Center for the Advancement of Natural Discoveries using Light Emission

Vacuum Tight Metal to Ceramic Joining

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Ceramic Properties

In general, most ceramic are:

- Hard,
- Wear-resistance,
- Brittle,
- Refractory,
- Thermal insulators,
- Electrical insulators,
- Nonmagnetic,
- Oxidation Resistant,
- Prone to thermal shock,
- Chemically stable.

Advanced Ceramics Materials











- Aerospace,
- Nuclear Waste Containment,
- Military Armor Systems,
- Automotive /Engine,
- Semiconductors,
- Heat Exchangers,
- Fluid Handling,
- Medical,
- Metallurgy,

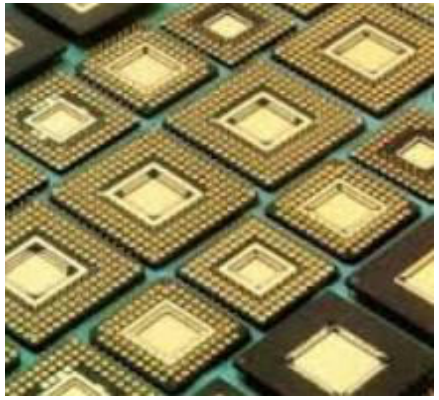
Aerospace Silicon Nitride

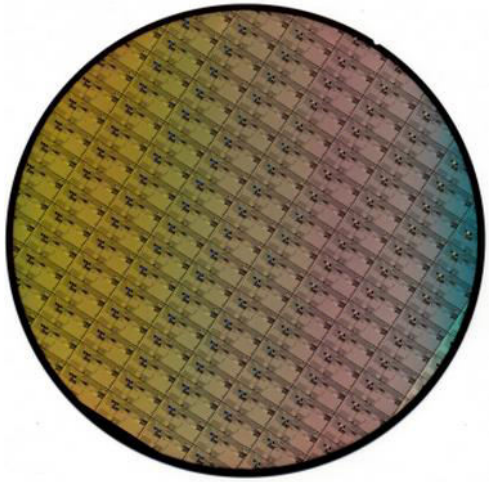
Silicon Nitride provide Superior mechanical reliability wear resistance.

- Jet engine igniters.
- Missile radomes.
- Aircraft engine parts.
- Hydraulic wear components.

Ceramic Cutting Tools (Si3N4, etc.)

Semiconductor Applications



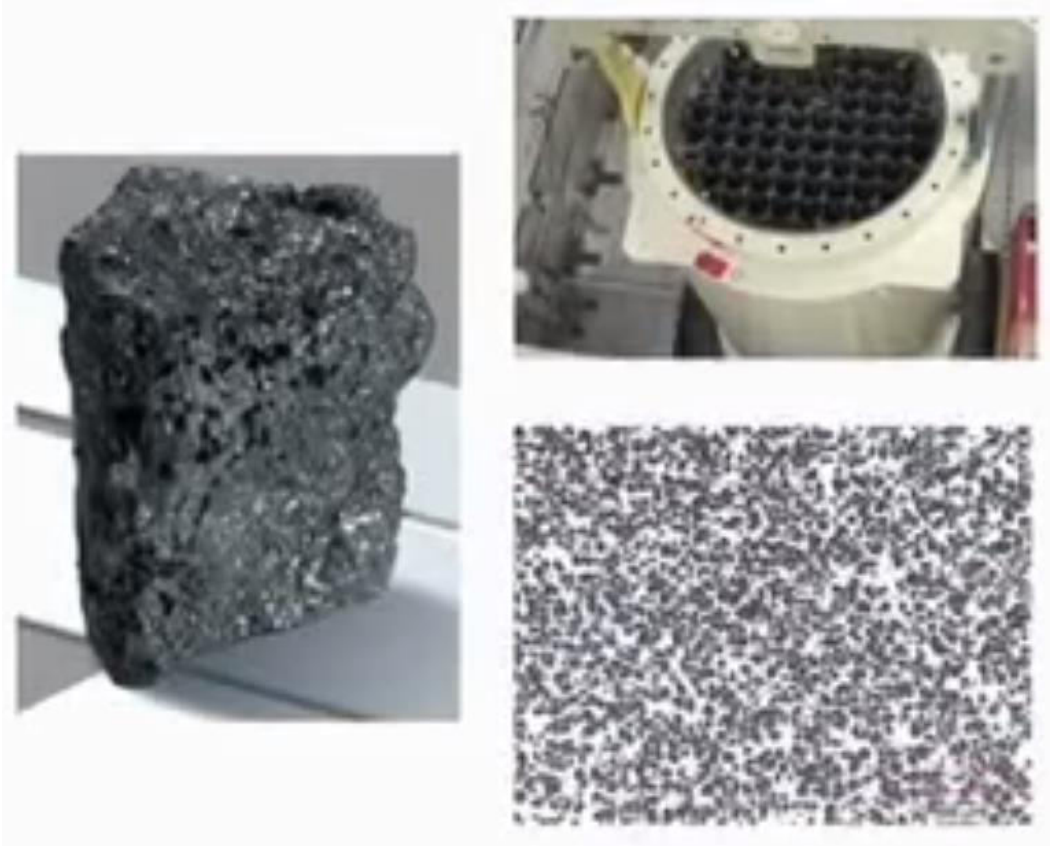


- Vacuum Chucks – AlN,
- RF Windows – Y2O3,
- Focus Rings,
- Gas Distribution Plantes – Si3N4,
- Crucibles – SiO2,
- Heaters – AlN.

Silicon Nitride

- Extreme Thermal Shock Resistance,
- High Toughness (for ceramic),
- Strong electrical insulator,
- 1/3 weight of steel,
- RF transparent.

Boron Carbide
Nuclear Waste Containment



Neutron absorption and shielding of nuclear fuel storage and transport

Metal to ceramic junctions in UHV technology

Bayard Alpert Gauge

Molecular Pumps

UHV multiple pin feedthoughs

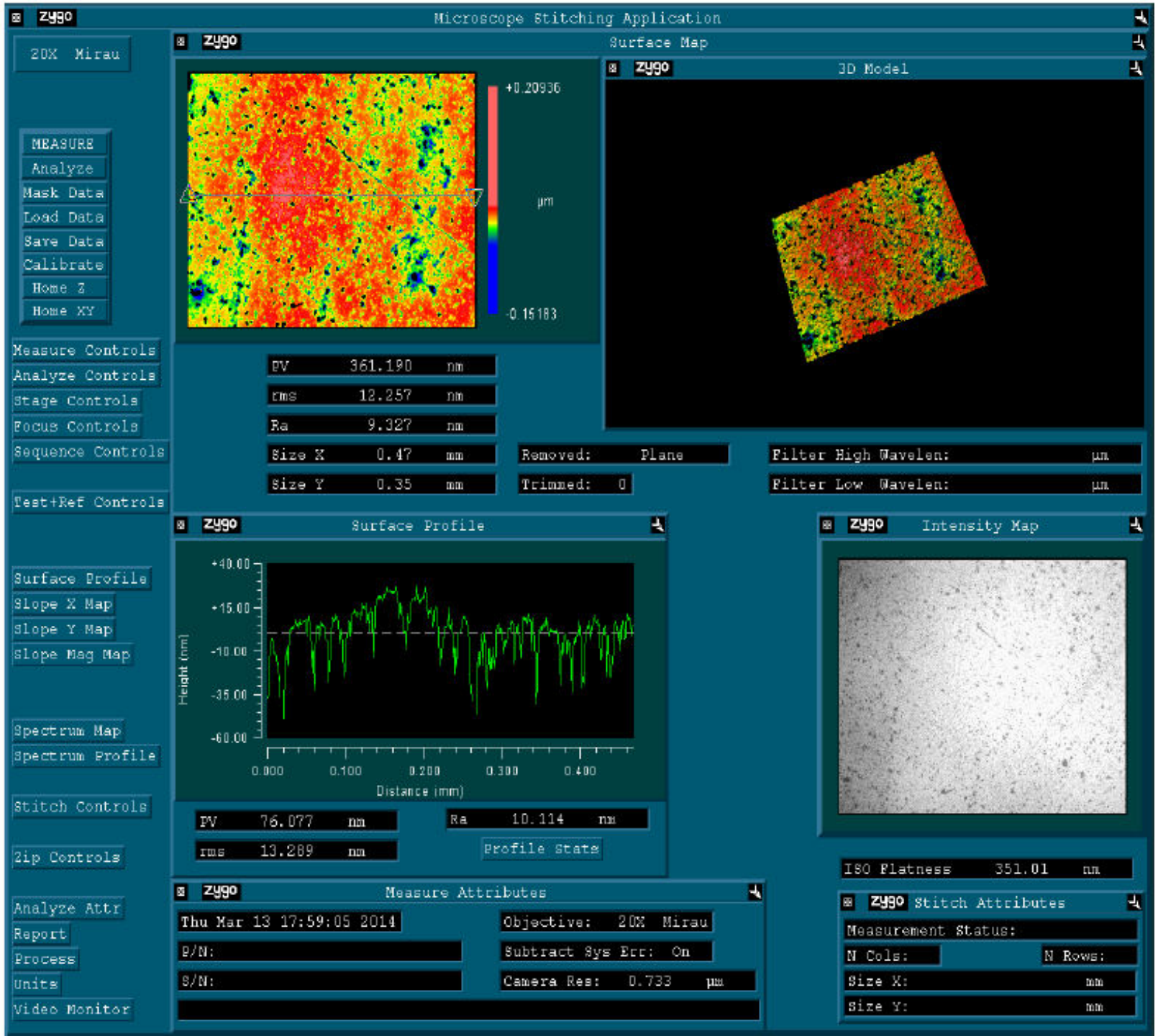
Vacuum Chambers for Accelerators

Metal – Ceramic Joint Parameters

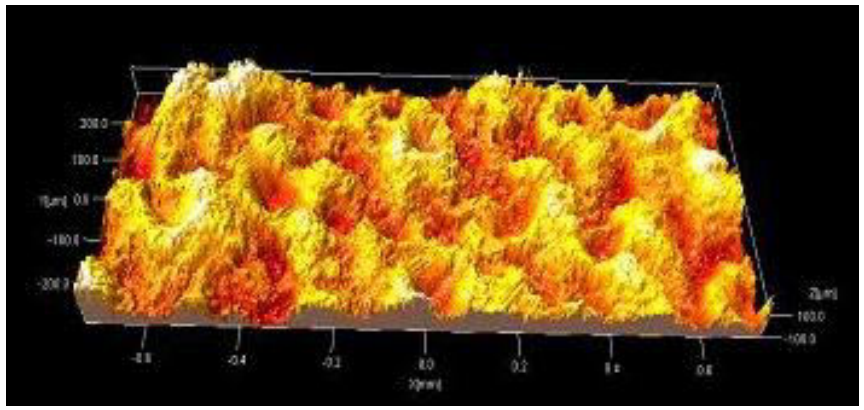
- High Mechanical strength,
- Outgassing Low Level,
- Reliable during long time,
- Electro – Magnetic Parameters,
- Low Material Penetration,
- Dimensional Stability,
- Thermal shock resistance.

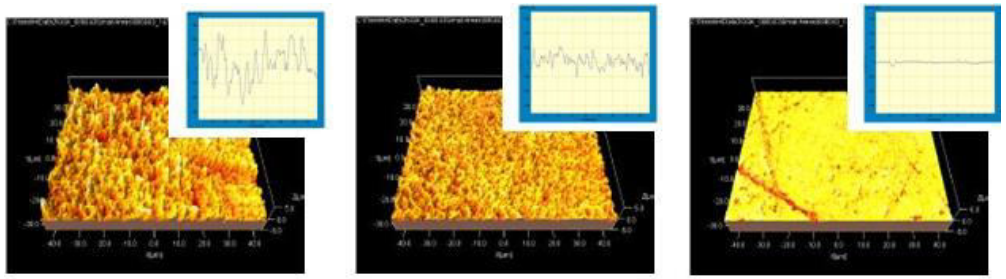
Parameters

Colour	White
Density	3.70 g/cm^3
Porosity (apparent)	0% (fully dense) % nominal
Tensile Strength	30,000 psi
Compressive Strength	300,000 psi
Coefficient of Thermal Expansion	6.6 (10^{-6}/°C) 25-300°C
Hardness (Mohs Scale)	9
Thermal Conductivity	20.5 W/m.K @RT
Dielectric Strength	230 (dc V/mil) @ RT
Volume Resistivity	>10^{13} (Ohm-cm) @ RT
Dielectric Constant	9.3 (1 MHz @ RT)
Dissipation Factor	0.0004 (1MHz @ RT)

