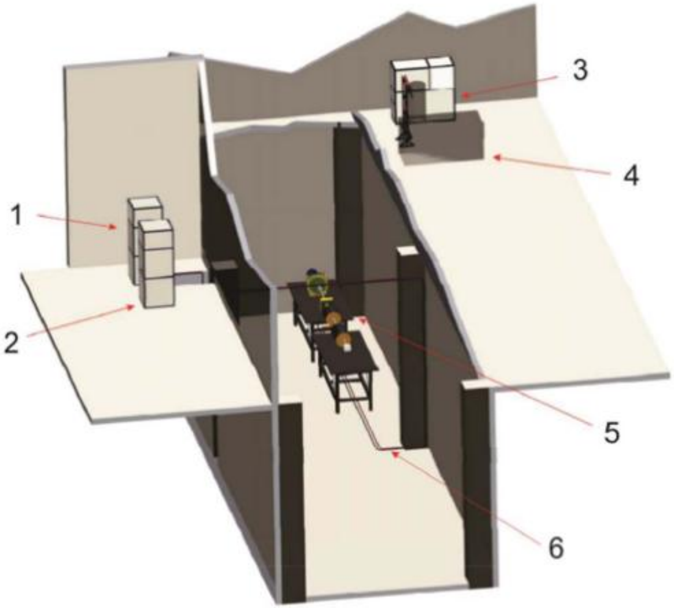


Main parameters

Vacuum level	Up to 10^{-10} Torr
Main construction material	304 stainless steel
Max. temperature	Up to 450 ^{0}C
Vacuum gauges	Inverted magnetron
	Penning
	Pirani
Vacuum pumps	Ion pumps
	TPS pump

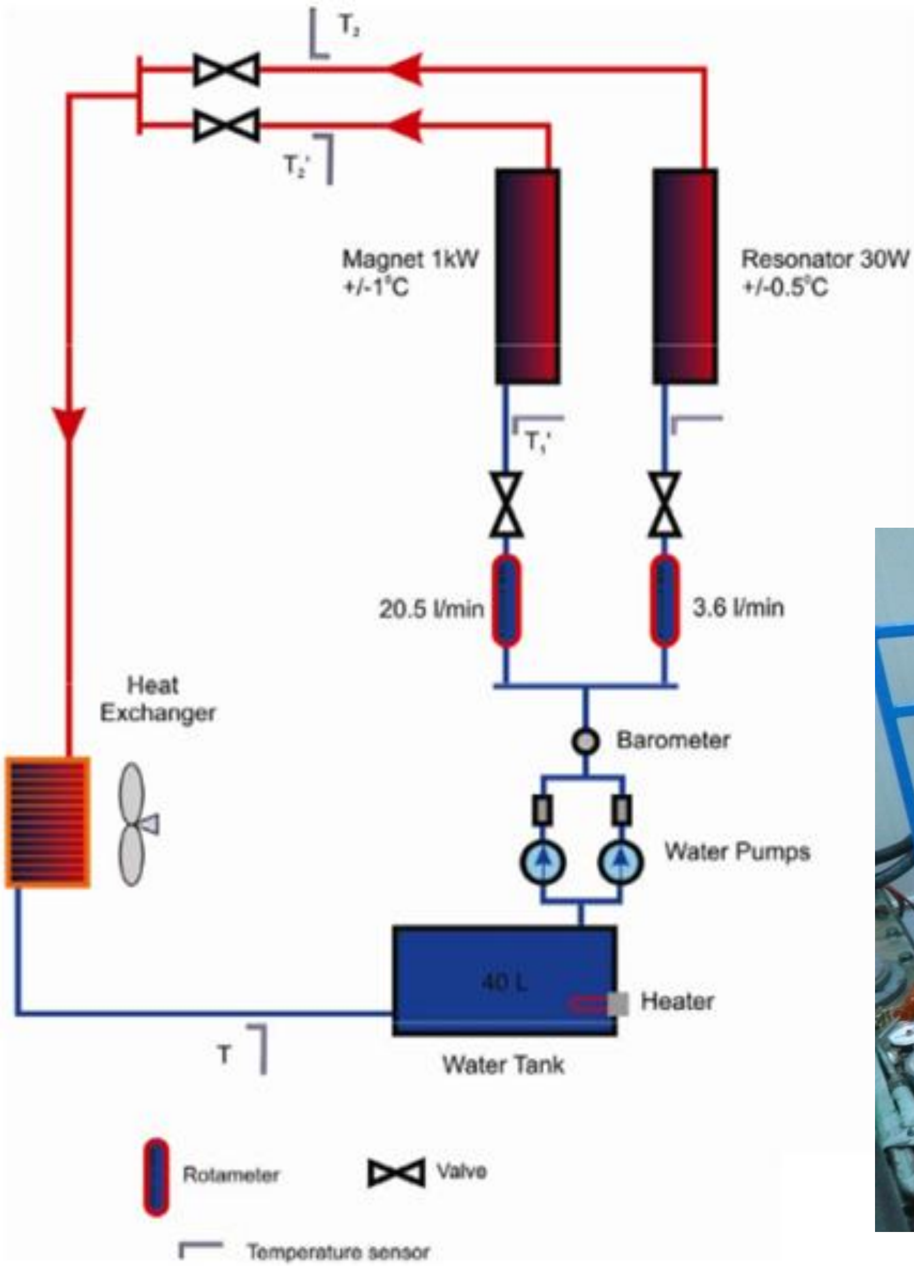
The main purposes of UHV test stand are testing of all UHV components, control systems, devices, vacuum-tight joints (metal-metal, metal-ceramic), in vacuum movers, bake out processes of UHV components, etc. before installation in the main acceleration system.

Thermoregulation systems for AREAL linear accelerator.



1. Thermoregulation system of RF gun,
2. Cooling system of solenoid magnet ,
3. Thermoregulation system of Klystron ,
4. Klystron,
5. Linear accelerator ,
6. Feed water pipes for RF gun and solenoid magnet – water input and output pipes).

Thermoregulation System of Klystron



Characteristics	Value	
	Resonator	Magnet
Cooling capacity (W)	500-1500	
Temperature range	30-55	
Temperature stability °C	+/-0.5	+/-1
Water flow rate (l/min)	3.64	20.5
Pressure (kg/cm²)	<4.2	
Temperature sensor	Pt100	
Coolant	Distilled water	
Water deionization range	50M Ω·cm	
Nominal pressure (bar)	3.3	

•Fast and precise thermoregulation systems have been designed and assembled for an effective and reliable operation of electromagnetic vacuum systems of AREAL linear accelerator. These include the thermoregulation system of the RF gun, the thermoregulation system of the klystron and the cooling system of the solenoid magnet. These thermoregulation systems are able to provide a temperature control with up to +/-0.5^{0}C accuracy.

•An UHV test stand has been designed and assembled for testing and investigating the components of the UHV system, as well as the metal-metal and metal-ceramic junctions in the UHV.

•An UHV system has been designed and assembled for an effective operation of electro-magnetic systems of AREAL linear accelerator.

Chapter 2. Experimental investigations of ceramic-metal brazing technologies for accelerators UHV systems.

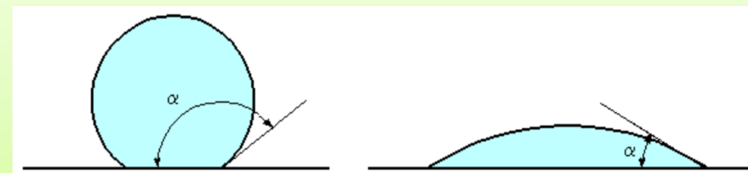
Materials for vacuum-tight ceramic-metal joints.

Review Part

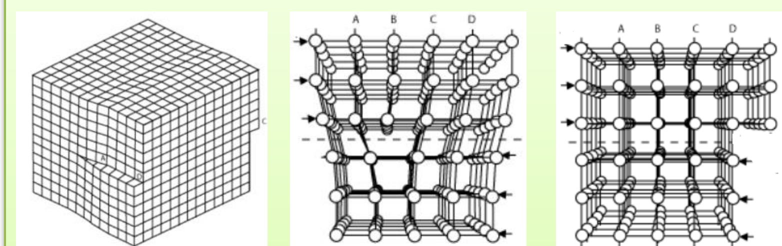
Main requirements for materials

- Low outgassing rate,
- Thermal shock resistance,
- Low material penetration,
- High mechanical strength,
- High weldability and brazability,
- High machinability,
- Reliable during long time,
- Repeatability and Dimentional stability,
- Eappropriate electro-mechanical characteristics,
- Absorbcion and desorbcion characteristics,
- Surface oxidation characteristics,
- Corrosion resistance,
- Metallization characteristics.

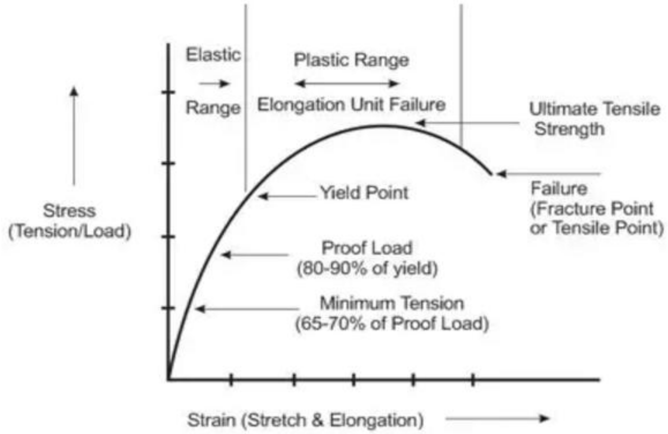
Solders and materials wettability



Materials structures - Homogeneity

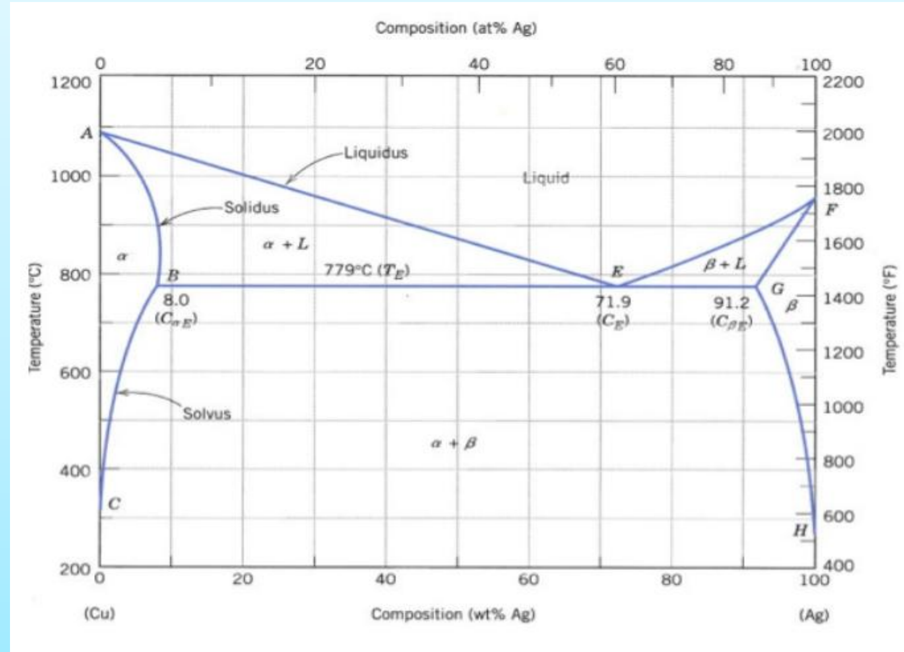


Without dislocations, content inhomogeneities, cracks, inner impurities, etc.

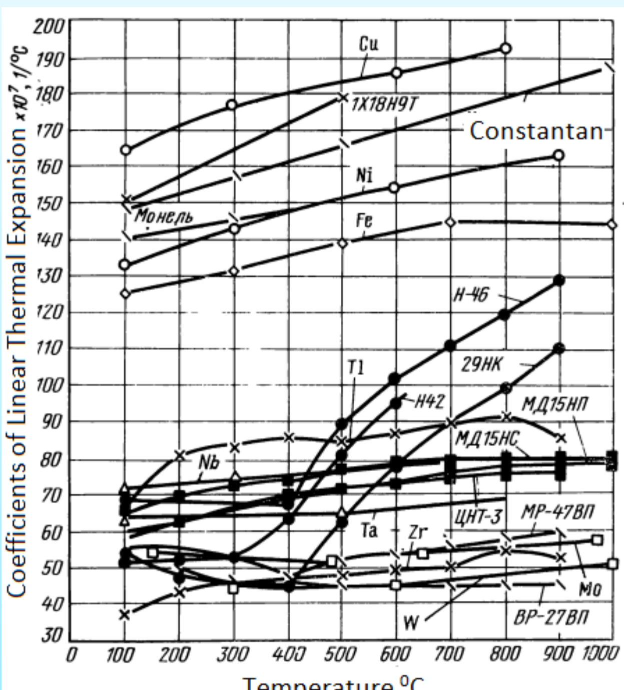


Tensile Stress-Strain Diagram

Phase diagram Cu-Ag



Thermal expansion of metals compared with Al2O3 ceramic



Selected materials

- Stainless steel 316;
- Cu
- Kovar
- 99.5% alumina
- 95 % alumina
- Mn, Mo, Ni, etc.