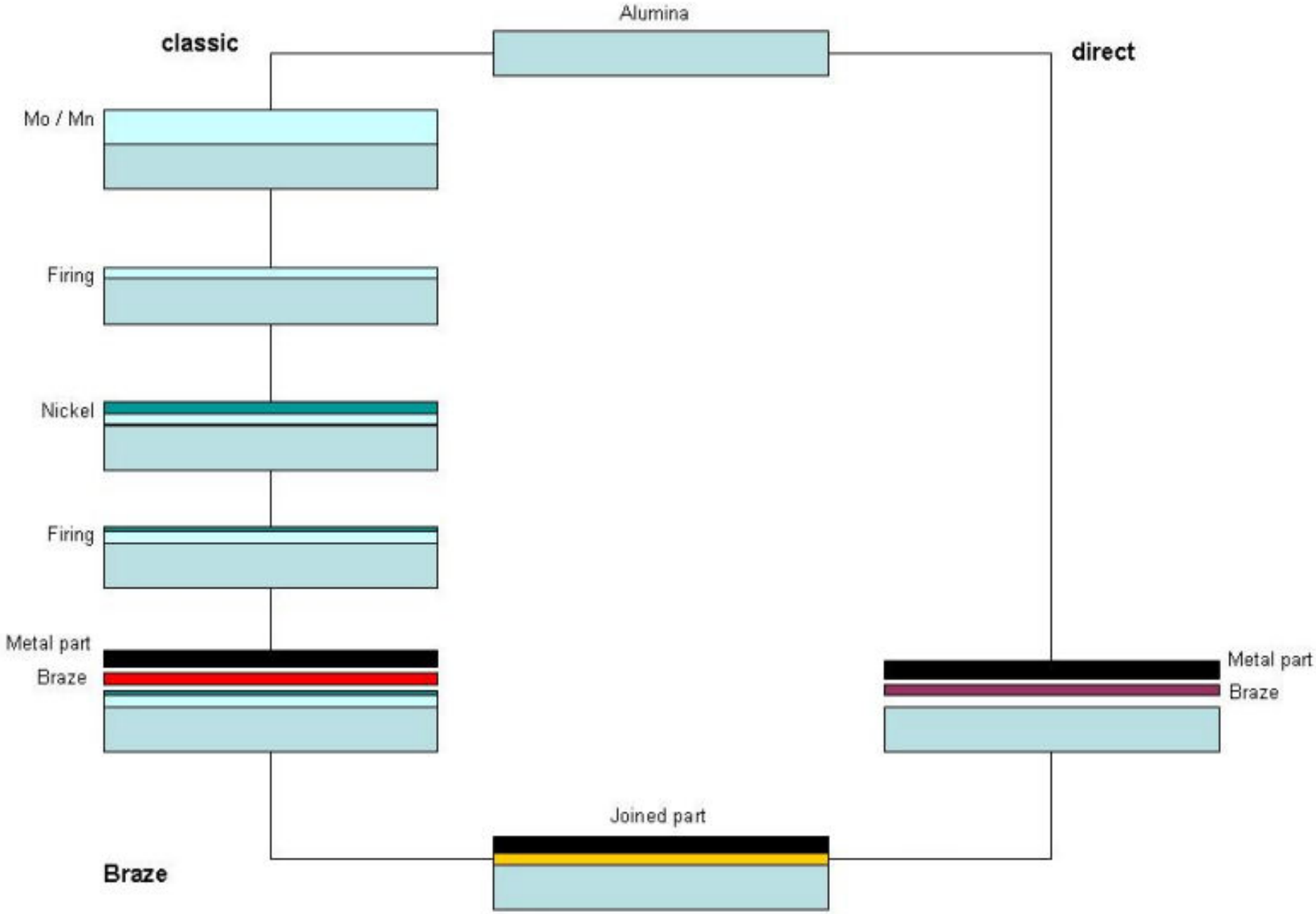


Interferometric profilometer





No treatment | Non-Fluoride Mouthwash | Fluoride Mouthwash


Alumina
classic
direct
Mo / Mn
Firing
Nickel
Firing
Metal part
Braze
Metal part
Braze
Joined part
Braze

Diffusion Welding Laboratory

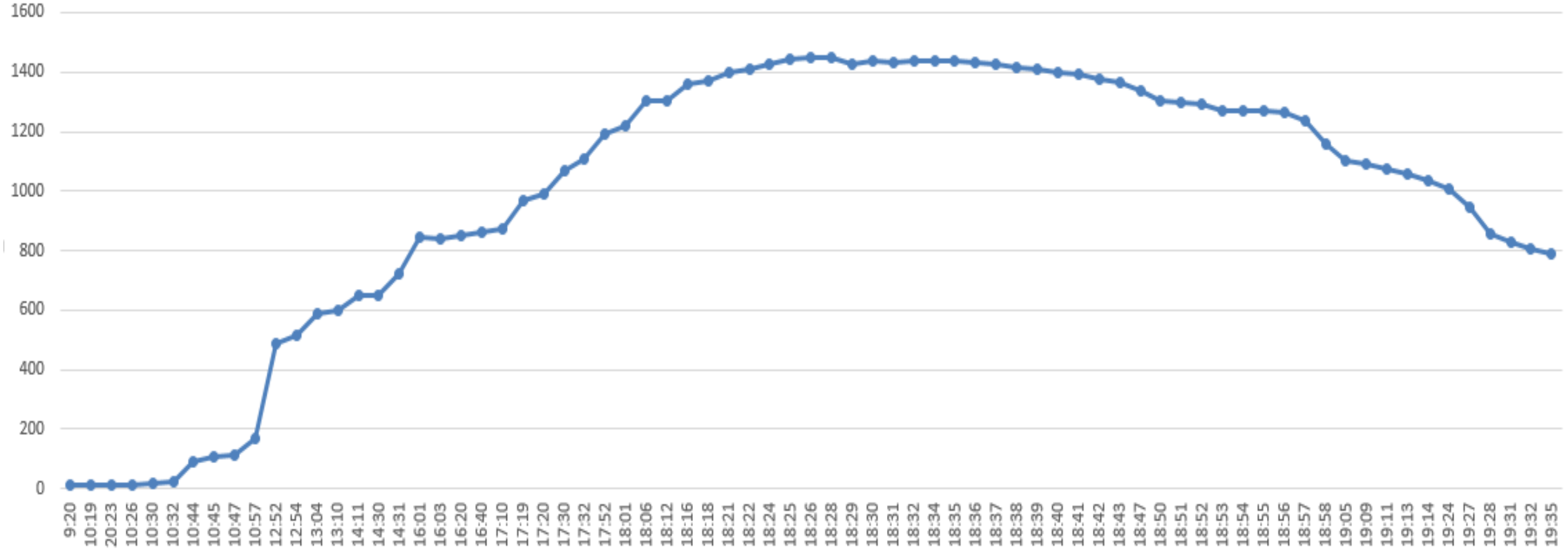
Induction-Vacuum Furnace

Tunnel Furnace

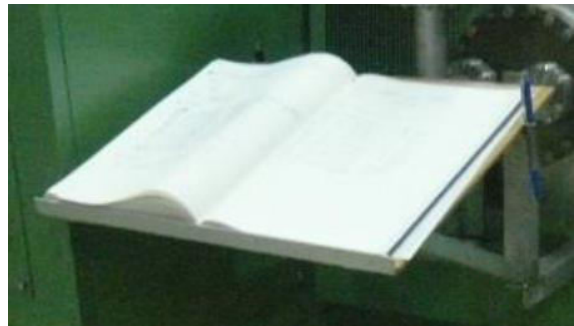
Vacuum Furnace

Diffusion Welding , Brazing parameters

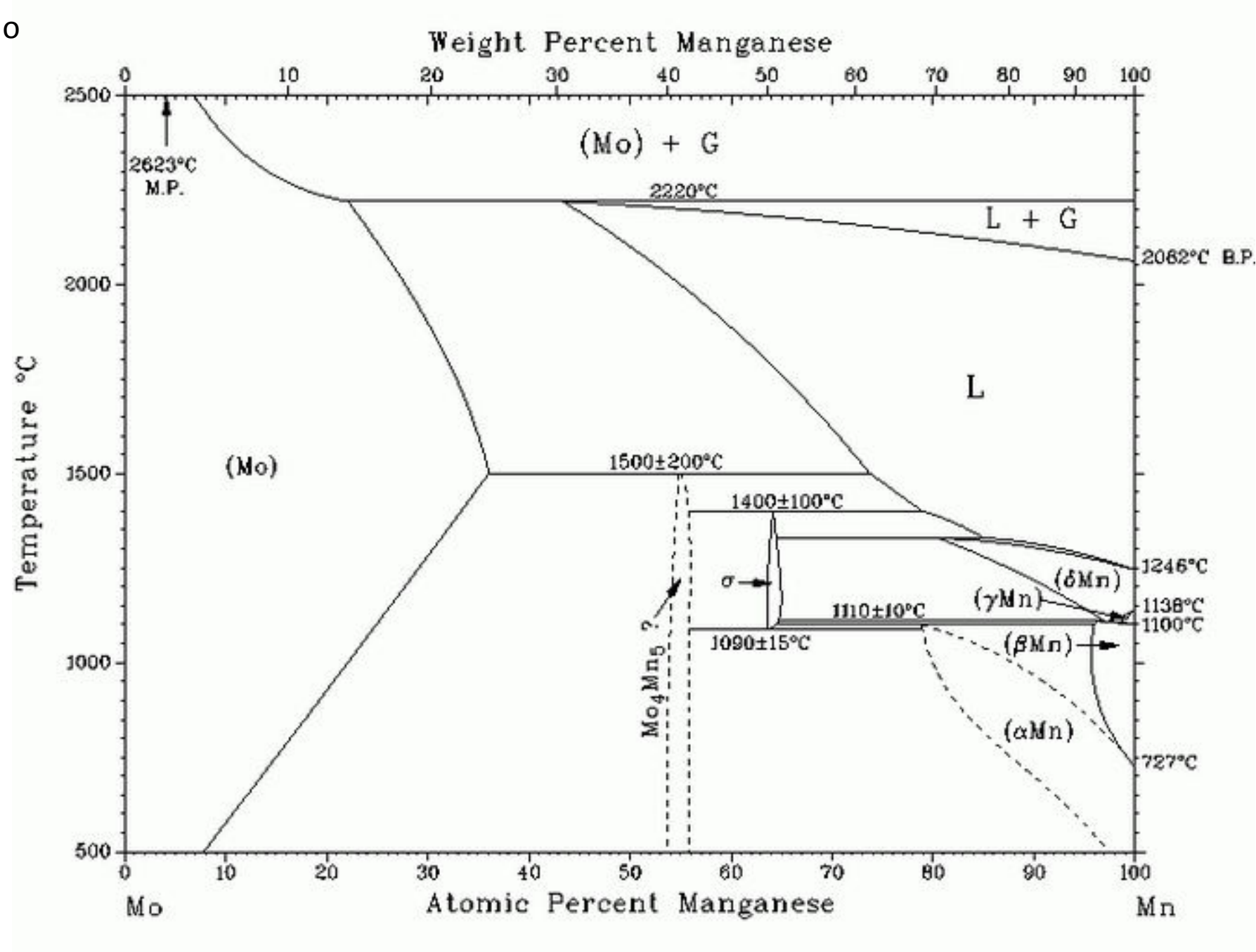
- Pressure,
- Temperature,
- Time,
- Environment



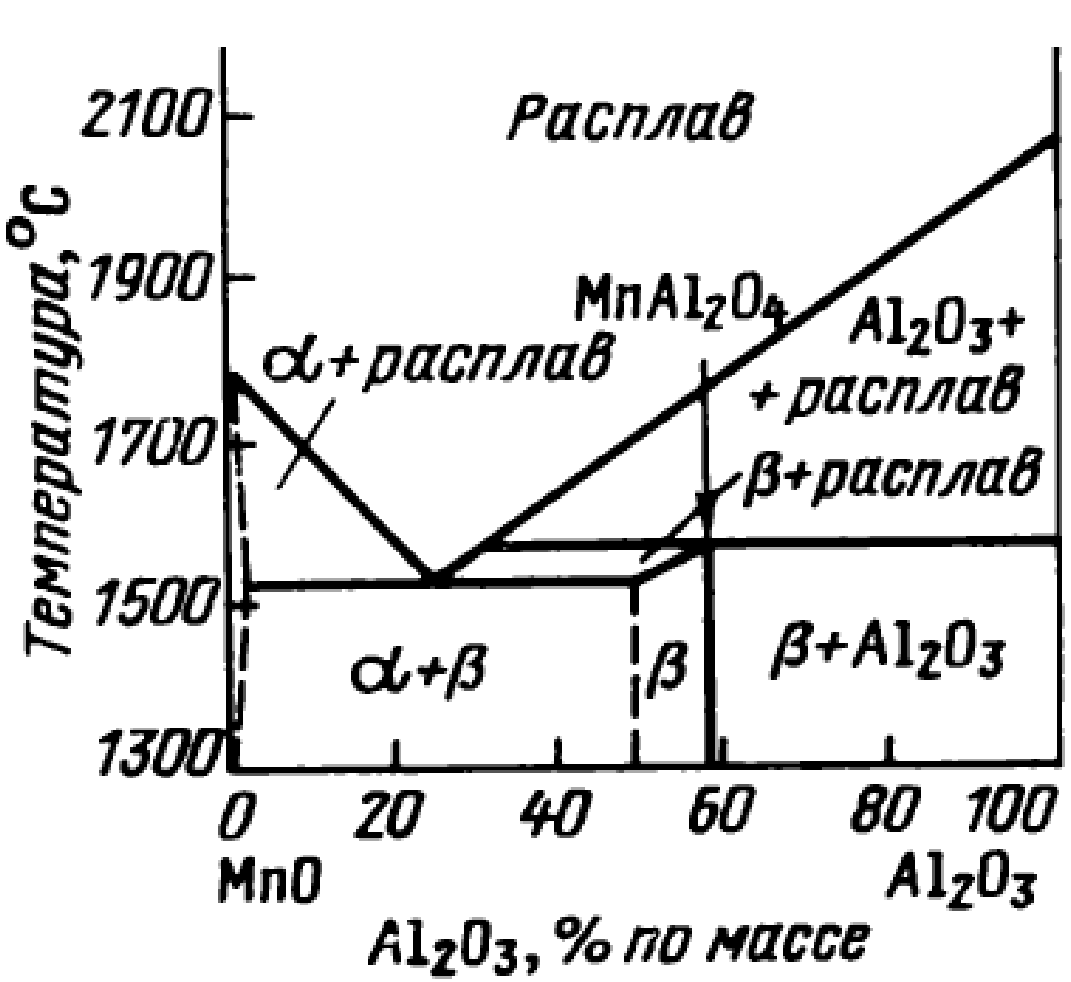
Regime of Metalization (Our experiments)