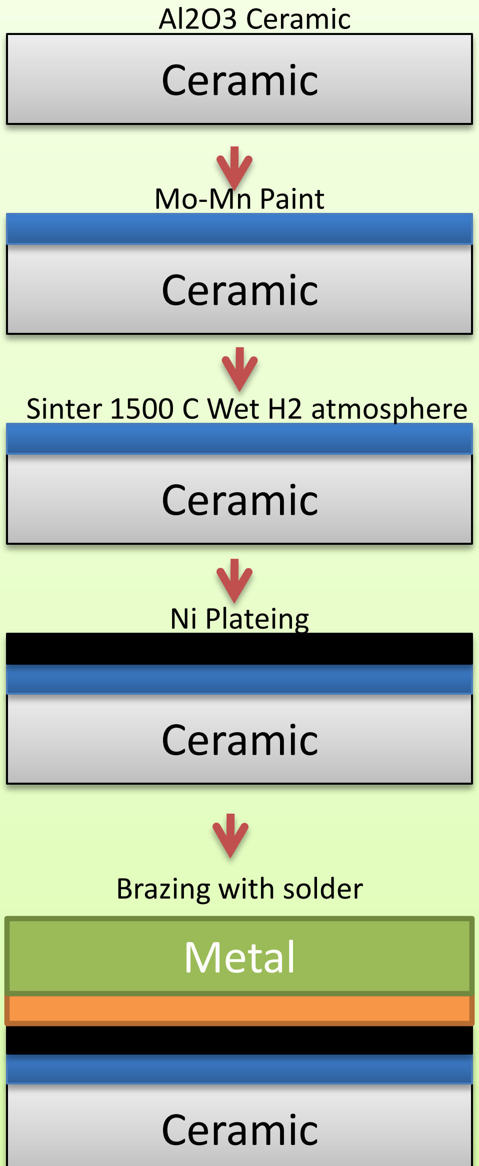
Main requirements for vacuum-tight ceramic-metal joints.

- High mechanical strength (wide range of temperature);
- Thermal shock resistance (numerous thermal cycles);
- Low outgassing level (corresponding bake-out, adsorption, desorption level);
- Low material penetration for gasses (H_2, Ar, N_2, H_2O, etc.);
- Corresponding electromagnetic features;
- High reliability and durability (geometry and structure stability during operation);
- Reproducibility – geometry, structure;
- Non-magnetic properties of brazed materials (depends on operational environment);

Bonding Technologies for Vacuum-tight Ceramic-metal joints.

- Active brazing technology;
- Brazing based on ceramic metallization technology;
- Thermo-compression bonding technology (diffusion welding);
- Gluing technics;
- Mechanical sealing technics.

1. Moly Manganese Metallization



2. Active brazing

3. Gluing technics

Molybdenum - Manganese Metallization

Ceramic - Type		Metallization Past	Concentration %
Alumina	22X, 22XC	Mo Mo : Mn Mo : Mn : Si Mo : Mn : TiH_2 Mn : Mo_2B_5, Mo	100 80 : 20 80:20 (+5) 80 :20 : 10 20 : 10-15 : 70-65
	A-995	Mo : Mn : Mo_2B_5 : БД-22	74 : 15 : 5 : 6
	Sapphirite	Mo : Mn : V_2O_5	75 : 20 : 5
Monocrystal	sapphire	Mo : Mn : Mo_2B_5 : БВ22	74 : 15 : 5 : 6
	Rubine	Mo : glass CT-1	70 : 30
Beryllium ceramic		Mo : Mn : Si	80 : 20 : (+5%)

Metallization process

Coating type	Temperature ^0C	duration	Cooling speed ^0C/min	Environment
Mn, Mo (22X, 22XC, A-995, M-7)	1 270 – 1 400	20 - 40	5 - 10	$N_2 : H_2 = 2 : 1$ $N_2 : H_2 = 3 : 1$ dew point +15 - +25^0C
Mo, Mn, Si	1 280 – 1 320	40	Cooling - 4.2	$N_2 : H_2 = 3 : 1$ dew point +15 - +25^0C

Electrochemical Ni plating

Laboratory equipment

Vacuum Furnace

Max. temperature	2000 ^{0}C
Vacuum level	10^{-6}Torr
Heater	Tungsten
Power	35KW
Working chamber size	150/150/460mm
Chamber cooling	Water
Chamber shielding	Molybdenum
Heater voltage increasing and decreasing	By hand

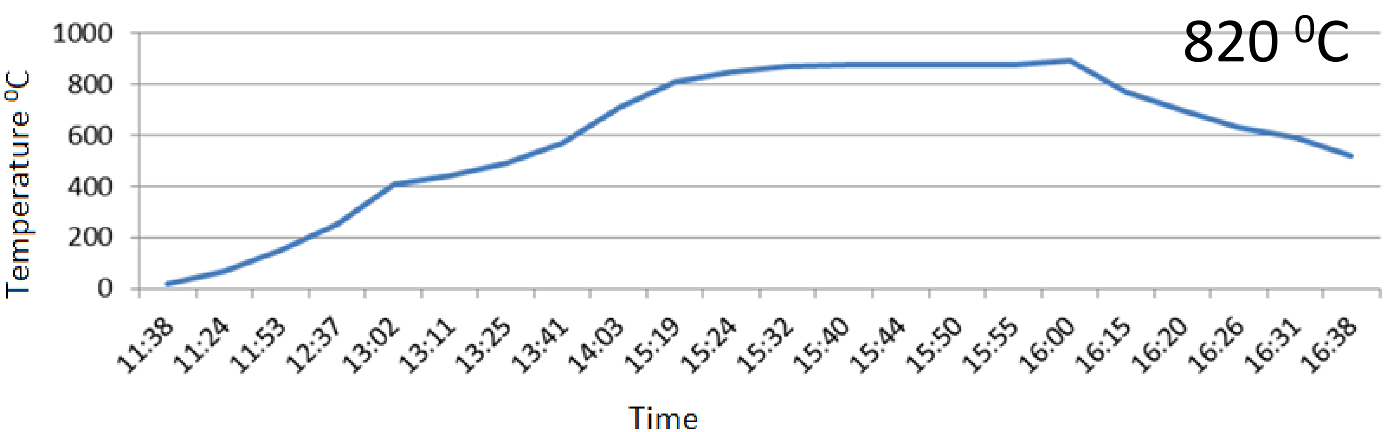
Ceramic machining system

Mixing machine (metal powders)

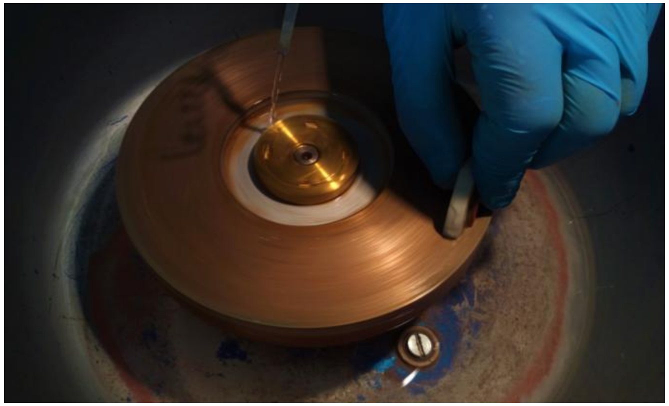
Polishing machine

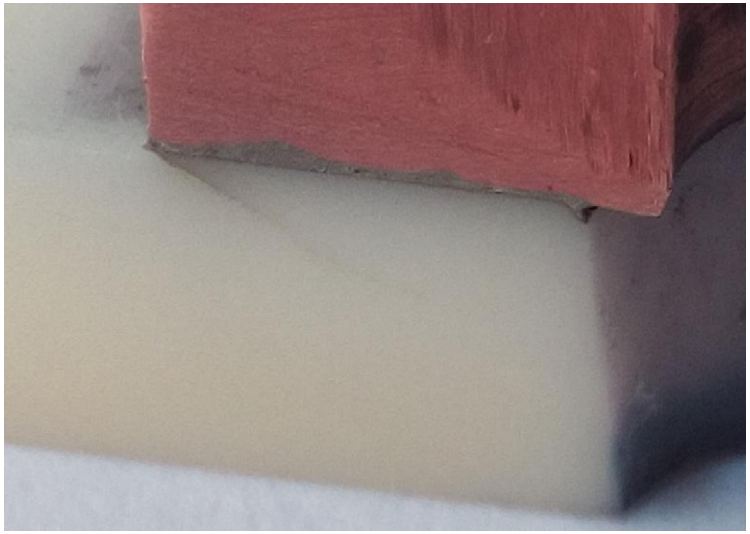
Metallurgical Microscope

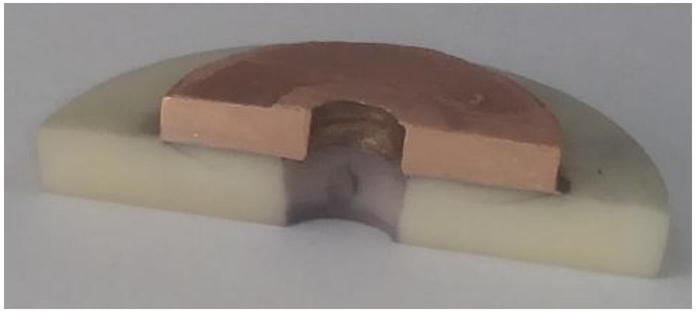
Experimentation: Active Brazing Technology



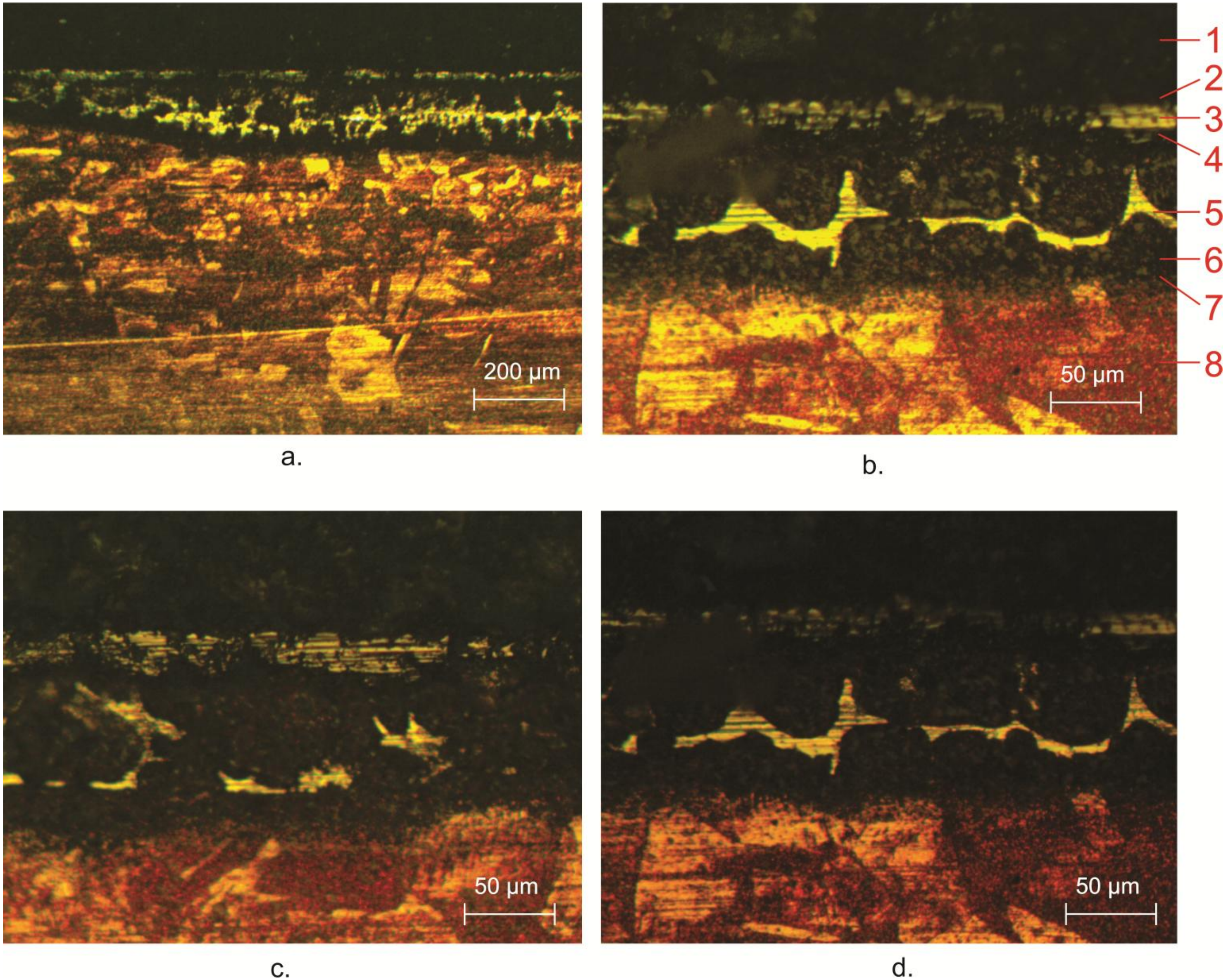
Temperature dependence on time for brazing process