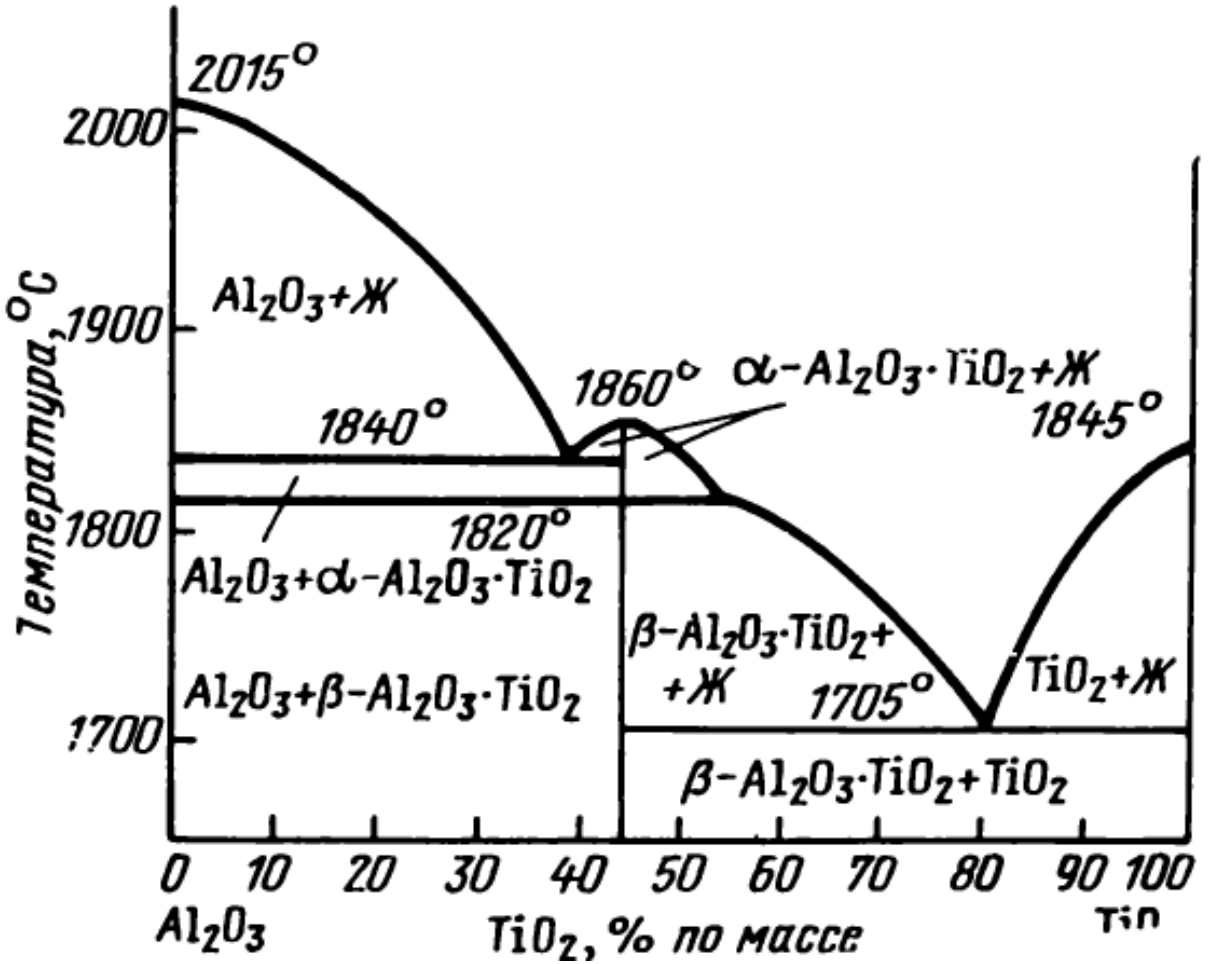
Phase Diagram Mn-Mo



Phase Diagram MnO - Al_2O_3



Phase Diagram TiO - Al_2O_3



Material Descriptions

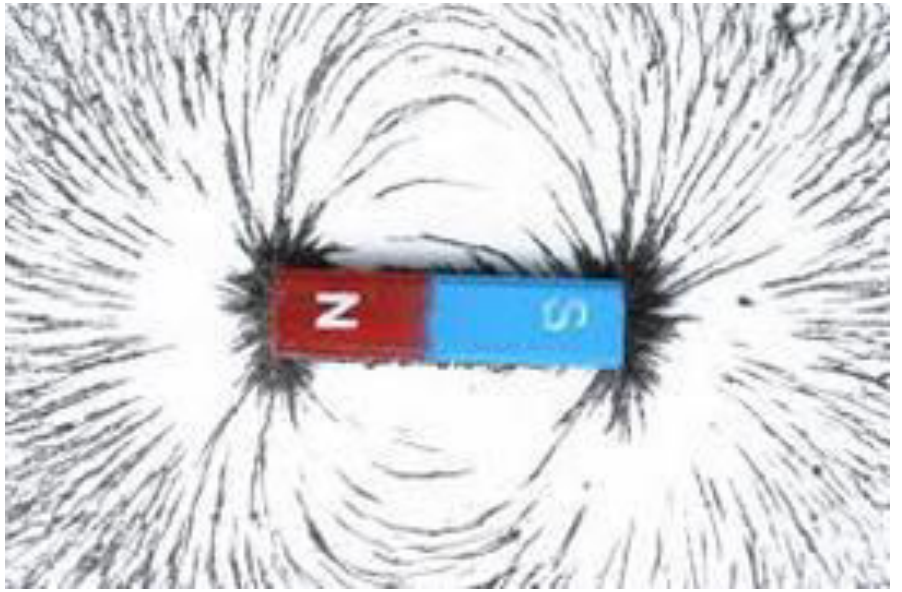
	Materials	Melting Point (°C)	Density ($g \cdot cm^{-3}$)	Thermal Expansion $\mu m \cdot m^{-1} \cdot K^{-1}$ (at 25 °C)	Description	Brinell Hardness (MPa)
1	Mo	2623	10.28	4.8	paramagnetic	1370–2500
2	Mn	1246	7.21	21.7	paramagnetic	196
3	Ni	1455	8.908	13.4	Ferromagnetic	667–1600
4	Cu	1084.62	8.96	16.5	diamagnetic	235–878
5	Ti	1668	4.506	8.6	paramagnetic	716–2770
6	Ag	961.78	10.49	18.9	diamagnetic	206-250
7	Kovar	1450		5.5		

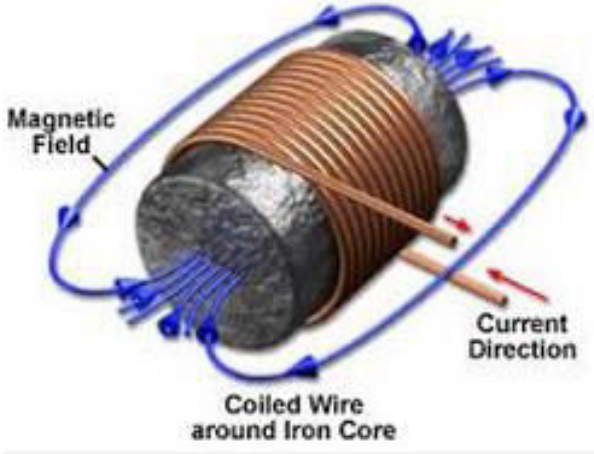
Braze Material	**Interval (°C)**
Ag Cu 28	780
Ag Cu 26,6 Pd 5	807 – 810
Ag Cu 21 Pd 25	910 – 950
Au Ni 18	950
Cu Ge 10	900 – 1000
Au Cu 65	1000 - 1020

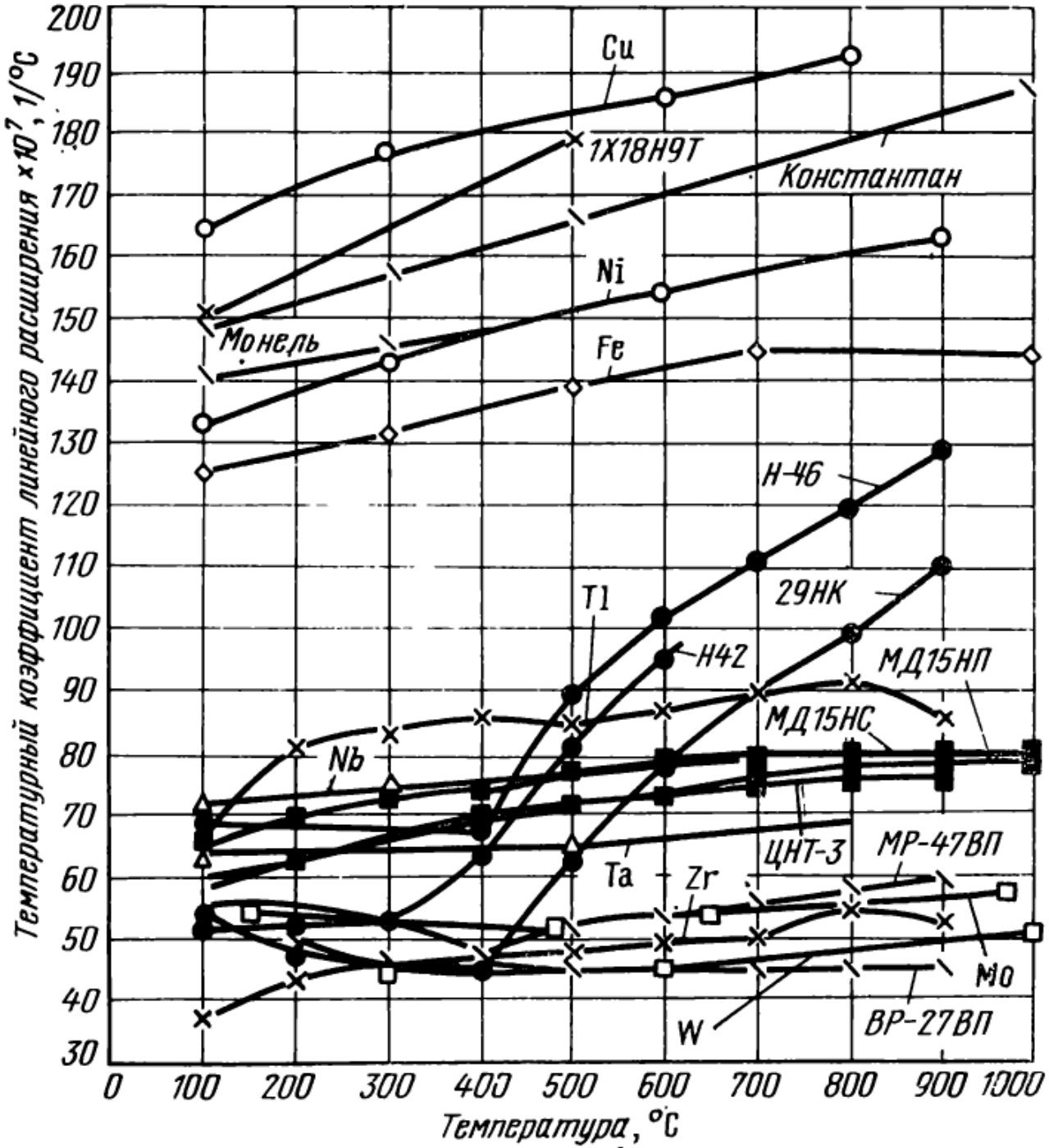
Curry point for different materials

	Tc, K
Fe	1043
Co	1403
Ni	631
Gd	289
Tb	223
Dy	87
Ho	20
Er	19,6

	Tc, K
Fe_3Al	743
Ni_3Mn	773
$FePd_3$	705
$MnPt_3$	350
$CrPt_3$	580
$ZnCMn_3$	353
$AlCMn_3$	275
TbN	43
DyN	26
EuO	77
MnB	578
$ZrZn_2$	35
Au_4V	42—43
Sc_3In	5—6





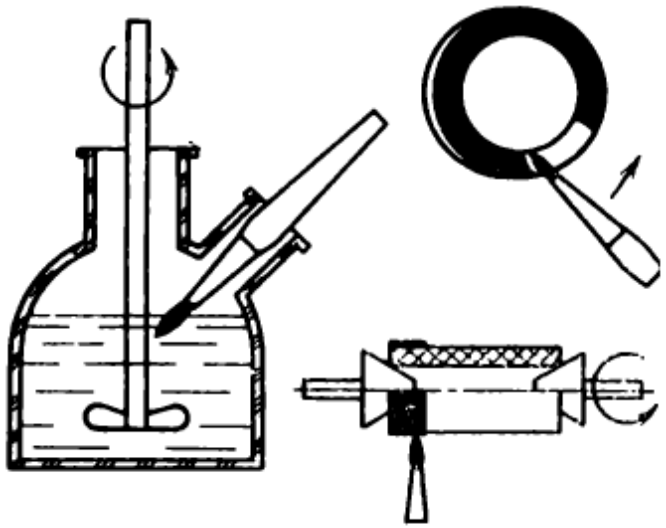


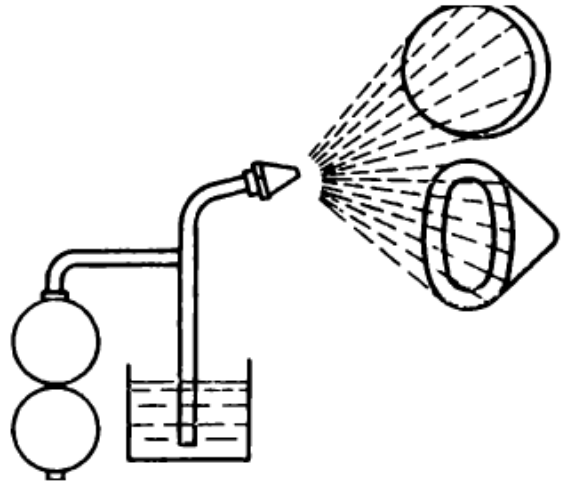
Coefficient of
Thermal expansion

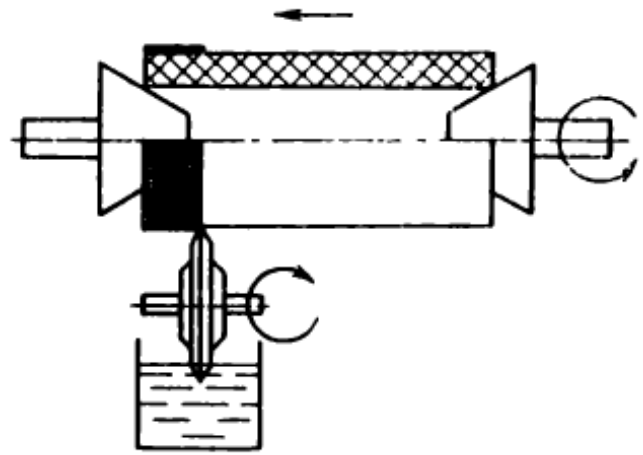
Our Experiments

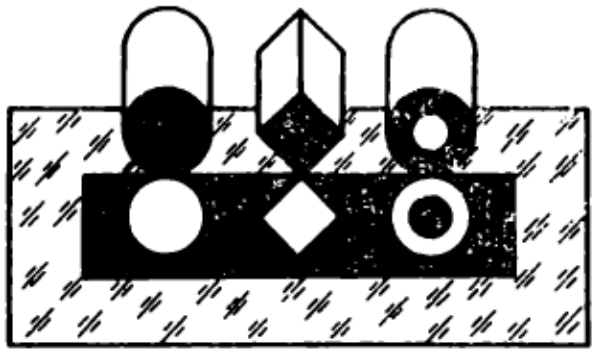
Cutting, grinding, etc.