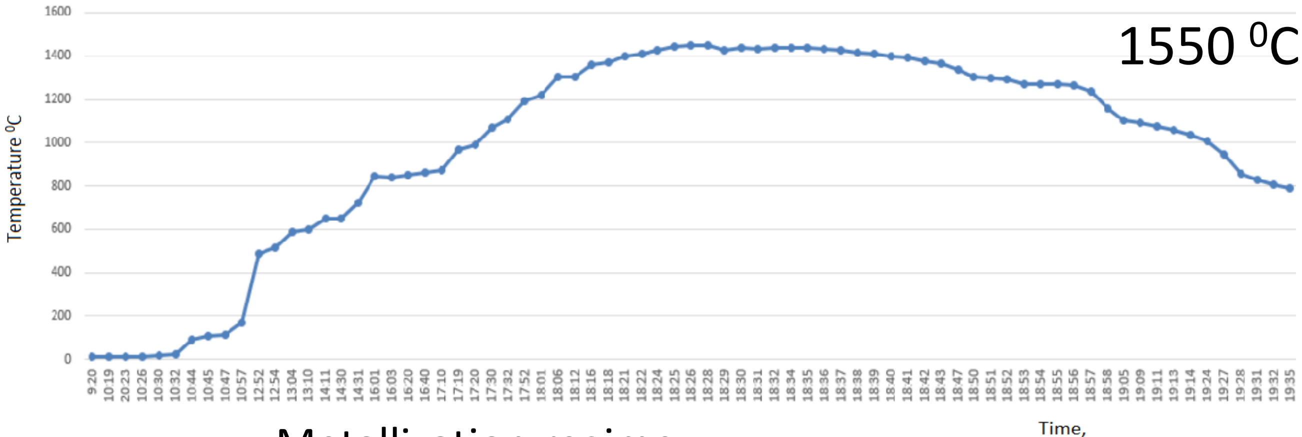


Alumina-Cu joint



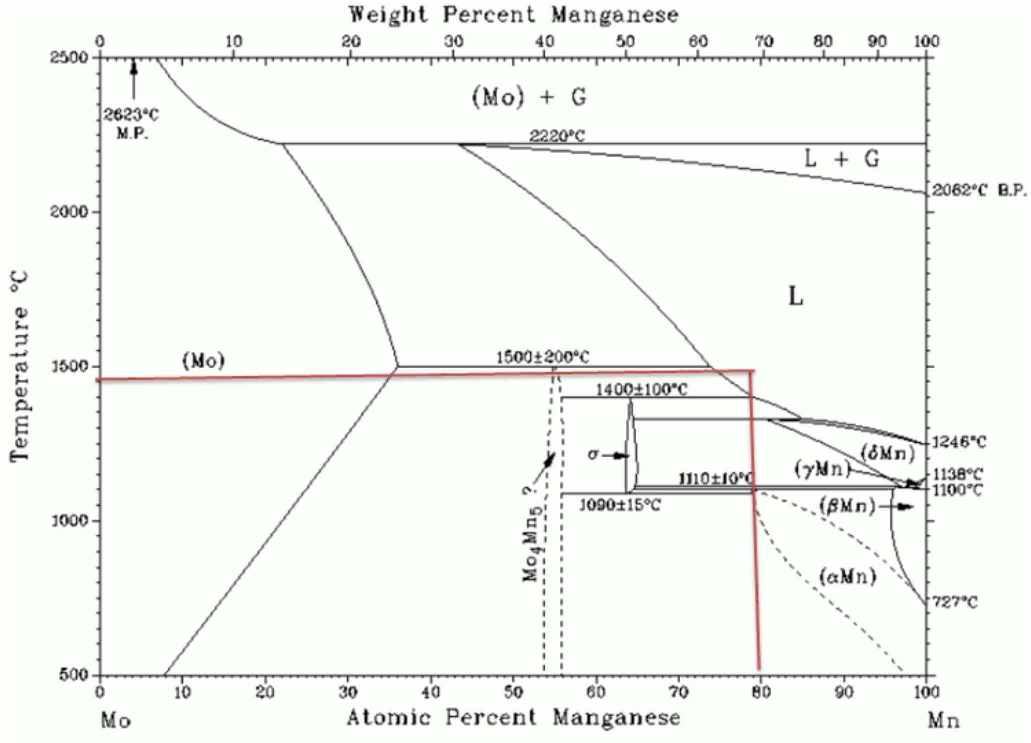
1 – Alumina
2- Interlayer
3-Ti-Cu
4-interlayer
5-Ag
6-Ag-Cu
7-interlayer
8- Cu

Alumina-copper structure zone - x200

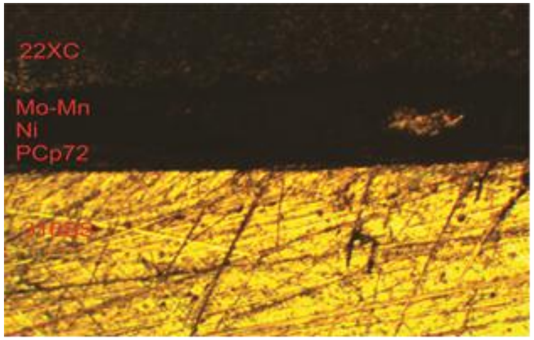


Metallization regime

thickness 5-10µm



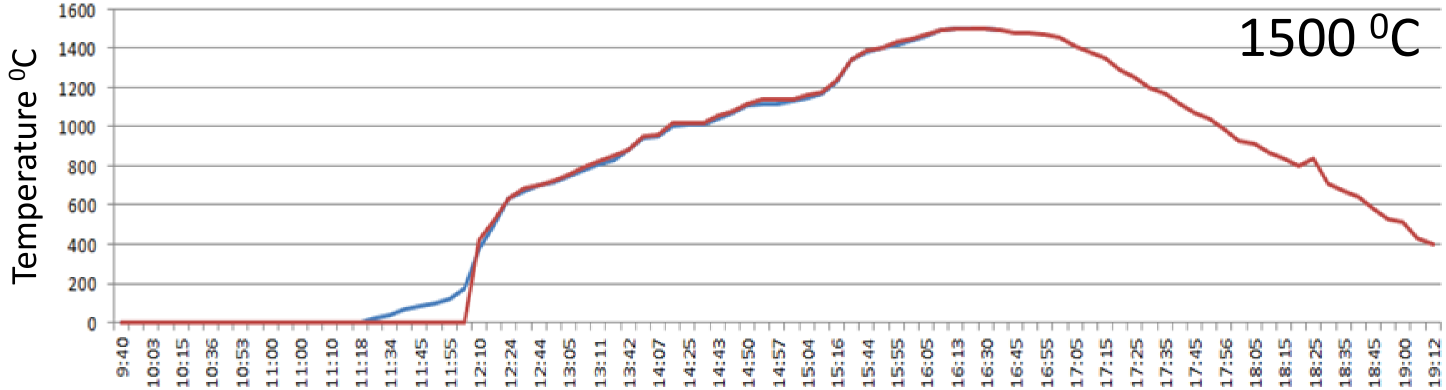
Phase diagram of Mo-Mn



95 % Alumina to SS brazing

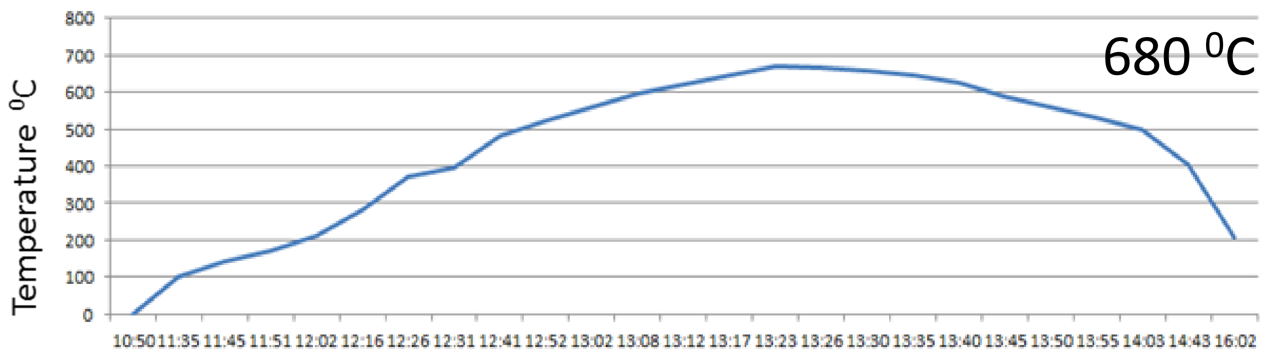
95 % & 99.5% Alumina to SS (Stainless Steel) brazing

Metallization samples



Time dependence on temperature

Time,



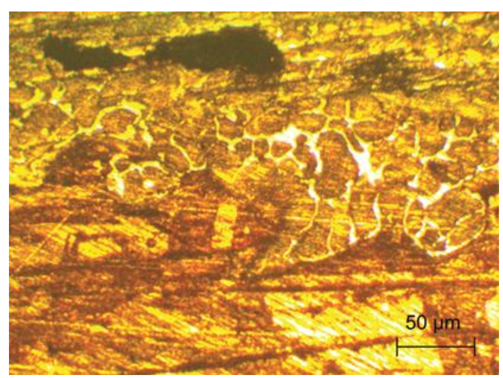
Temperature dependence to time.