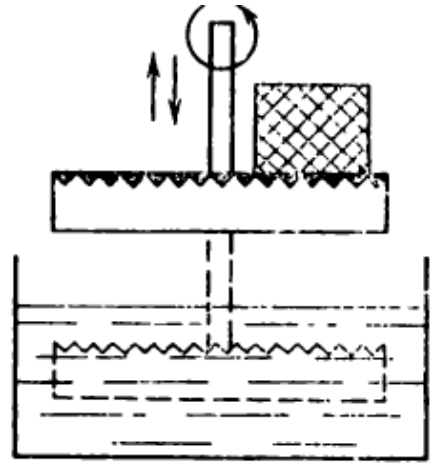
Methods of metallization layers

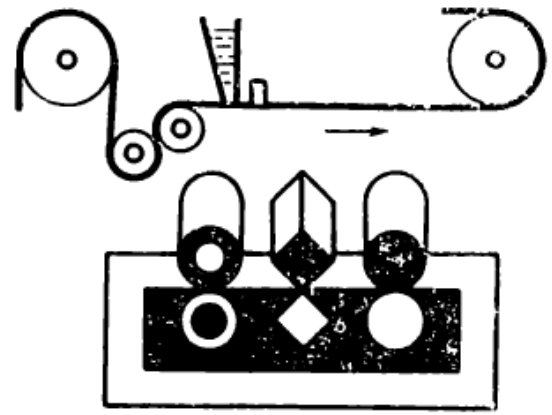






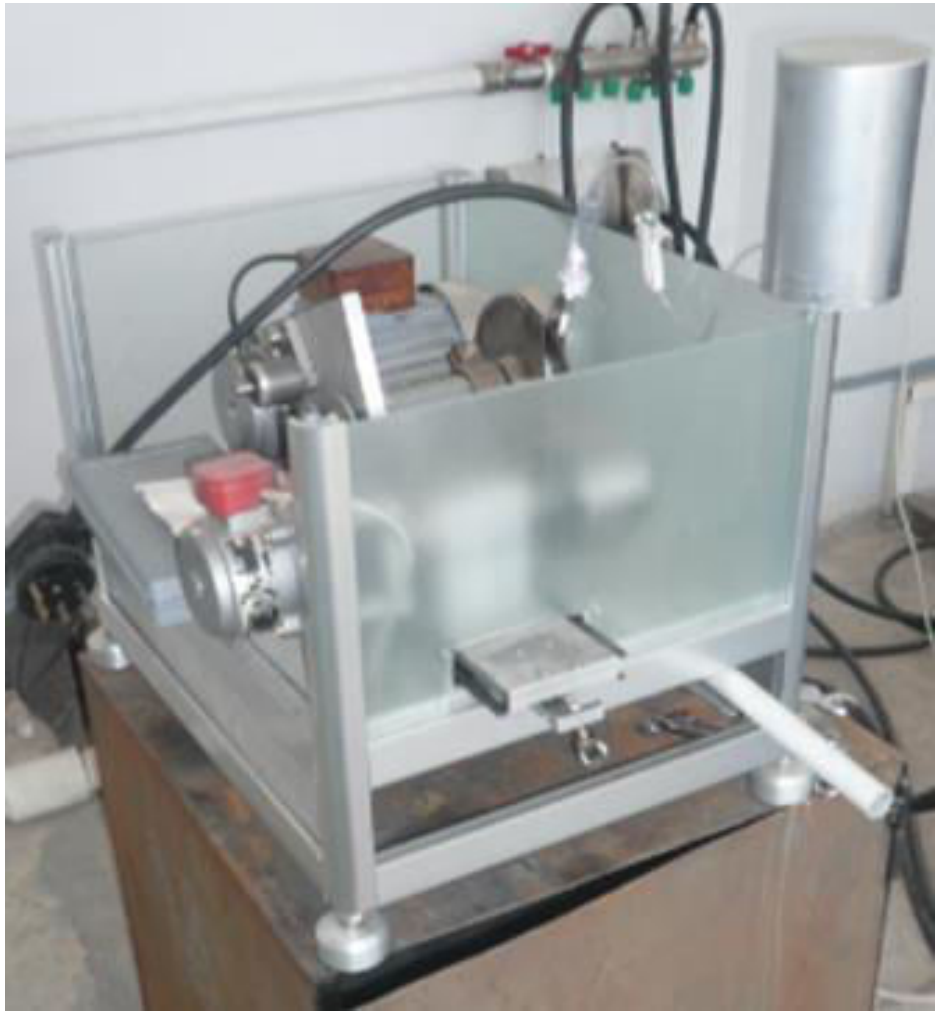






Regime of Metalization

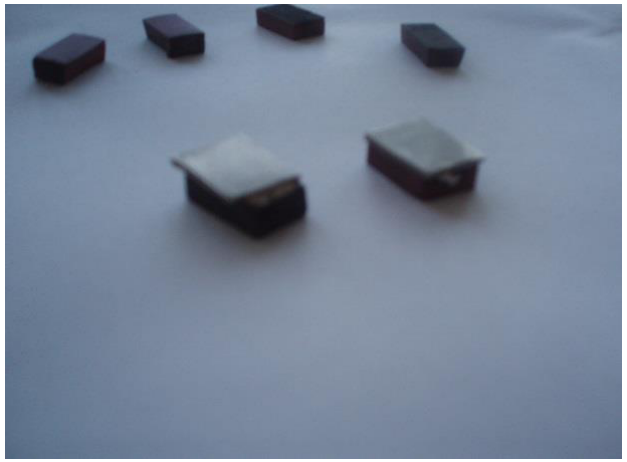
Coating type	Temperature ^0C	duration	Cooling speed ^0C/min	Environment
Mn, Mo (22X, 22XC, A-995, M-7)	1 270 – 1 400	20 - 40	5 - 10	$N_2 : H_2 = 2 : 1$ $N_2 : H_2 = 3 : 1$ dew point +15 - +25^0C
Mo, Mn, Si	1 280 – 1 320	40	Cooling - 4.2	$N_2 : H_2 = 3 : 1$ dew point +15 - +25^0C