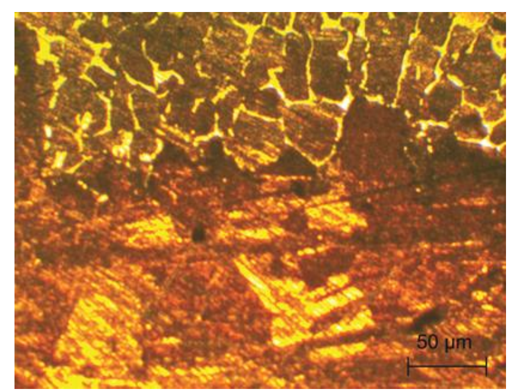
Time,

Ni Plated

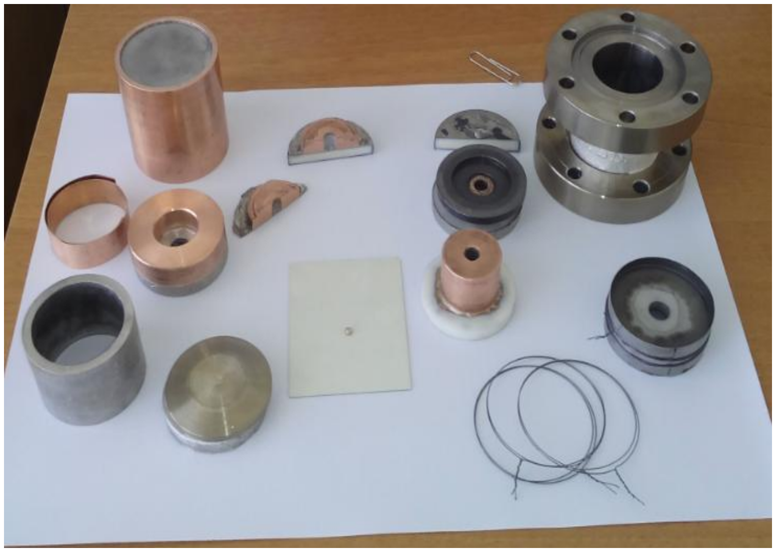
Alumina – Cu

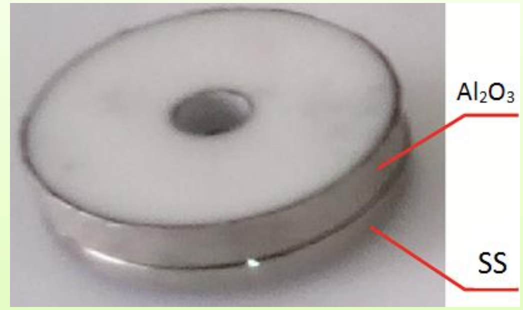
Brazed joint





Brazing of Alumina to Metals – Fixation Technologies



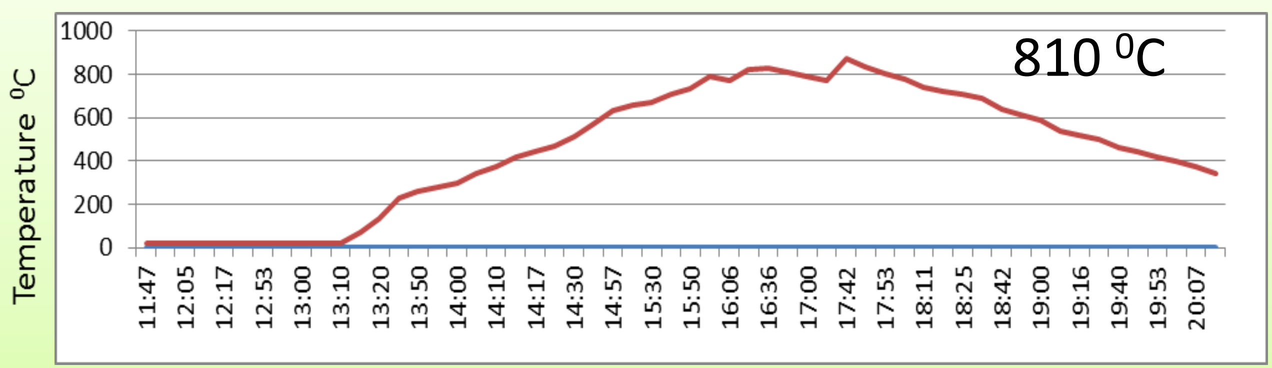


Alumina to SS brazing

Alumina to Cu brazing

Brazing Alumina to Kovar

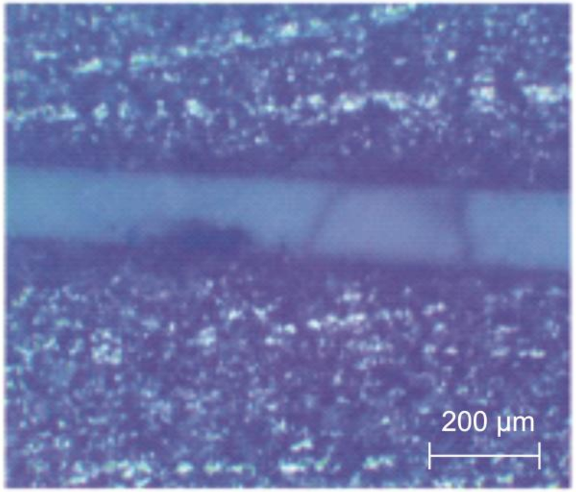
A- Brazed zone

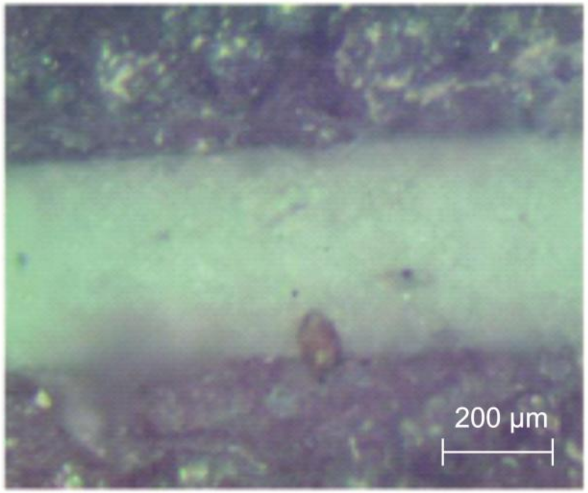


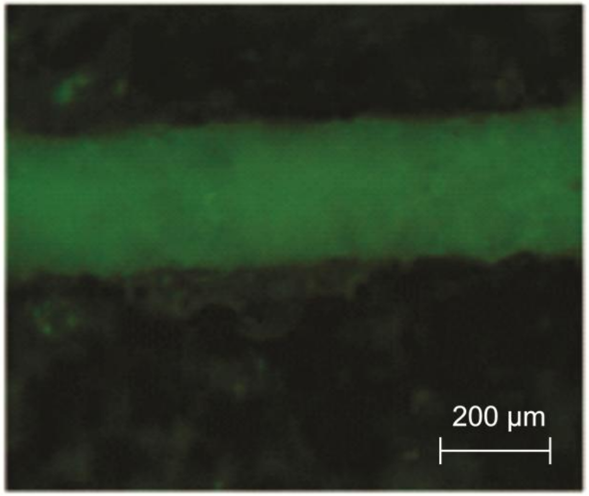
Time,

Temperature dependence on time of brazing process.

Gluing technics – special silicate glue







Alumina to Cu gluing
Alumina to Alumina gluing

Disadvantages
-Low mechanical strength,
-Low working temperature,
-Short lifetime,

95% Alumina to Alumina Joint

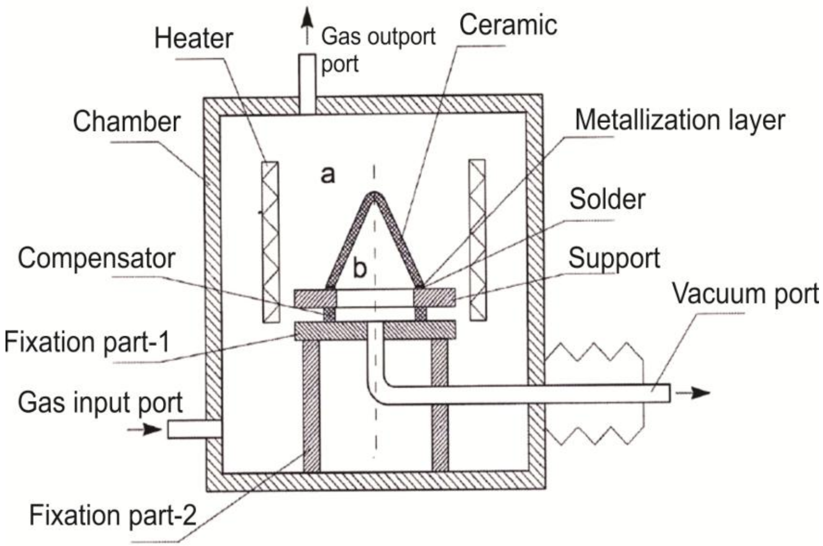
Summary

•Literature review for materials and brazing technologies for vacuum-tight ceramic-metal joints have been done. The molybdenum – manganese metallization technology was mentioned as more reliable technology for fabrication of vacuum-tight ceramic-metal joints.

•The importance of equal pressure creation and corresponding pressure level on items during pressing process for high quality metal-ceramic joints have been mentioned based on experimental and calculation methods.

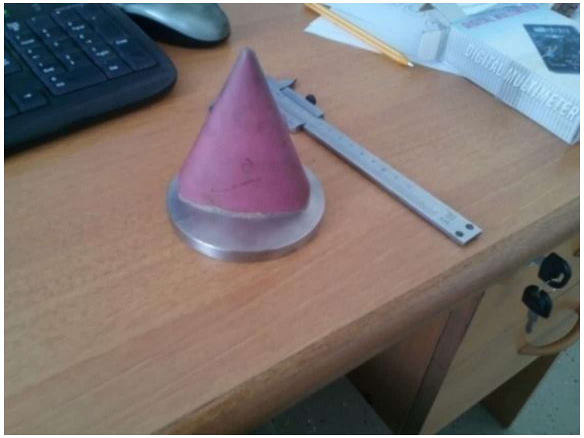
Chapter 3. Brazing technologies for ceramic-metal joints and thermo-mechanical simulations.

New Diffusion Brazing Method for Dissimilar and Difficult Geometry Items.



Patent Number 2883A

Advantages
- Equally pressure effects on items,
- Flexible pressure regulation,

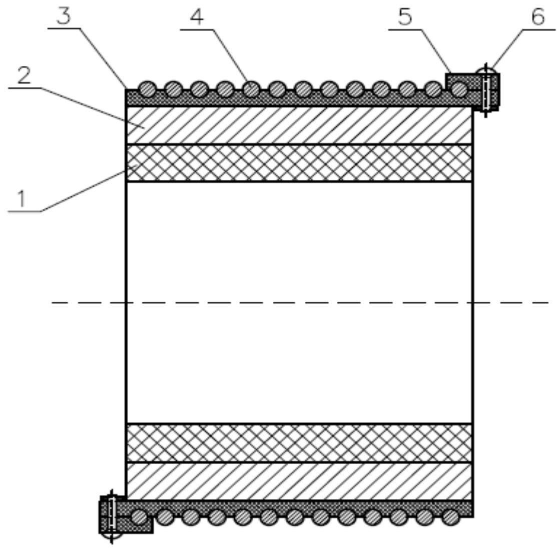


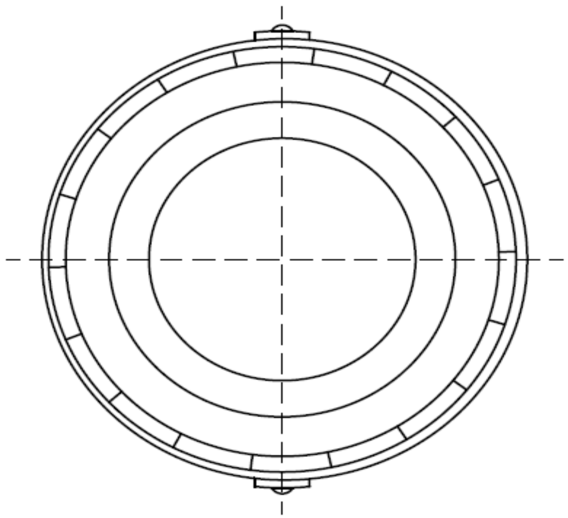
Experimental results

Side a. – 10^{-3} Torr
Side b. – atmospheric or 0.5 Atm pressure.

New Bonding Method for Materials – Patent Number - AM 20170070

New bonding method (diffusion brazing or welding) for cylindrical materials.





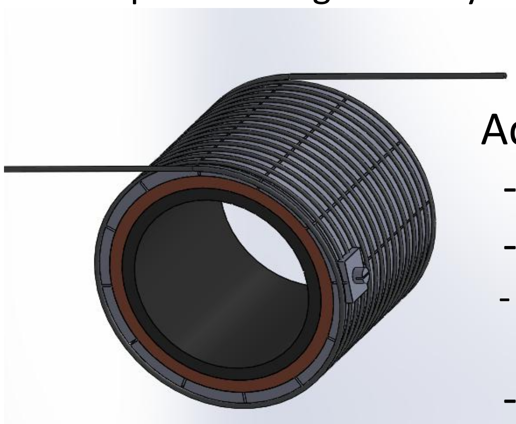
Patent Number 3130A

1. Ceramic,
2. Metal,
3. Ceramic,
4. Molybdenum wire,

5. & 6. Wire fixators.

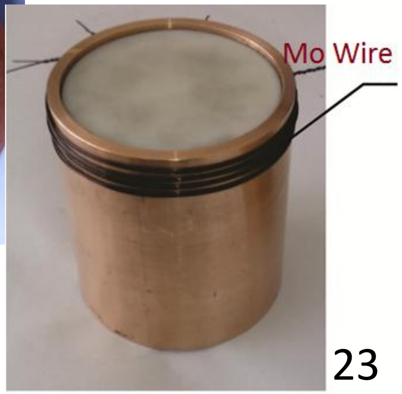
➢ New bonding method for bonding (diffusion welding or brazing) of cylindrical materials items, especially ceramic to metals by using locally heating and pressure receiving.

The temperature regulated by supplied electrical current.



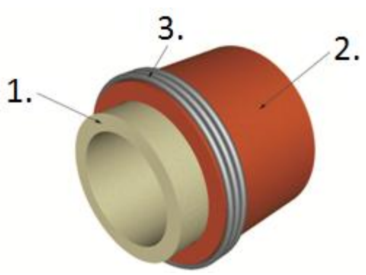
Advantages

- local heating;
- local pressure;
- Fluent regulation of temperature and pressure;
- Equal Pressure exert

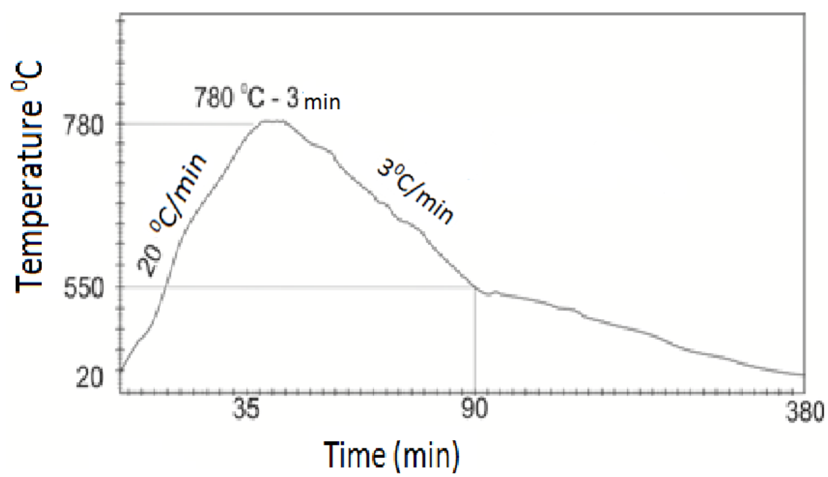


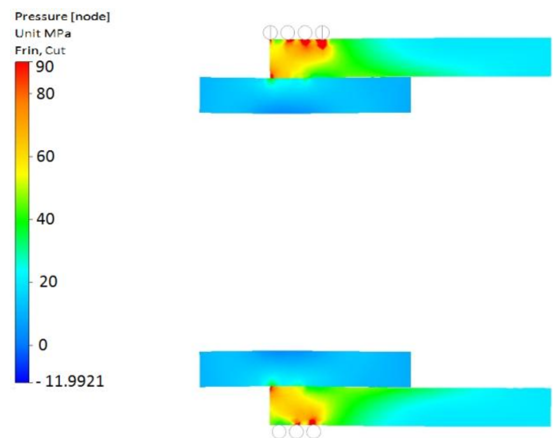
Alumina to Cu brazing

Mo wire fixation



1. Ceramic
2. Metal
3. Nb wire





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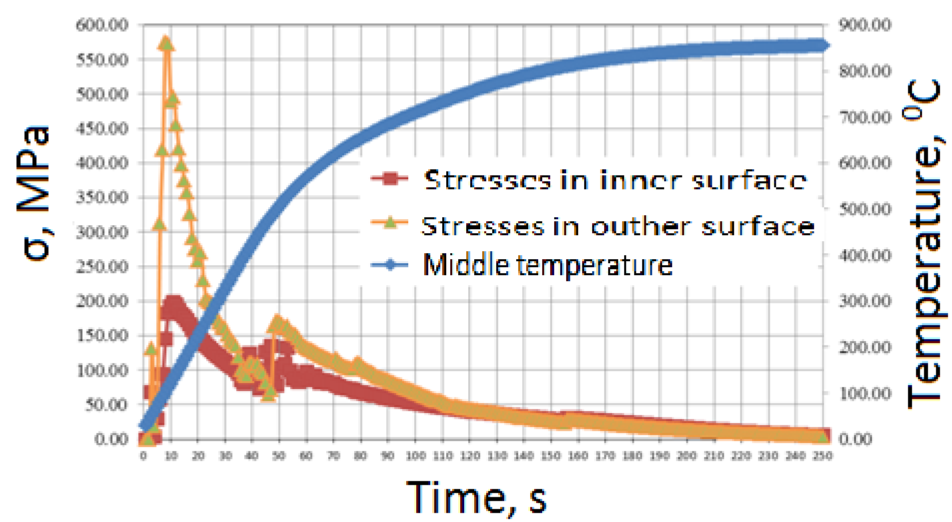
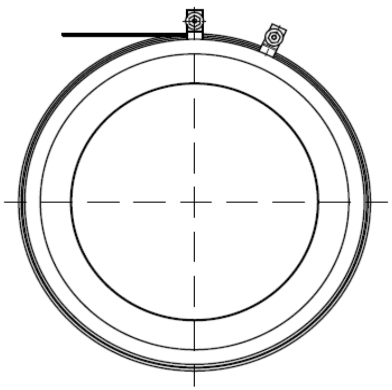
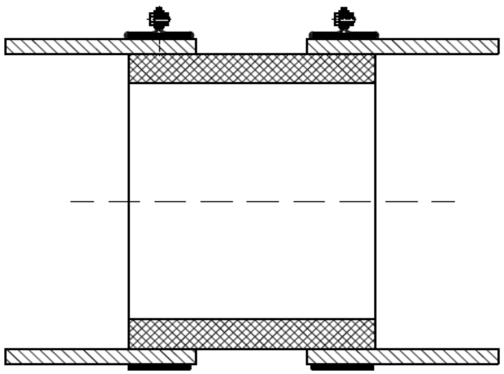
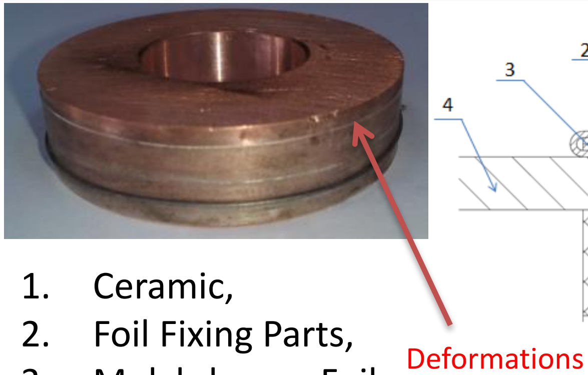
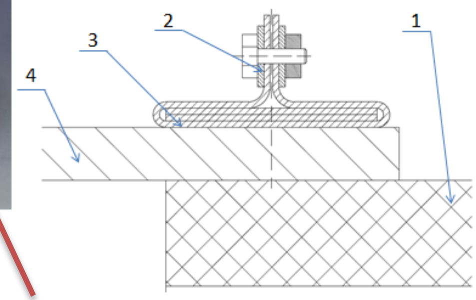


Fixation Method by use Metal Foil – (Molybdenum foil).



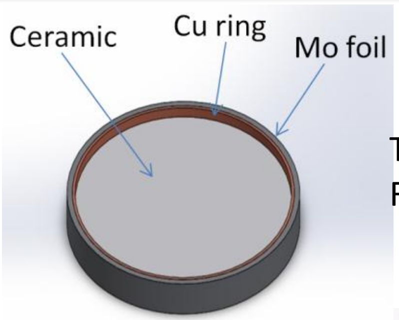






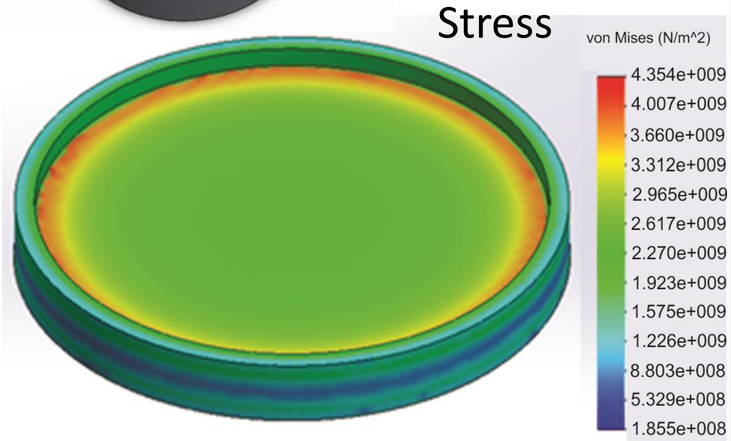
1. Ceramic,
2. Foil Fixing Parts,
3. Molybdenum Foil,
4. Metal

Thermo-Mechanical Simulations – (Preliminary)

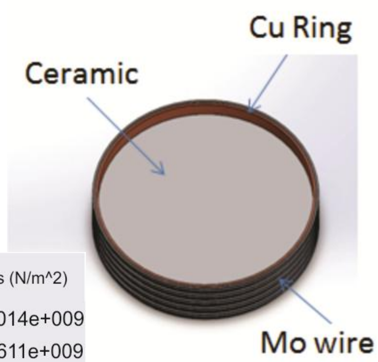


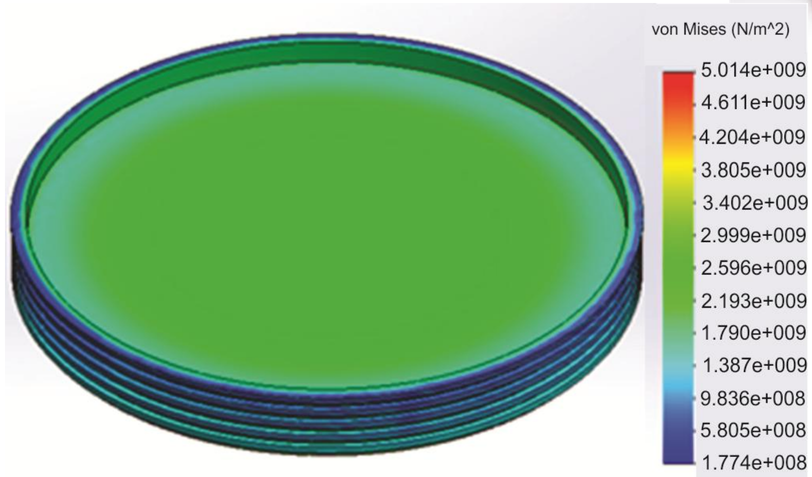
Temperature - 850 ^{0}C
Fixation - Mo Foil

Stress

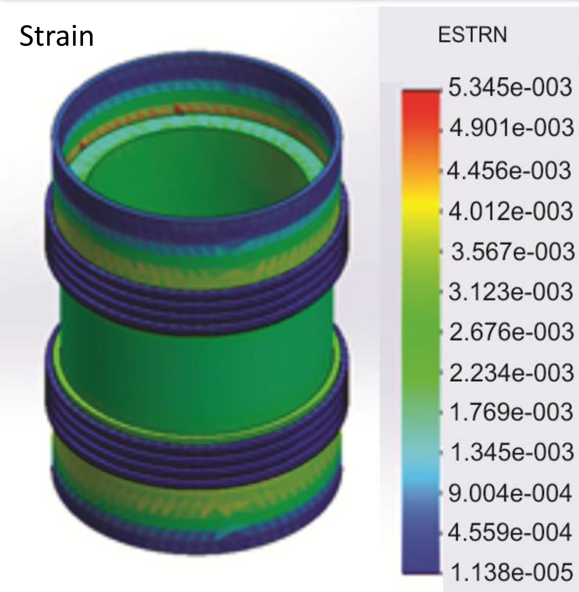


Temperature - 850 ^{0}C
Fixation - Mo Wire

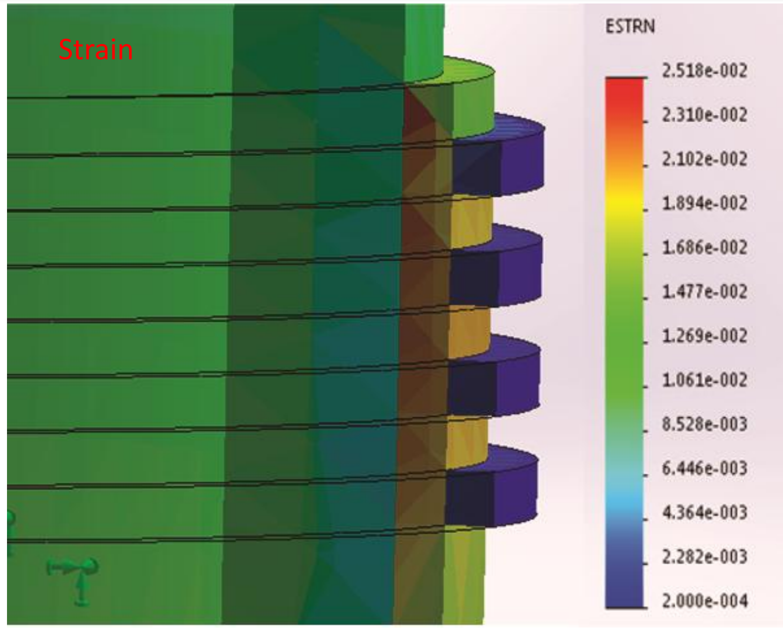




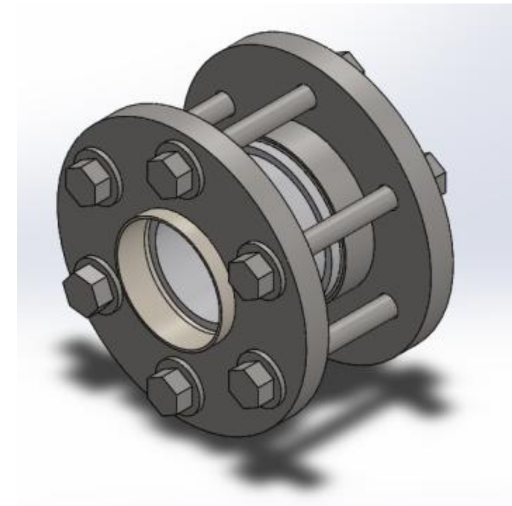
Stress

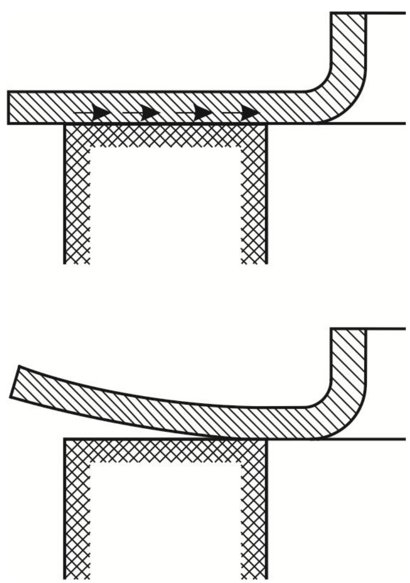


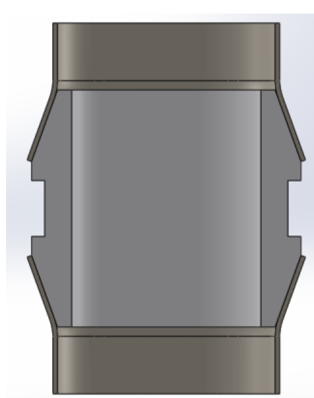
Temperature - 200 ^{0}C
Fixation - Mo Foil