Ceramic Cutting Machine

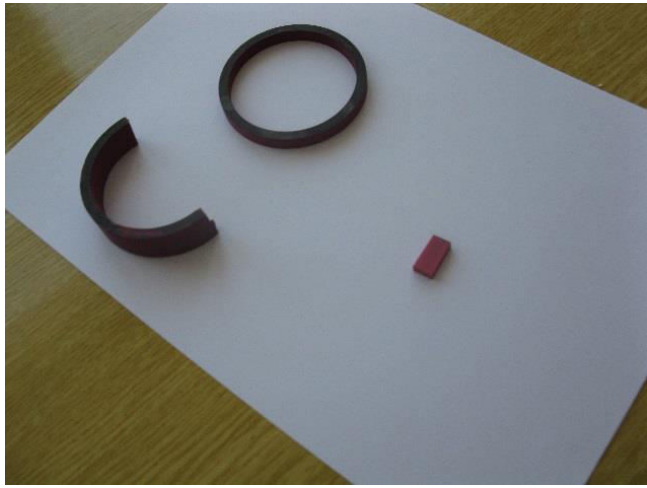
FRIALIT® - F99,7

Our Experiments

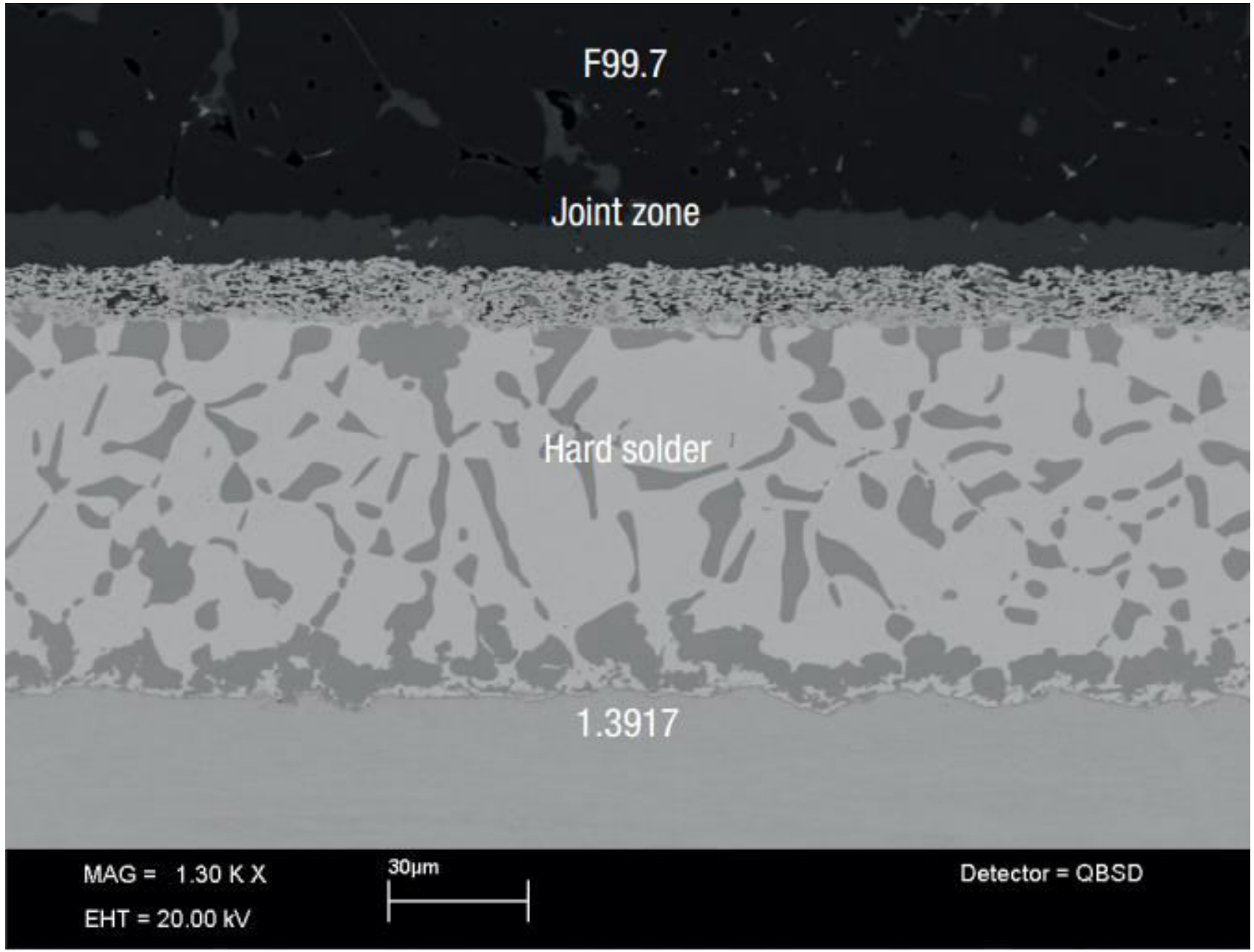





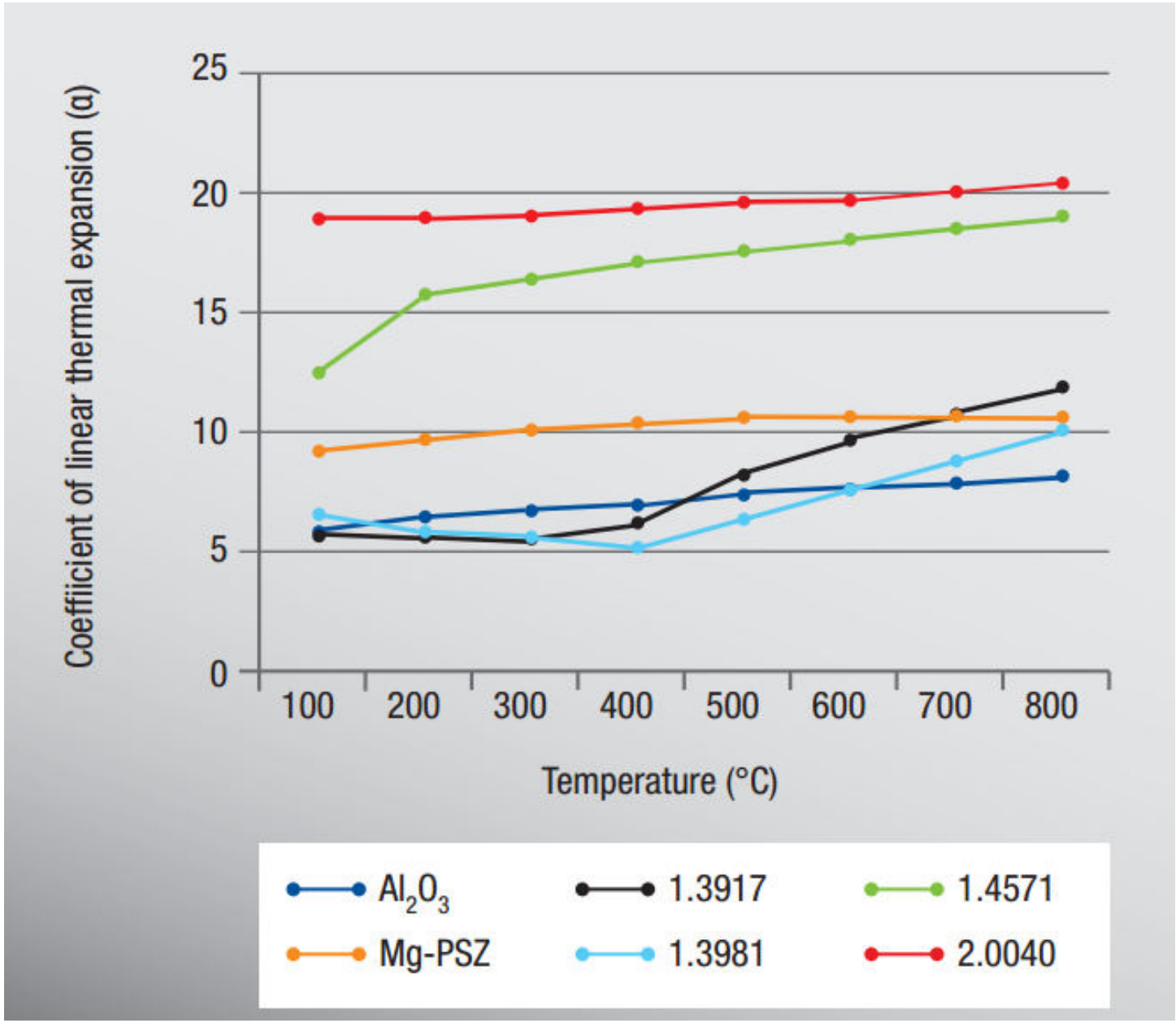




Ceramic - Type		Metallization Past	Concentration %
Cearmaic Steatit, К-1		Mo : Fe	98 : 2
Ceramic Forsterit, ФС-5Л, АФ-555		Mo : Mn Mo : TiH_2 : Al_2O_3	96 : 4 63.8-74,0.8-6.1
Alumina silicate ceramic		WC : TiC : Fe	60 : 10 : 30
Alumina	22Х, 22ХС	Mo Mo : Mn Mo : Mn : Si Mo : Mn : TiH_2 Mn : Mo_2B_5 , Mo	100 80 : 20 80:20 (+5) 80 :20 : 10 20 : 10-15 : 70-65
	М7	Mo ; Mn : MoB Mo : Mn : $MoSi_2$ Mo ; Mn : С-48	62.5 : 20 : 17.5 77 : 20 : 3 75 : 20 :5
	ВГ - 4	Mo : Mn : Si	75-78 : 20 : 5-3
	А-995	Mo : Mn : Mo_2B_5 : БД-22	74 : 15 : 5 : 6
	Sapphirite	Mo : Mn : V_2O_5	75 : 20 : 5
	Policore	Mo : Mn : Si	80 : 20 : (+5)
Monocrystal	sapphire	Mo : Mn : Mo_2B_5 : БВ22	74 : 15 : 5 : 6
	Rubine	Mo : glass СТ-1	70 : 30
Beryllium ceramic		Mo : Mn : Si	80 : 20 : (+5%)


F99.7
Joint zone
Hard solder
1.3917
MAG = 1.30 K X
EHT = 20.00 kV
30µm
Detector = QBSD

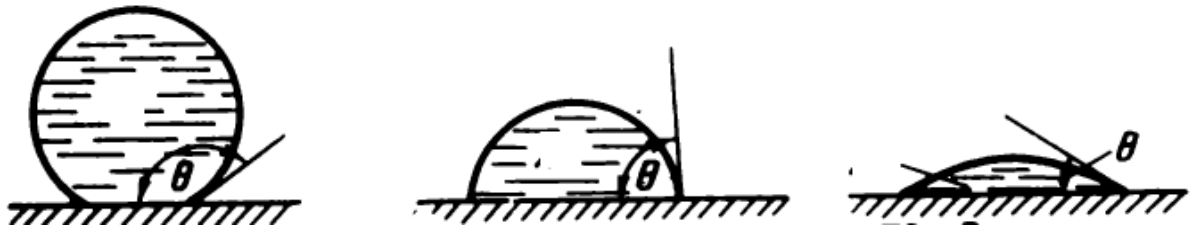
Thermal expansion of various oxide ceramic and metallic materials as a function of temperature.



Solder for Brazing

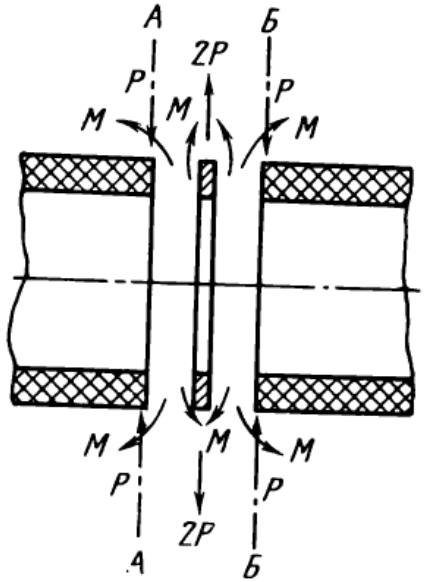
Марка припоя	Состав припоя, % по массе	Температура плавления, °С
ПСрМИн63	Ag:Cu:In = 63:27:10	685—710
ПСр72В	Ag:Cu = 72:28	779
ПСрМИн65	Ag:Cu:In = 65:30:5	770—800
ПСрМп66,5	Ag:Cu:Mn = 66,5:32,8:0,7	778—810
ПСрМПд68-27-5В	Ag:Cu:Pd = 68:27:5	807—810
ПСрМ83-17	Ag:Cu = 83:17	779—820
ПСрМПд59-31-10В	Ag:Cu:Pd = 59:31:10	830—850
ПСр50	Ag:Cu = 50:50	779—870
ПСр999	Ag = 99,99	960
ПСр85-15	Ag:Mn = 85:15	960—971
ПСрМПд52-28-20В	Ag:Cu:Pd = 52:28:20	890—920
ПЗлМСр75	Au:Cu:Ag = 75:12,5:12,5	892—900
ПЗлМН81,5	Au:Cu:Ni = 81,5:15,5:3	910—925
ПЗлН82	Au:Ni = 82,5:17,5	950
ПЗлМ50	Au:Cu = 50:50	955—970
ПЗлМ35	Au:Cu = 35:65	980—1 020
ПЗл	Au = 100	1 063
ПМТ28	Cu:Ti = 72:28	870
ПСрМН30	Cu:Ag:Ni = 65:30:5	830—900
ПСрМИн5	Cu:Ag:In = 85:10:5	900—950
ПЗлМ94	Au:Cu = 94:6	960—1 000
ПЗлМН35	Cu:Au:Ni = 62:35:3	980—1 020
ПМК2	Cu:Si = 98:2	1 000—1 050
Медь МВ или МБ	Cu = 99,99	1 083
ПМН10	Cu:Ni = 90:10	1 100—1 140
ПМН15	Cu:Ni = 85:15	1 120—1 180
ПМН25	Cu:Ni = 75:25	1 150—1 210
ПНПд60	Pd:Ni = 60:40	1 237
NiCr (ГОСТ 5632-57)	Ni:Cr = 80:20	1 400—1 420
Ni	Ni = 100	1 453
ПМГ12	Cu:Ge = 88:12	860—970
ПМГ9	Cu:Ge = 91:9	950—1 010
ПМГ5Б	Cu:Ge:B = 93,6:5,4:1	950—1 025

Wettability of Solder

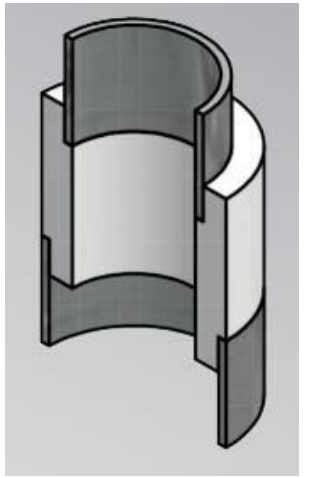


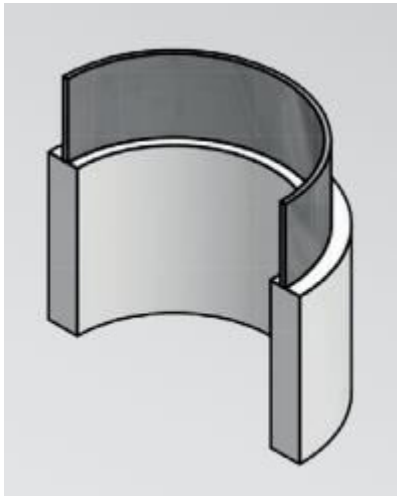
Solder	Surface Energy, эрг/см²
ПСр72В	950
ПЗлМ35	1 130
ПЗлМ50	1 190
ПЗлМН35	1 180
ПЗлМН81,5	1 260
ПЗлН82	1 410
ПЗлСрМ75	1 110
ПМГ12	1 200
Медь МВ	1 300

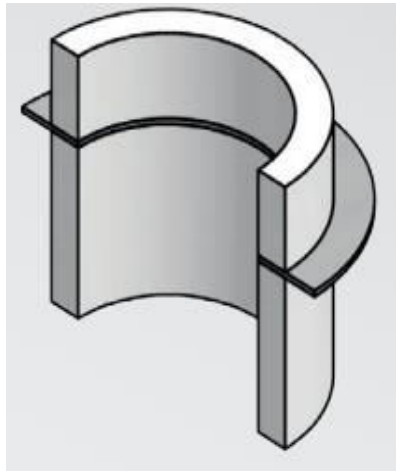
Mechanical stress calculation of metal - ceramic junctions

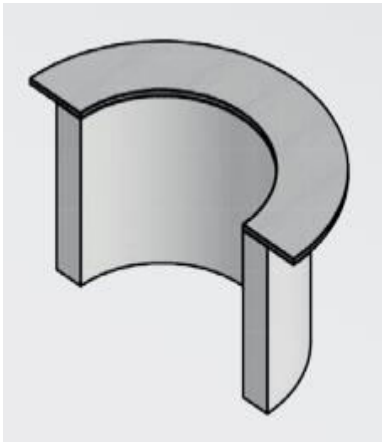


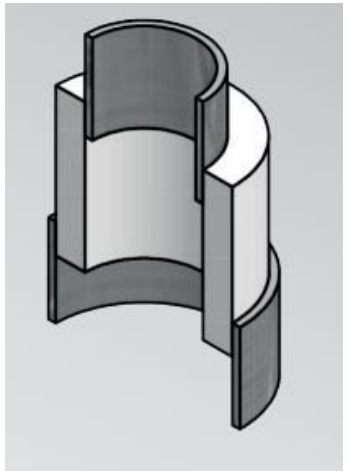
- Review of methods,
- Design – calculation – simulation,
- Experiment,
- Testing









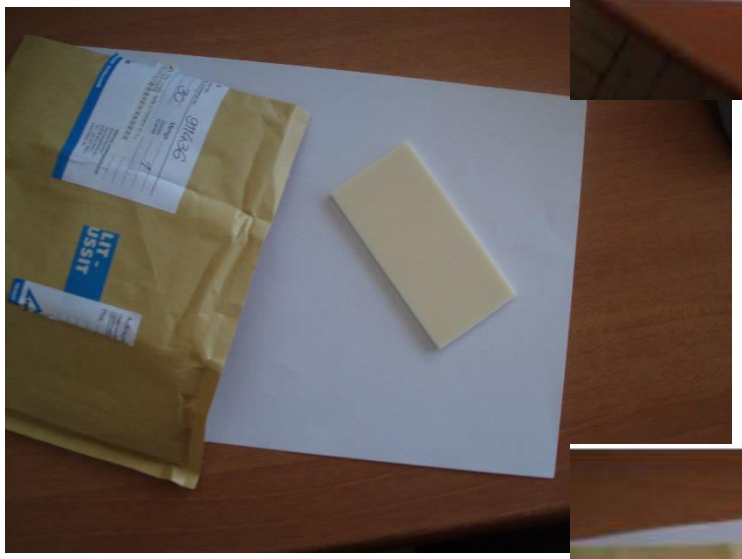


Types of junction

FRIALIT® - F99,7

Al_2O_3

Properties		Dimension	Specific Valu
Main component			a - Al_2O_3
Purity		Weight-%	> 99,5
Density		g / cm^3	3,9 - 3,95
Hardness	(Knoop, 100g)	GPa	23
Compressive strength		MPa	3500
Bending strength σ_m	DIN EN 843-1	MPa	350
Weibull modulus		-	> 10
Youngs modulus	static	GPa	380
Poisson number		-	0,22
Open porosity		Vol - %	0
Maximal operating temperature	air	℃	1950
Coefficient of expansion	20 - 1000℃	10^{-6} / K	8,5
Specific heat	20 ℃	J / (kg * K)	850
Thermal conductivity	20 ℃	W / (m * K)	34,9
	1000 ℃	W / (m * K)	6,8
	1500 ℃	W / (m * K)	5,3
Specific electrical resistance	20℃	Ω * cm	10^{15}
	1000℃	Ω * cm	10^{7}
Electric dielectric strength	20 ℃	kV / mm	> 30
Dielectric coefficient	70 MHz	-	10
	180 MHz	-	9,9
	30 - 40 GHz	-	9,8
Dielectric loss angle	70 MHz	-	270 * 10^{-4}
	180 MHz	-	150 * 10^{-4}
	30 - 40 GHz	-	20 * 10^{-4}
Colour			ivory