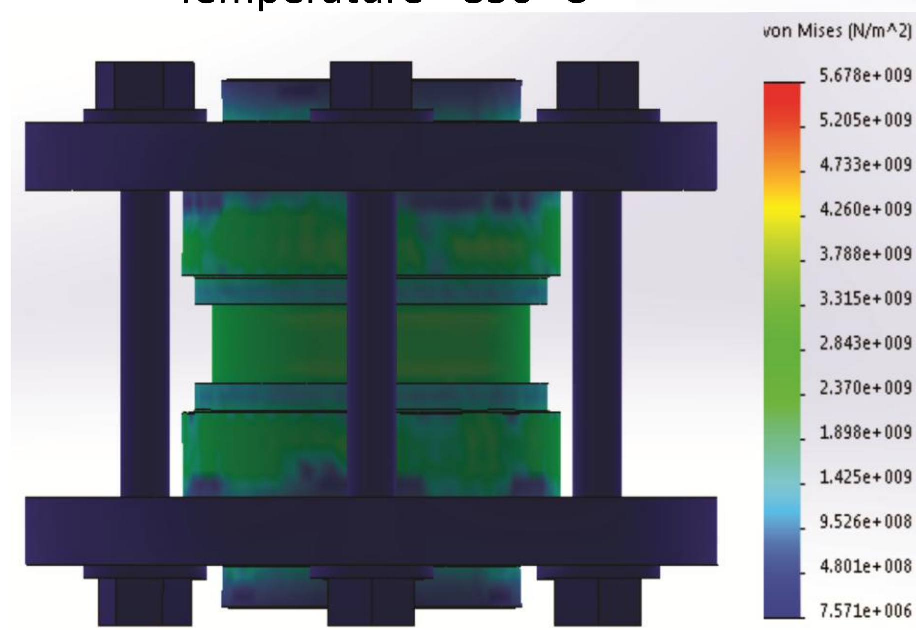
Temperature - 850 ^{0}C
Fixation - Mo Wire





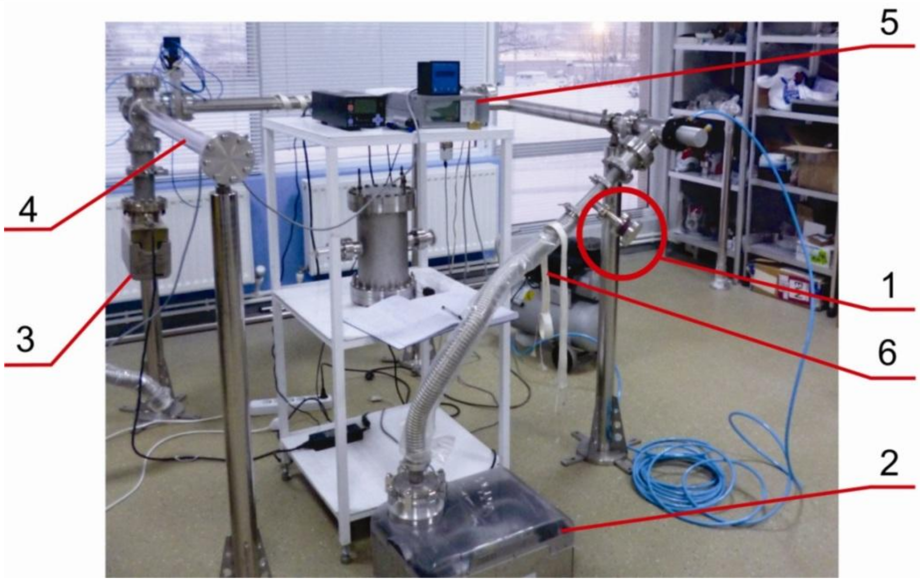


Temperature - 850 ^{0}C



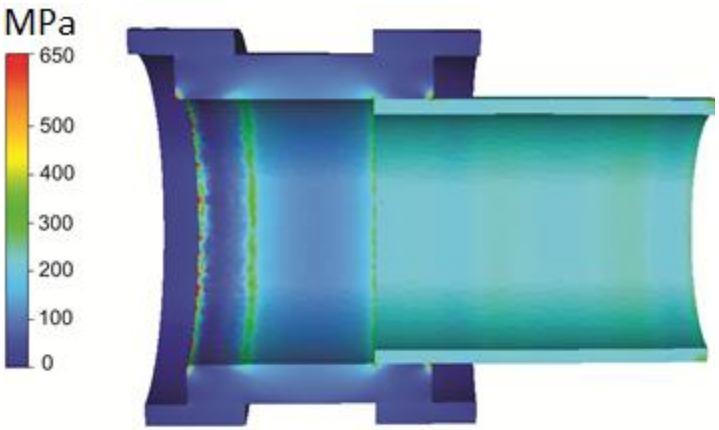
Stress

Experimentation

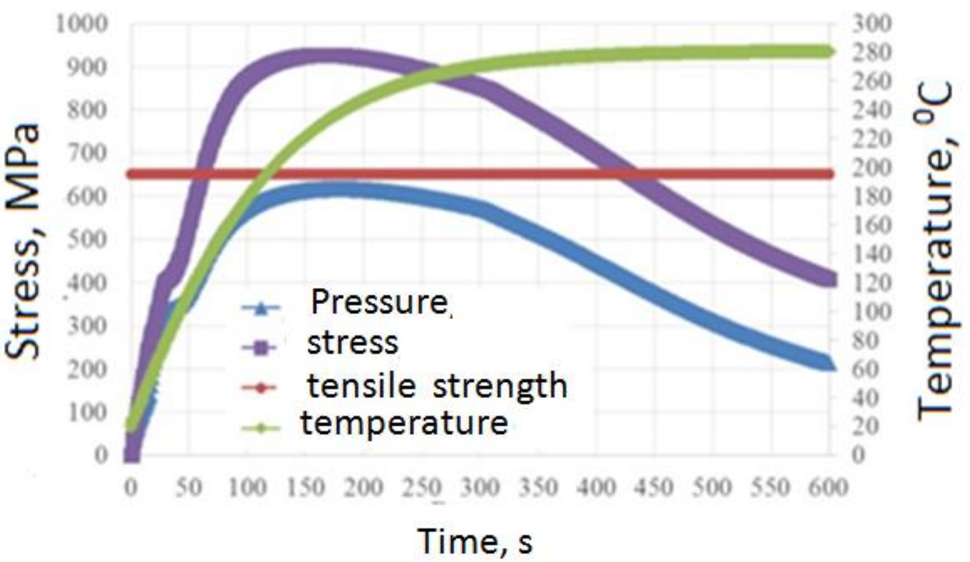


1. ceramic-metal joint;
2. TPS pump;
3. Ion pump;
4. Vacuum chamber;
5. Vacuum gauges;
6. Heater.

Max. working temperature 134 ^{0}C



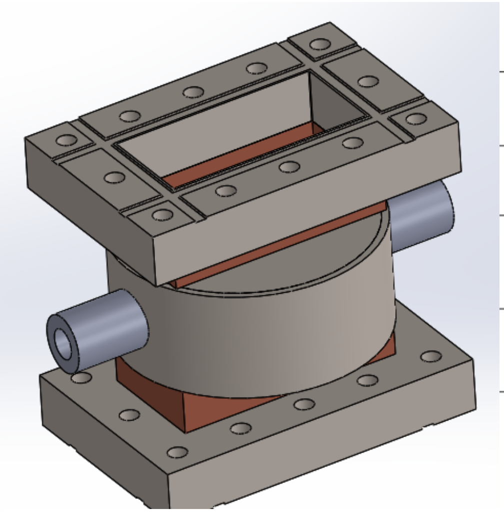
Stress.

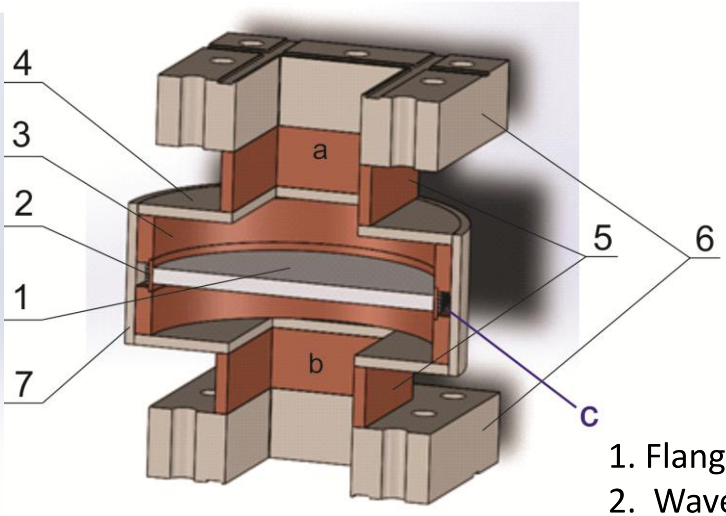


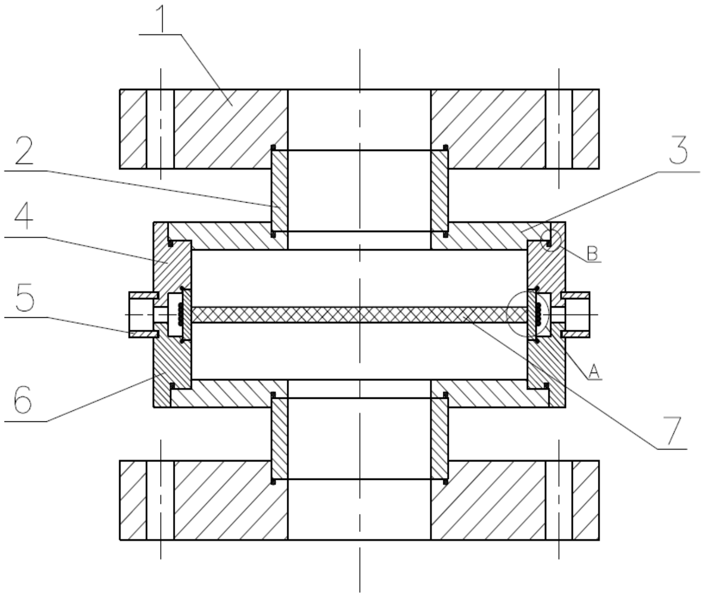
• New diffusion brazing method for dissimilar and difficult geometric ceramic and metal items was developed and designed.
• New diffusion bonding method for bonding of cylindrical ceramic and metal items was developed and designed.
• Thermo-mechanical simulations of ceramic disc – Cu ring system with different fixation methods and temperature conditions have been done.

Chapter 4. Vacuum-tight ceramic-metal joints in particle accelerators – RF window.

Pill box type RF window



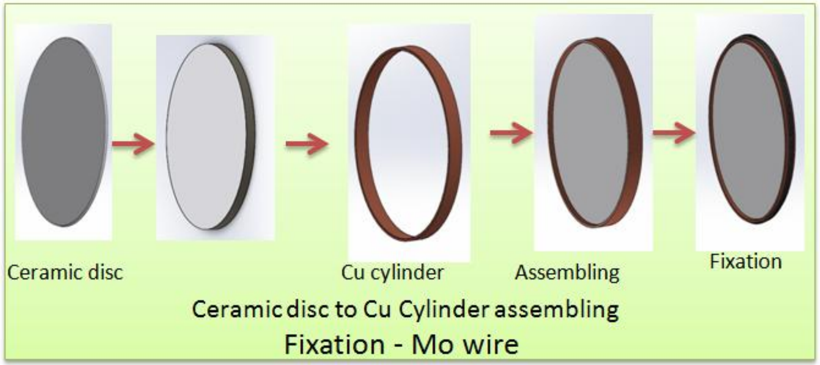




Requirements

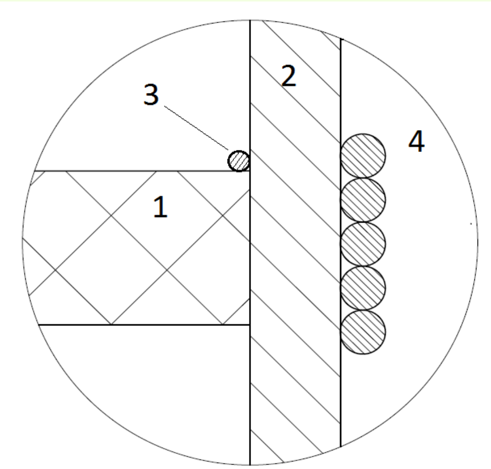
- High mechanical strength;
- High transparent level for electro-magnetic waves;
- Lower secondary electron emission;
- High conductivity of walls (metal);
- Reliability and durability;
- Outgassing low level;
- Higher reproducibility;
- High level of homogeneity of materials structures;

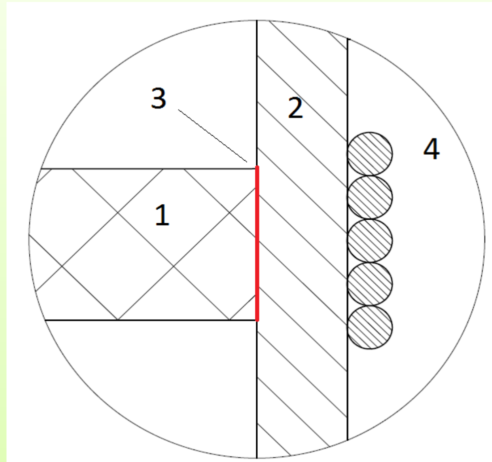
1. Flange
2. Waveguide
3. Pill box cover
4. Cylinder 1.
5. Water input and output pipes
6. Cylinder 2.
7. Ceramic disc