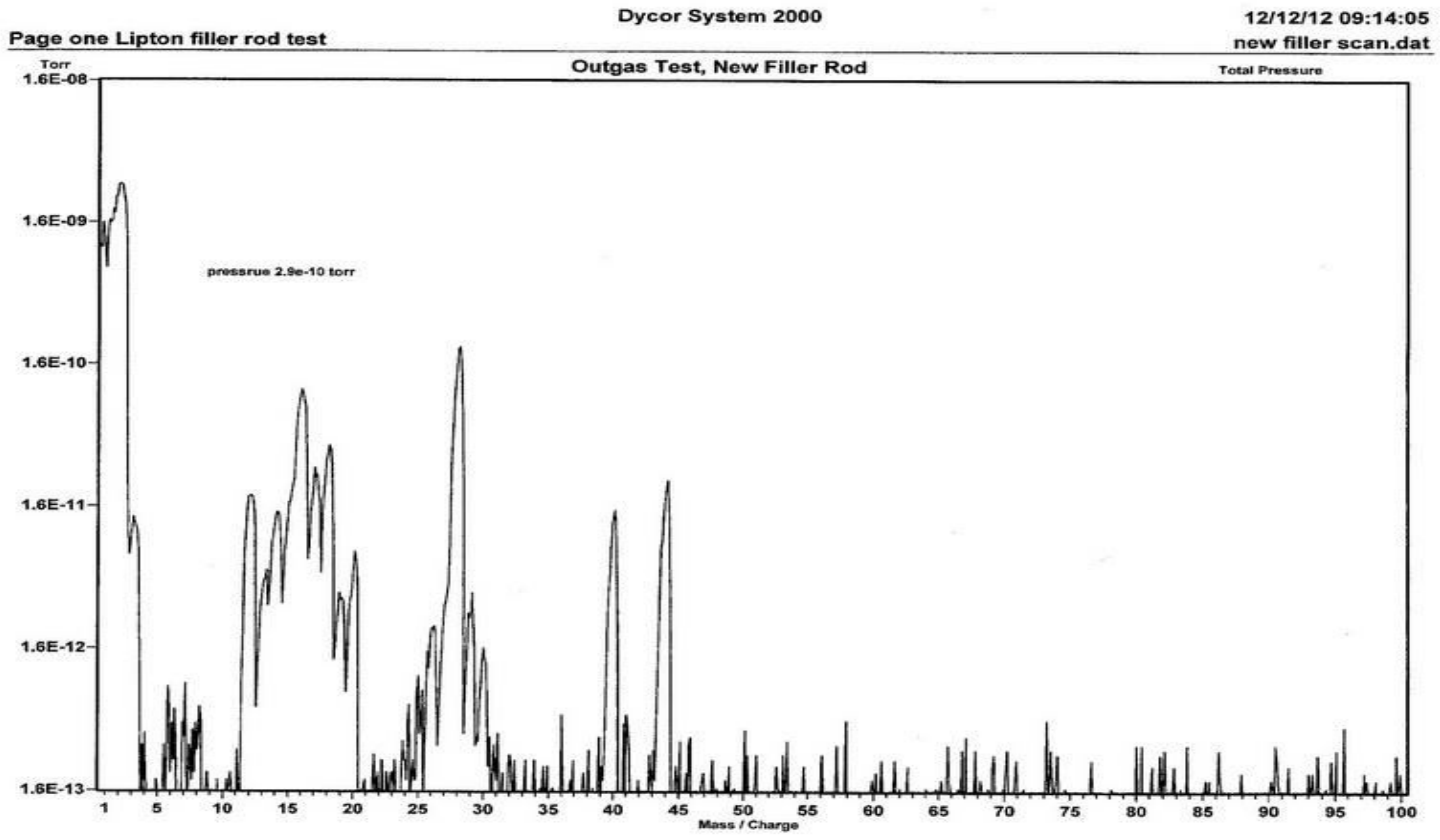


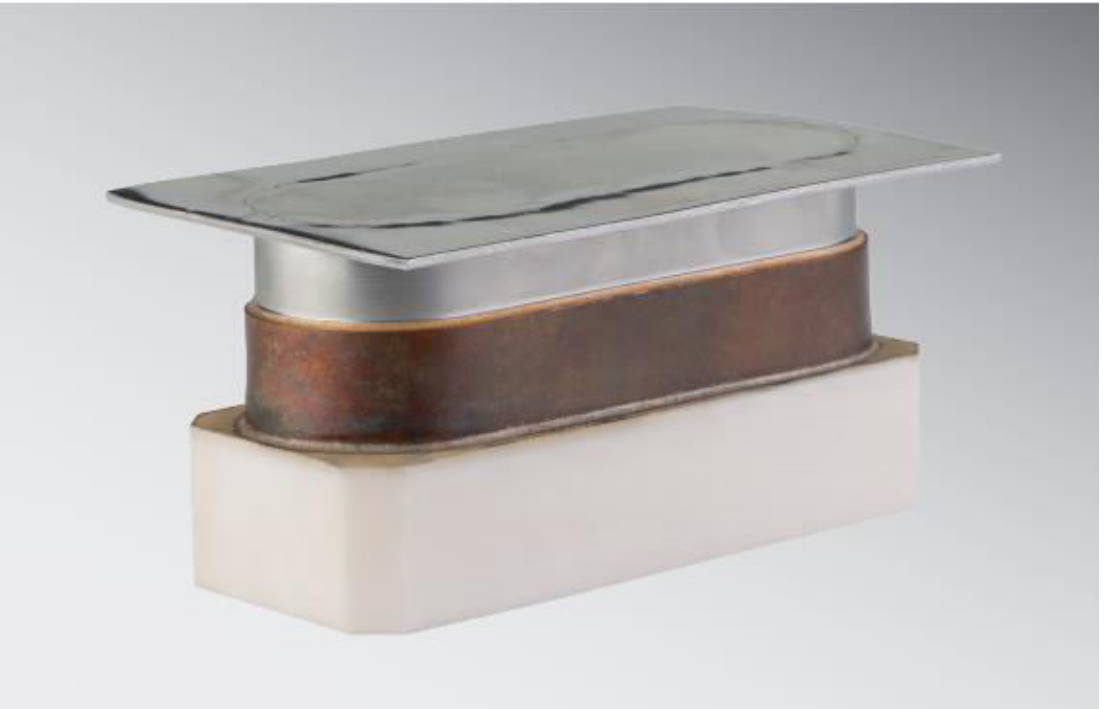
Kovar

Fe	Ni	Co	C	Si	Mn
balance	29%	17%	< 0.01%	0.2%	0.3%

Property	sintered	HIPed
Density / g cm^3	8.0	8.35
Hardness / HV1	160	150
Youngs Modulus / GPa	138	138
reduction of area at fracture / %	30	30
yield strength / MPa	270	270
thermal conductivity / W/Km	17	
Curie Temperature / °C	435	
electrical resistivity Ω mm^2 / m	0.49	
specific heat J/gK	0.46	
thermal expansion coefficient/10^{-6} K^{-1} (25 – 200 °C)	5.5	
(25–300 °C)	5.1	
(25–400 °C)	4.9	
(25–450 °C)	5.3	
(25–500 °C)	6.2	

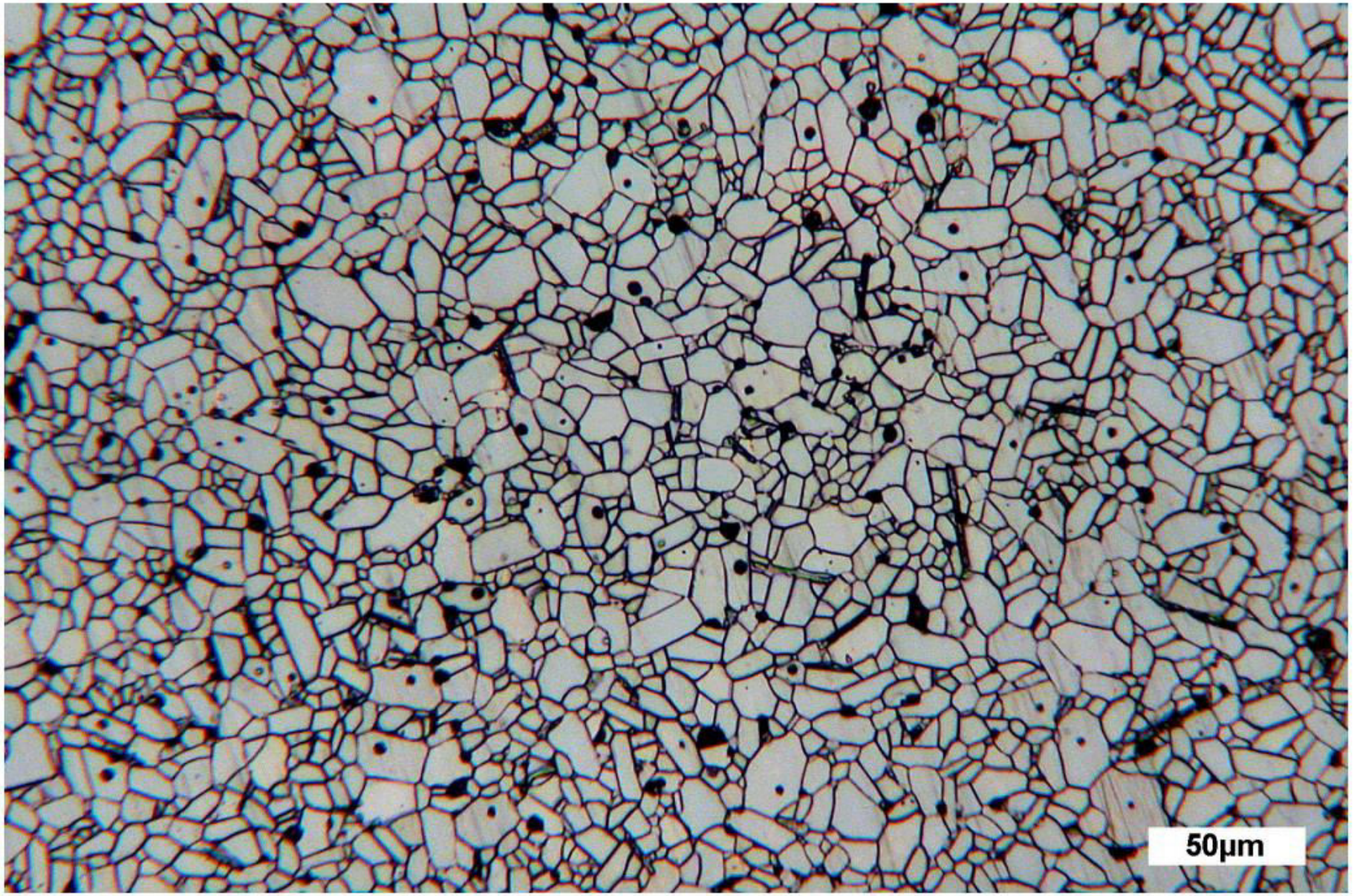
Mass Spectrometry
Residual gas analysis in high vacuum



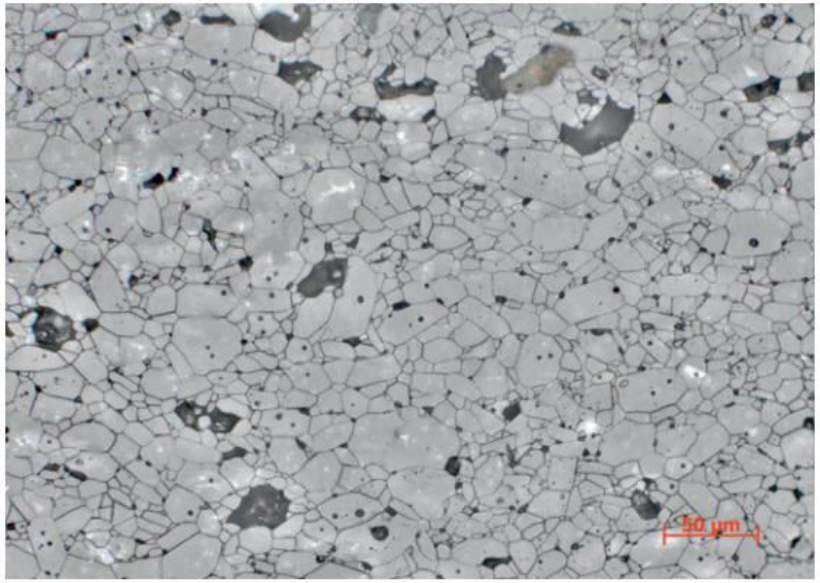


FRIATEC

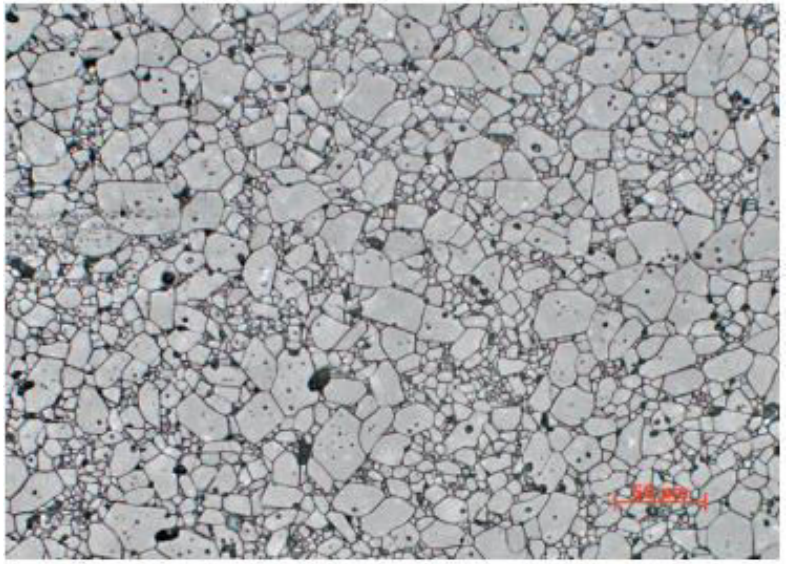
Microstructuries



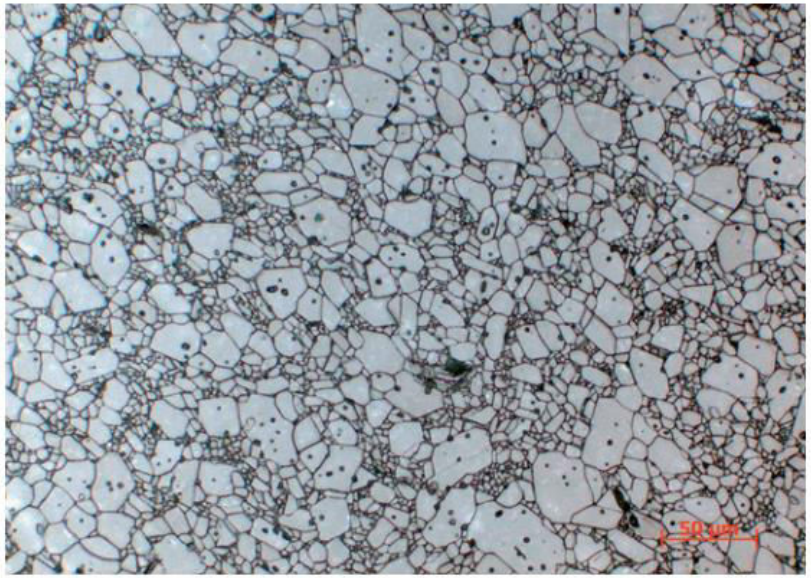
Microstructure of DEGUSSIT AL23 material