Solder fixation

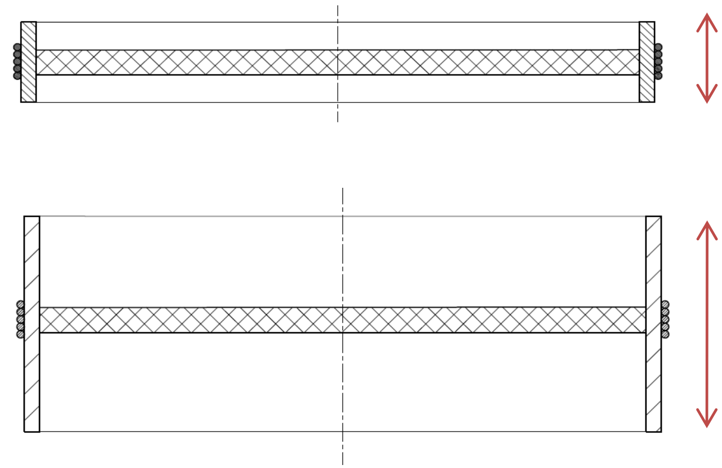
Ceramic Disc to Metal (Cu) Cylinder Brazing – Solder Fixation

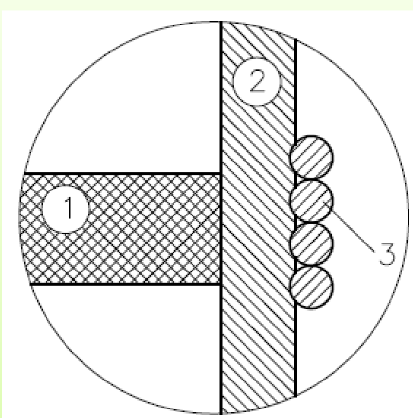




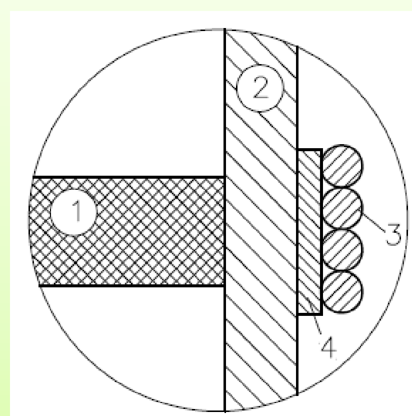
a – Solder Foil
b – Coated Solder Layer

1 – ceramic disc, 2- metal cylinder (Cu), 3- solder, 4- Mo wire



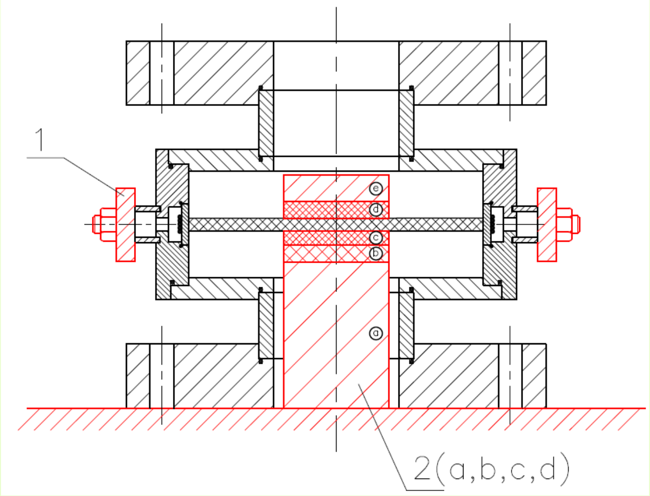


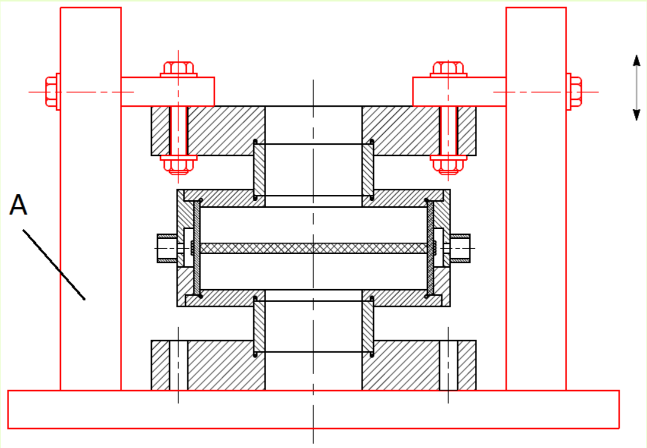
Mo wire immersion (deformation)



Mo Wire to Mo foil combination

1-ceramic disc,
2- Cu cylinder,
3-Mo wire,
4-Mo foil

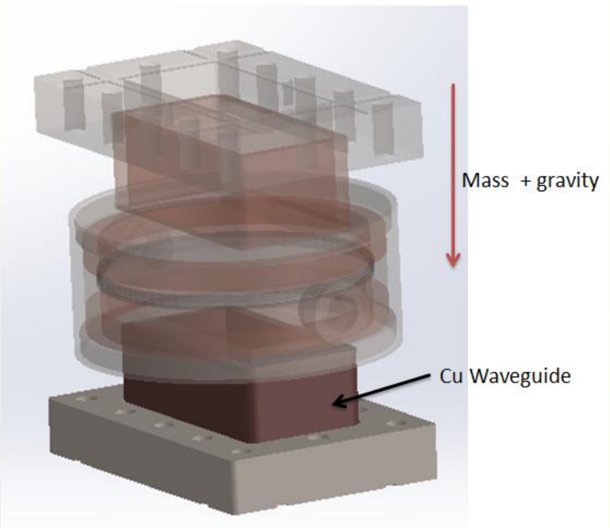


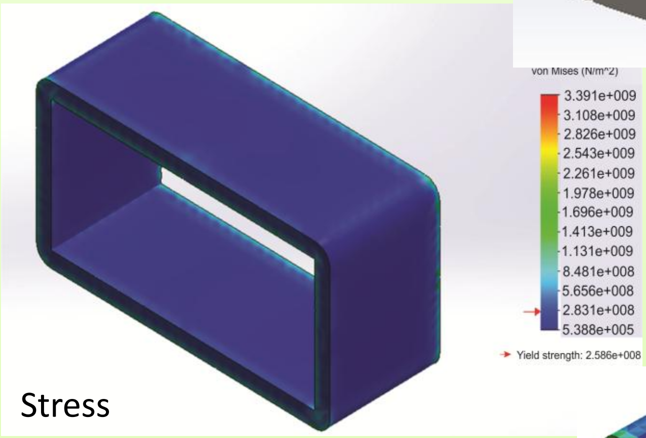


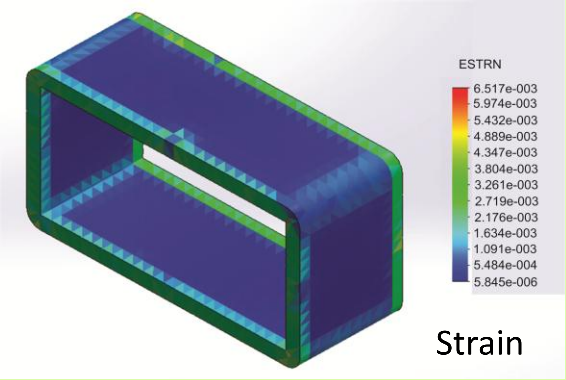
Fixation methods for Diffusion Brazing

Weight of RF window is 3.5 kg

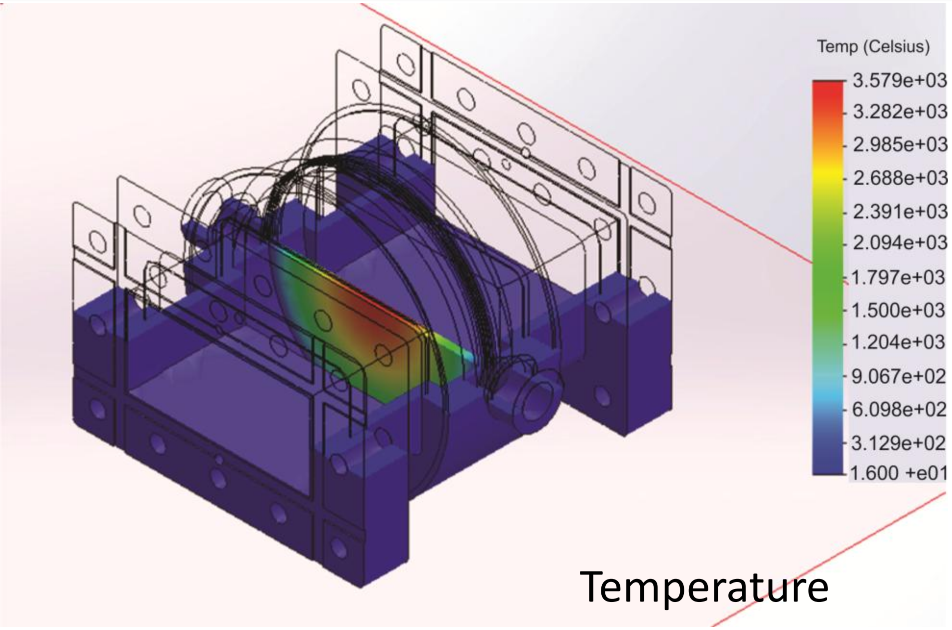
Pressure - 15kg.
Temperature -800 ^{0}C



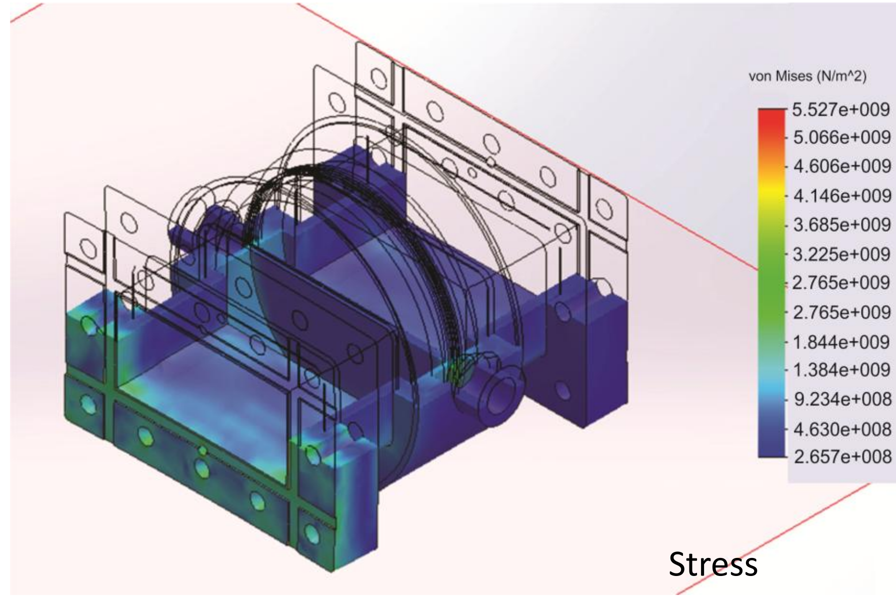




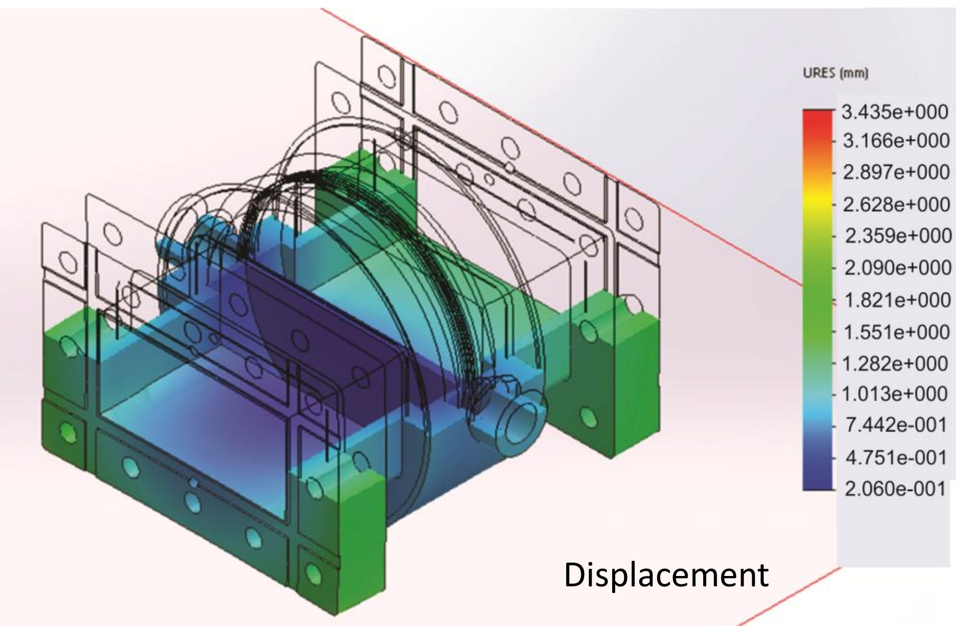
Thermo-mechanical Simulation of RF windows



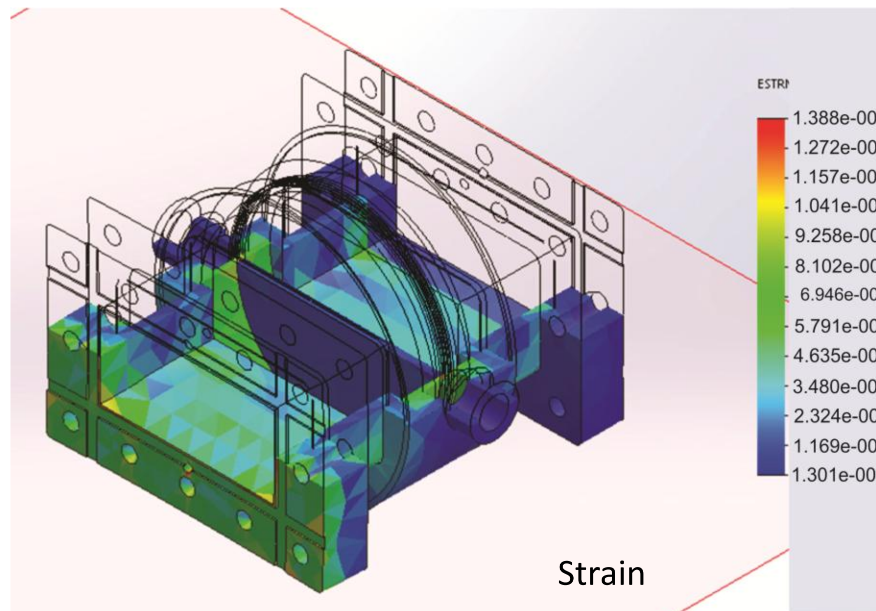
Temperature



Stress

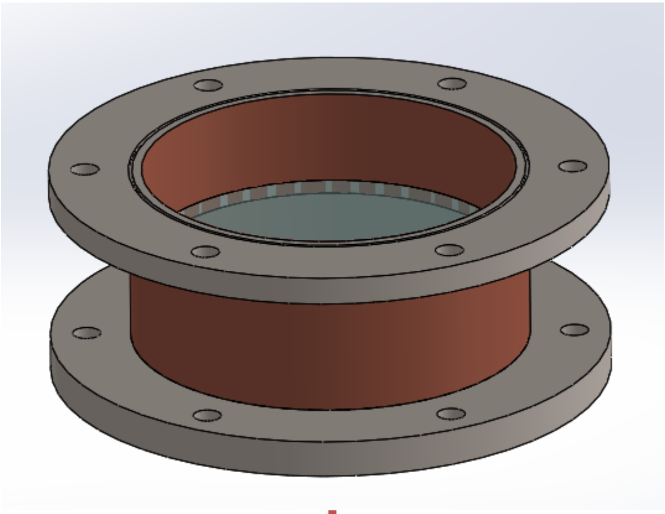


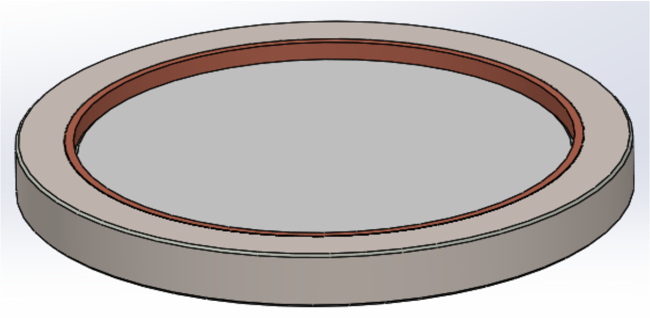
Displacement



Strain

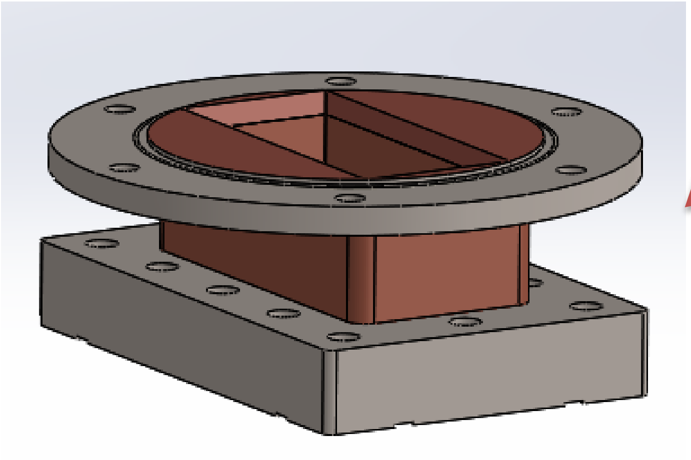
RF Window Test Stand

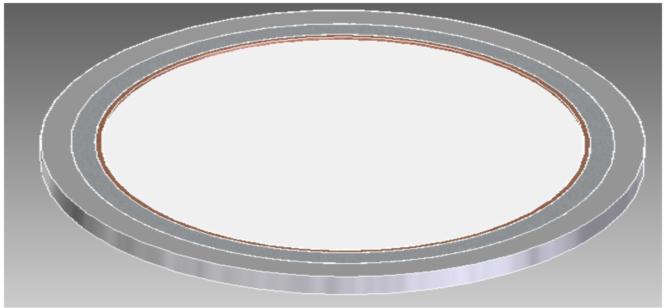




Ceramic- Cu-Mo ring

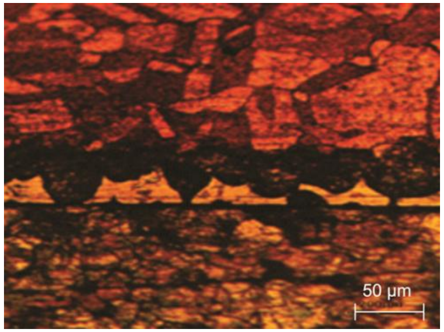
For testing of Ceramic to metal, metal to metal joints for RF windows



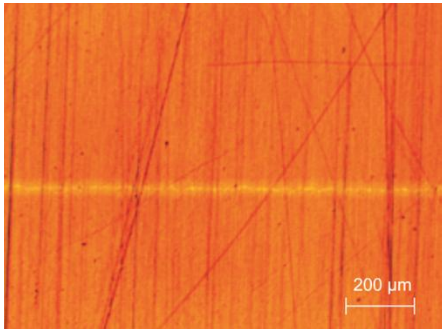


Ceramic- Cu-SS-Mo ring

Metals Brazing Technology for RF windows



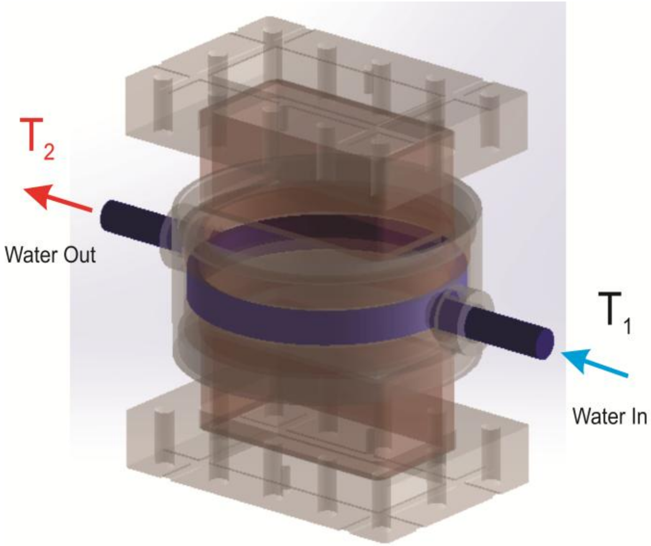
Cu to SS



Cu to Cu

SS – Stainless Steel

Thermoregulation for RF window



Coolant – distilled or deionized water

Testing technologies for mechanical design of RF windows

- Thermal tests;
- Vacuum tightness tests;
- Mechanical Tests;
- Vibration tests;
- Outgassing tests;
- Electro-mechanical tests;
- Humidity tests;
- Roughness measurement;
- Hardness measurement;
- Rotation test.

Summary – Chapter - 4

- The vacuum RF window has been designed including the materials selection, the thermo-mechanical simulation, the developed brazing technology, the evaluation of materials fixation methods to braze under the high temperature conditions.
- The vacuum RF window test stand has been designed and developed to test ceramic-metal joints under UHV, high temperature and RF conditions.
- The testing methods for the vacuum RF window are mentioned including leak detection, RGA (residual gas analyzing) under room and high working temperature conditions.

Conclusions

1. The diffusion brazing, a new method for dissimilar ceramic and metal items with difficult geometric shape, has been developed by the implementation of pressure differences in the inner and outer volumes of a joint system. The pressure in the inner volume of a ceramic-metal system is from 0.1 to 10^6 times lower than the pressure in the outer volume.
2. The diffusion bonding, a new method for cylindrical ceramic and metal items, has been developed. The assembled items are heated by the provided electrical current due to the fixed metal wire. The inner ceramic material and fixed metal wire have similar coefficients of thermal expansion which are lower than the ones of the outer cylindrical metals.
3. The thermoregulation systems for a precise operation of the vacuum electromagnetic equipment (Klystron, RF gun) of AREAL linear accelerator have been designed and fabricated. The thermoregulation system of klystron provides +/-0.5^{0}C temperature accuracy. The thermoregulation system of the RF gun is a precise and fast system which provides +/-0.1^{0}C temperature accuracy.
4. The Ultra High Vacuum (UHV) test stand has been designed and fabricated to test and investigate separated accelerator UHV components. The commissioning temperature range of a vacuum tight ceramic-metal joint has been defined by calculation and experimental results. The experiments have been carried out under 130^{0}C temperature and $7\cdot10^{-9}$Torr vacuum conditions.
5. The vacuum RF (Radio Frequency) window has been designed. The stress and strain properties of the RF window have been defined by a modeling method under the diffusion brazing temperature.

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2. V.V. Vardanyan. **Brazing technologies of ceramic to metals for UHV systems of charged particle accelerators.** Mechanical Engineering and Metallurgy, UDK 655.344.022.72. Volume 14, Number1, Yerevan, 2017, pp. 117-120.
3. V.V. Vardanyan, V.S. Dekhtyarov, V.A. Danielyan, V.Sh. Avagyan, A.A. Gevorgyan, T.H. Mkrtchyan. **Investigation and testing of brazed metal/ceramic zones in ultra high vacuum conditions.** Proceedings of engineering academy of Armenia, Volume 14, Number 2, Yerevan, Armenia, 2017, pp.254-259.
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5. V.V. Vardanyan, V.Sh. Avagyan. **Compearing of metal-ceramic bonding methods for ultra high vacuum.** ISSN 1829-0043, PROCEEDINGS OF ENGINEERING ACADEMY OF ARMENIA (PEAA), V.12,N.1, Yerevan, Armenia, 2015, pp. 192-195.

6. V.V. Vardanyan, V.Sh. Avagyan. **Investigation and design of metal to ceramic bonding technologies for particle accelerator's vacuum RF window.** International Conference ''Advanced Materials and Technologies'' 21-23 October, Tbilisi, Georgia, Dedicated to the 70th anniversary of foundation of Ilia Vekua Sukhumi Institute of Physics and Technology, 2015, pp. 230 – 235.

7. V.V. Vardanyan, V. Avagyan, B. Grigoryan, A. Gevorgyan, et al. **Distributed Cooling System for the AREAL Test Facility.** Proceedings of IPAC2014, Dresden, Germany, 2014, pp. 4010-4012.

8. A.A. Gevorgyan, V.Sh. Avagyan, B.A. Grigoryan, T.H. Mkrtchyan, A.S. Simonyan, V.V. Vardanyan. **Design and Performance of Ultimate Vacuum System for the AREAL Test Facility.** Proceedings of IPAC2014, Dresden, Germany, 2014, pp. 2311-2313.

9. V.V. Vardanyan, V. Sh. Avagyan, A. A. Gevorgyan, B.A. Grigoryan, N.W. Martirosyan, T.H. Mkrtchyan, A.S. Simonyan, A.S. Vardanyan. **Precise cooling system for electro-magnetic equipment.** Proceedings of Engineering Academy of Armenia, Volume 10, Number 3, Yerevan, Armenia, 2013, pp. 537-541.

10. Vardan Shavarsh Avagyan, Vahagn Vanik Vardanyan, “Diffusion brazing methods of difficult geometry dissimilar items”, Invention, Intellectual Property Agency of Armenia, AM201453 , N 2883 A, 2014, Yerevan, Armenia.

11. Vahagn Vanik Vardanyan, Vardan Shavarsh Avagyan, “Diffusion bonding method”, Invention, Intellectual Property Agency of Armenia, AM20170070, N 3130A, 2017, Yerevan, Armenia.

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Thank You for Your Attention