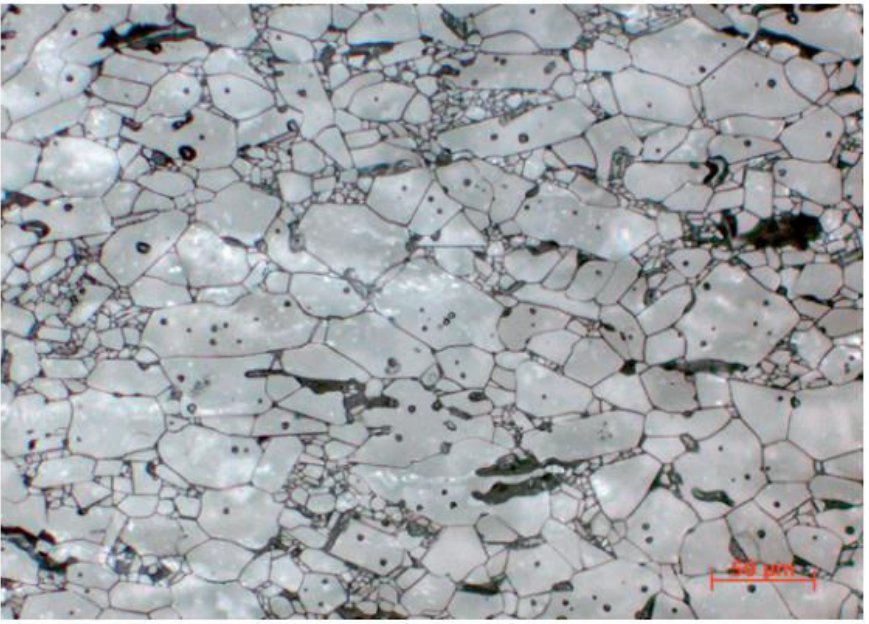
Microstructure AL23



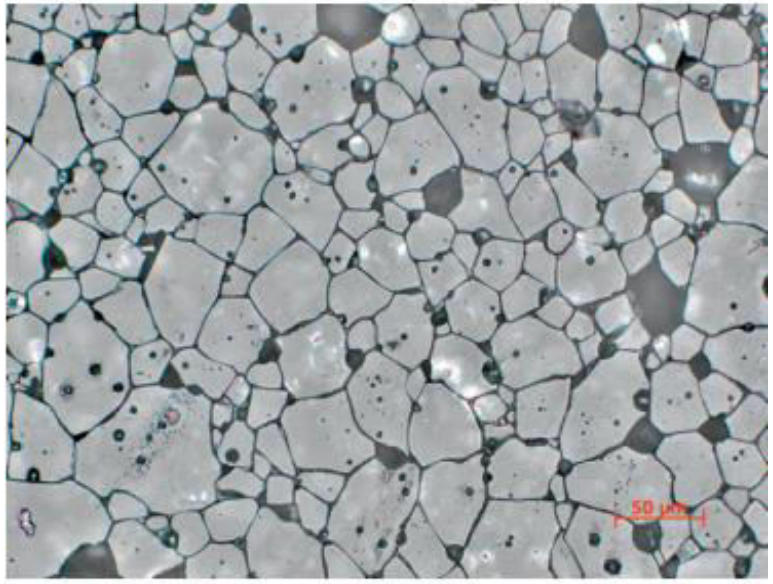
Microstructure U



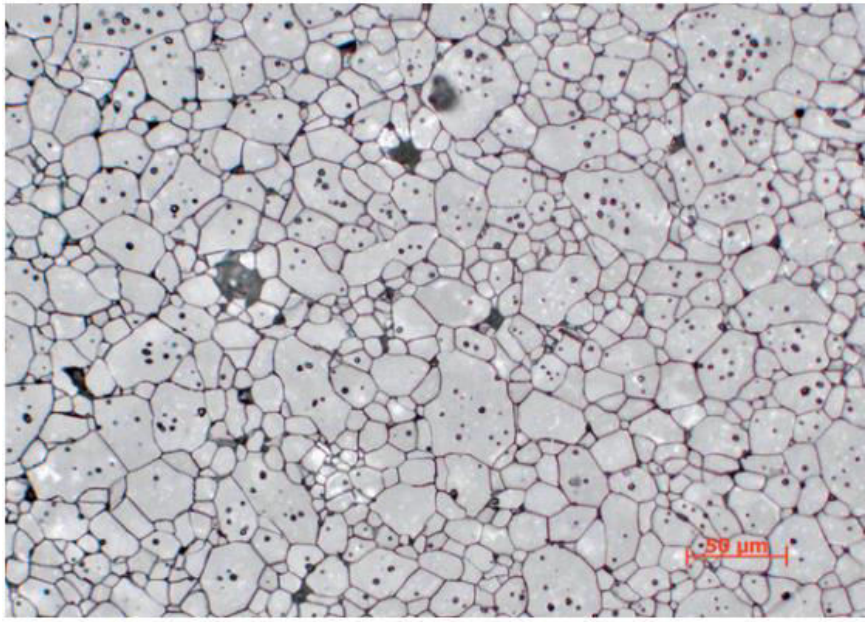
Microstructure D1



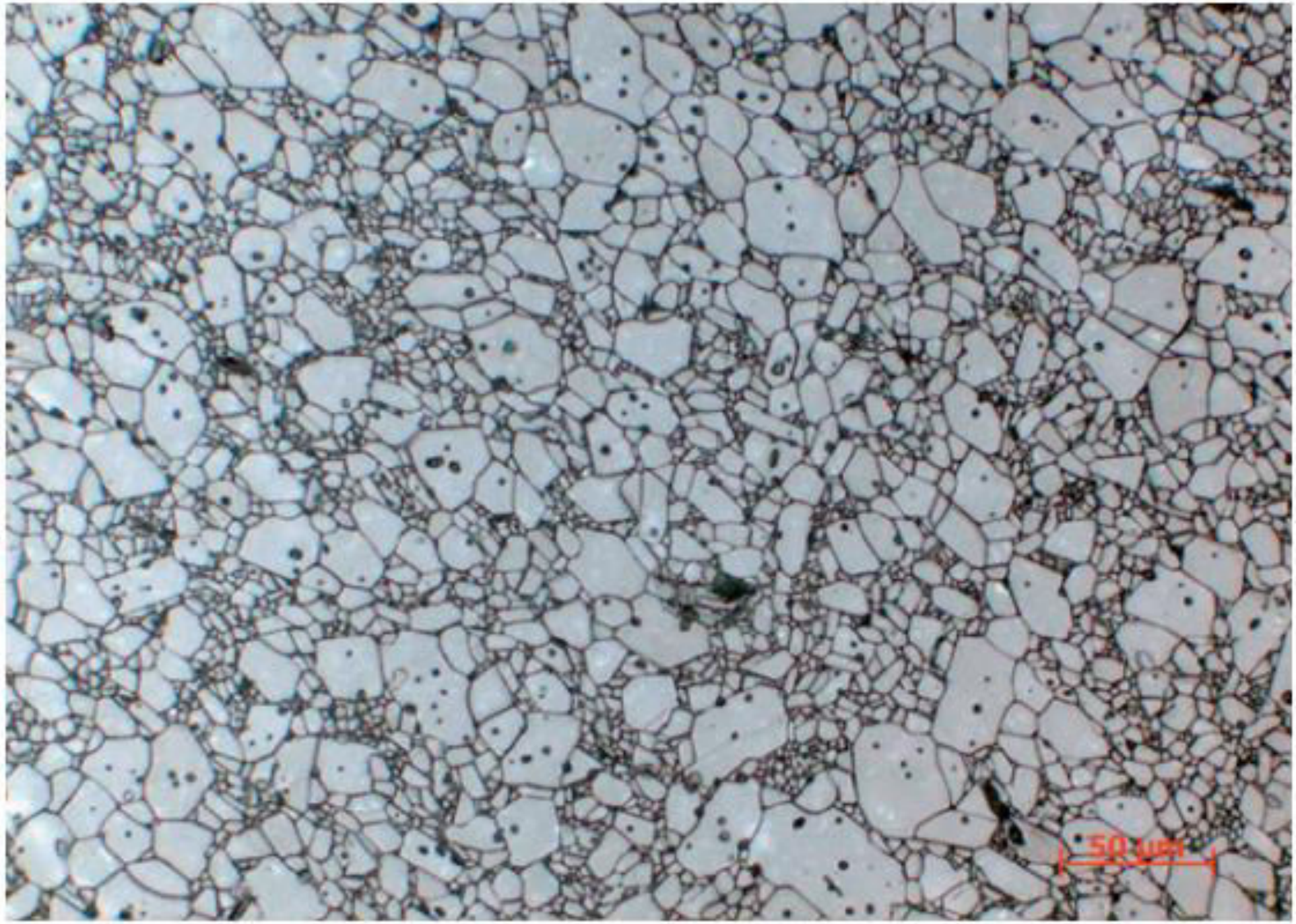
Microstructure D2

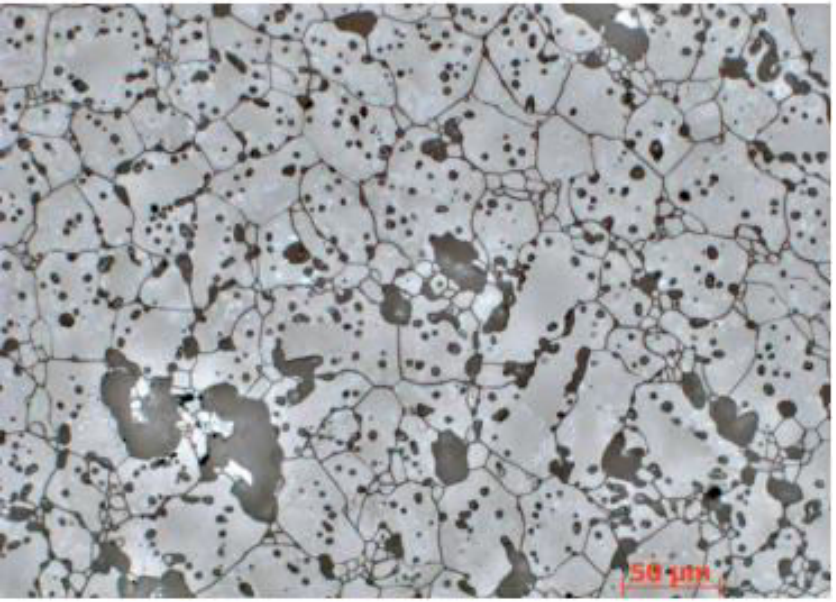


Microstructure D3, bottom

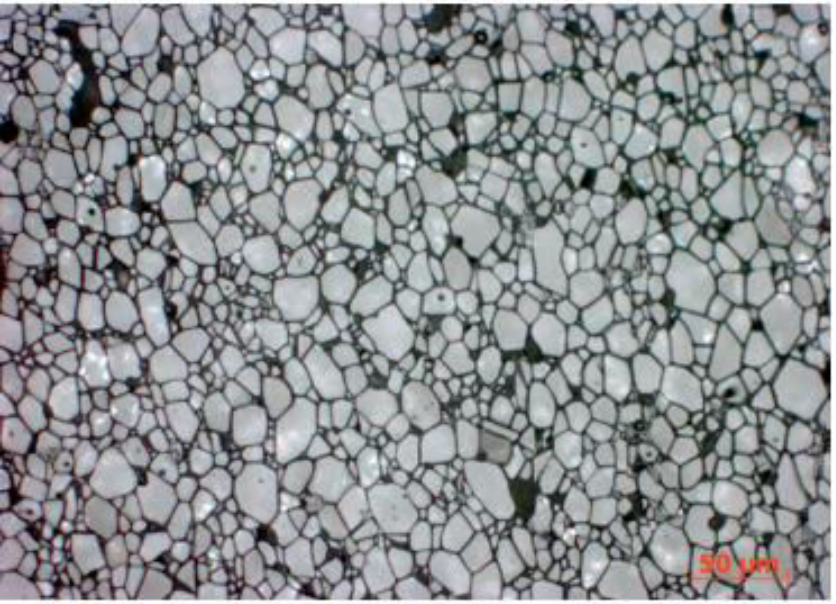


Microstructure D3, tube


50 µm

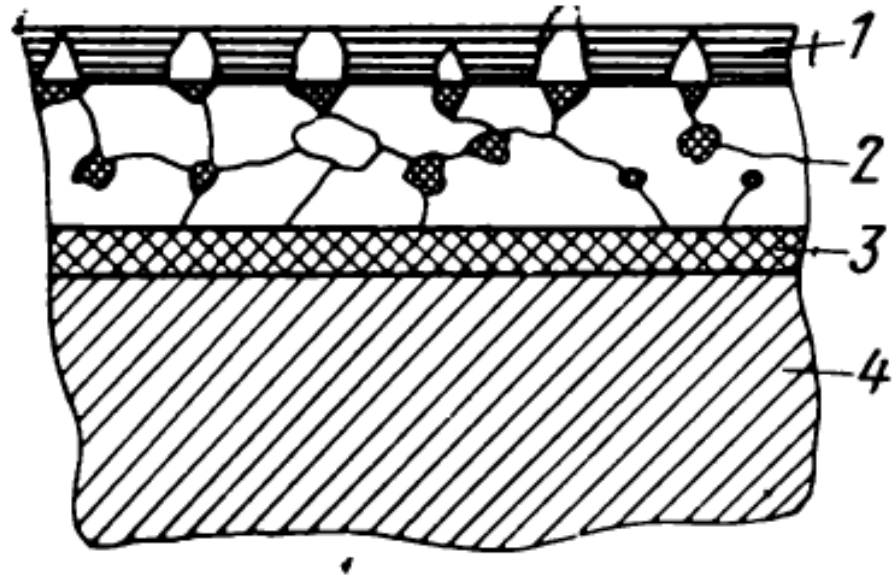


Microstructure AL24



Microstructure C

Type	Purity (mass - %)	Density (g/cm^3)
C	98,43	3.75
Al23	99,70	3.81
D1	99.70	3.84
D2	99.73	3.91
D3	99.74	3.90
U	99.74	3.93
AL24	99.77	3.59



Никелевый слой на поверхности металлизированной керамики.

1 — никелевый слой; *2* — металлизационный слой (Мо); *3* — переходный слой; *4* — керамика.

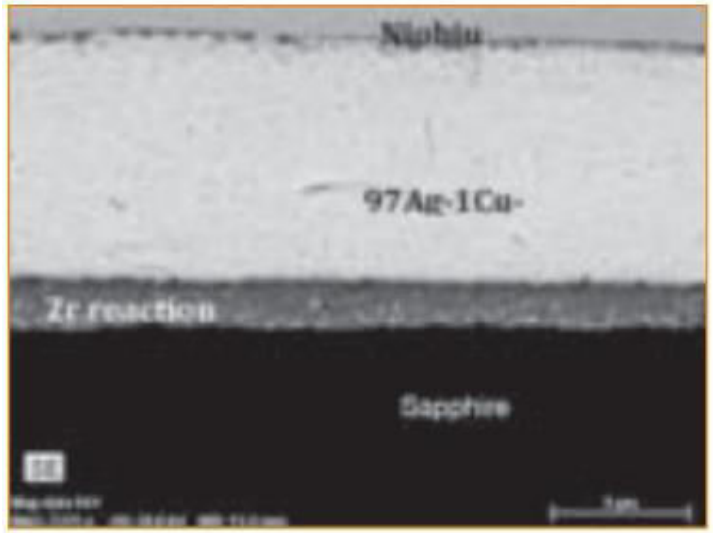
Windows for Particle Accelerators

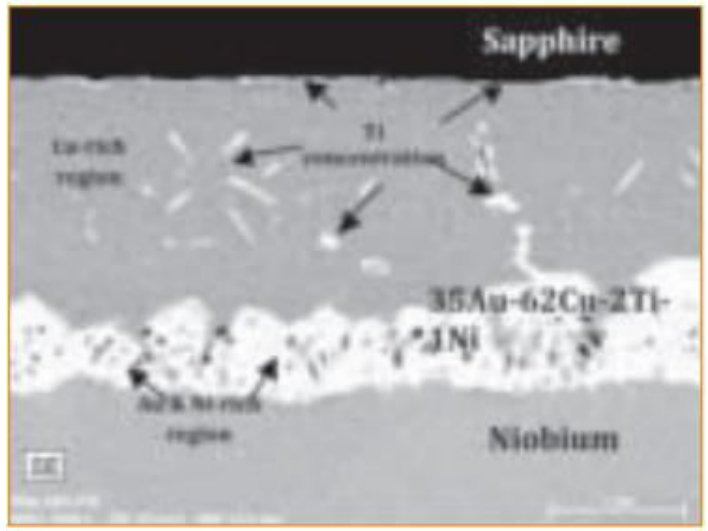
- Diamond windows,
- Silica windows,
- Sapphire windows,
- Kapton Windows,
- Titanium Windows,
- Beryllium Windows,
- Alumina Windows,
- Glass Windows
- …..

7056 Glass Viewport
Kovar sleeve

Sapphire windows

Direct Brazing of Sapphire to Niobium





Brazing Filler Metal	Solidus Temperature (°C)	Liquidus Temperature (°C)
BAu-3	1000	1030
35Au-62Cu-2Ti-1Ni	983	1020
Cusil-ABA	780	815
97Ag-1Cu-2Zr	940 (est.)	962 (est.)

Niobium to Sapphire

Series		Material		Braze Filler Metal		Peak Soak		Leak Check	Average
Number	Quantity	Tensile Buttons	Interlayer	Thickness	Type	Thickness	Temperature (°C)	Time (Min)	Number Hermetic	Tensile Strength (MPa)
1	6	Nb	sapphire	0.41 mm	BAu-3	0.076 mm	1060	2	6/6	34.7
2	4	Nb	sapphire	0.41 mm	Nicoro+2% Ti	0.076 mm	1060	2	2/4	21.5
3	4	Nb	sapphire	0.41 mm	97Ag-1Cu-2Zr	0.076 mm	985	5	4/4	37.9
4	5	94ND10	niobium	0.25 mm	Cusil-ABA	0.076 mm	855	5	5/5	102.0
5	5	94ND10	niobium	0.25 mm	BAu-3	0.076 mm	1060	2	5/5	55.1
6	5	94ND10	niobium	0.25 mm	BAu-3	0.076 mm	1060	240	5/5	95.5
7	5	94ND10	niobium	0.25 mm	BAu-3	0.076 mm	1260	120	0/5	39.4
8	5	Nb	sapphire	0.25 mm	BAu-3	0.076 mm	1060	240	5/5	5.9
9*	4	Al-500	niobium	0.25 mm	BAu-3	0.076 mm	1058	3	2/4	61.0

New Diffusion Brazing Method


Հայաստանի Հանրապետություն

ԱՐՏՈՆԱԳԻՐ

№ 2883 A

Հայաստանի Հանրապետության
մտավոր սեփականության գործակալությունը
օրենքի համաչափ պահից
Բարդ երկրաչափական ձև ունեցող տարասեռ դետալների
դիֆուզային զոդման եղանակ
գյուտի սույն արտոնագիրը

ԱՐՏՈՆԱԳՐԱՏԵՐ «Քենդլ» սինքրոտրոնային հետազոտությունների ինստիտուտ, հիմնադրամ

ՀԵՂԻՆԱԿ(ՆԵՐ) Վարդան Շավարշի Ավագյան, Վահագն Վանիկի Վարդանյան

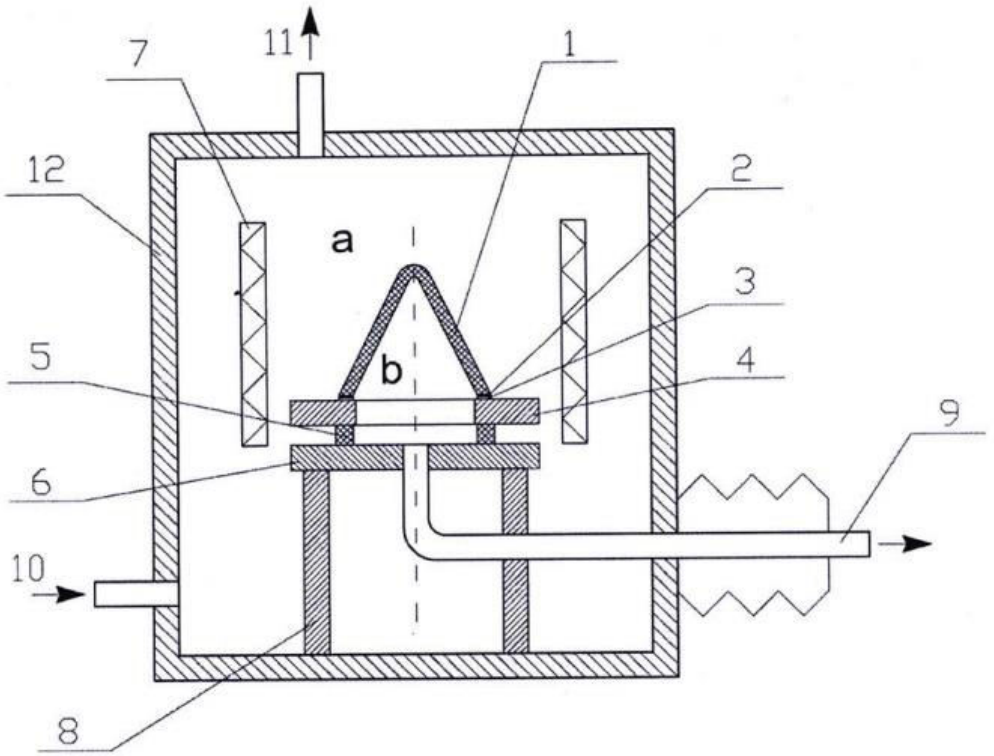
ՀԱՅՏԻ № AM20140053

Ներկայացման թվականը 17.04.2014

Գյուտի առաջնությունը 17.04.2014

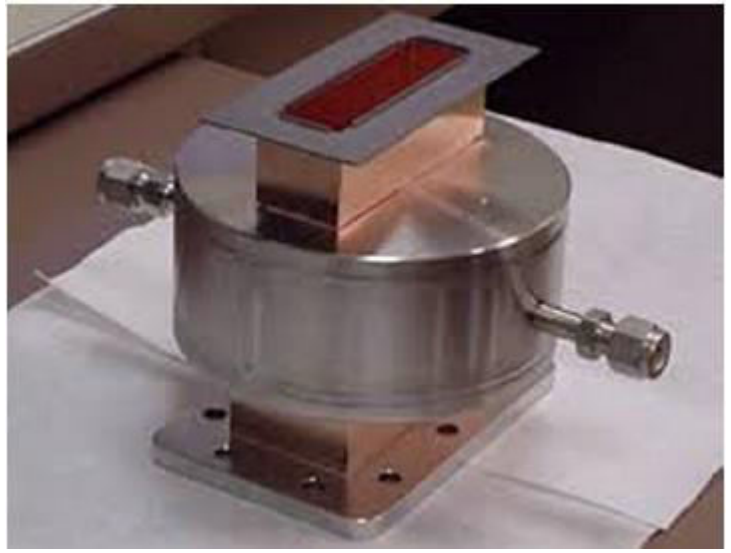
Գրանցված է պետական գրանցամատյանում 25.11.2014

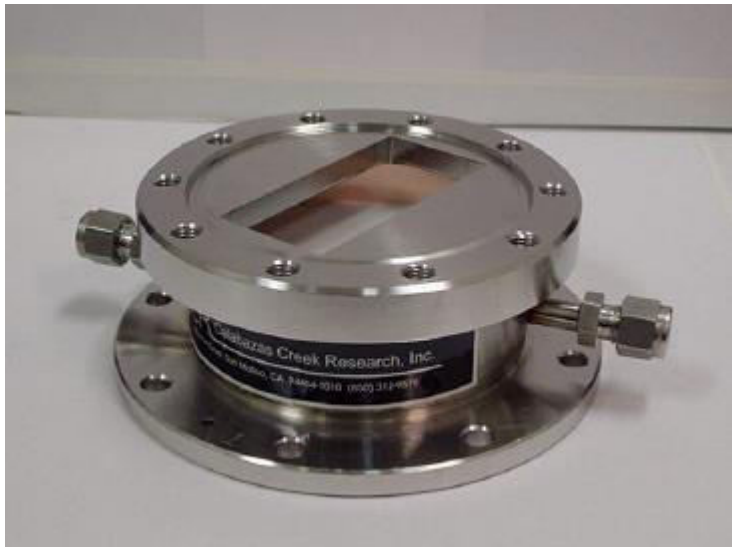
Գործակալության պետ

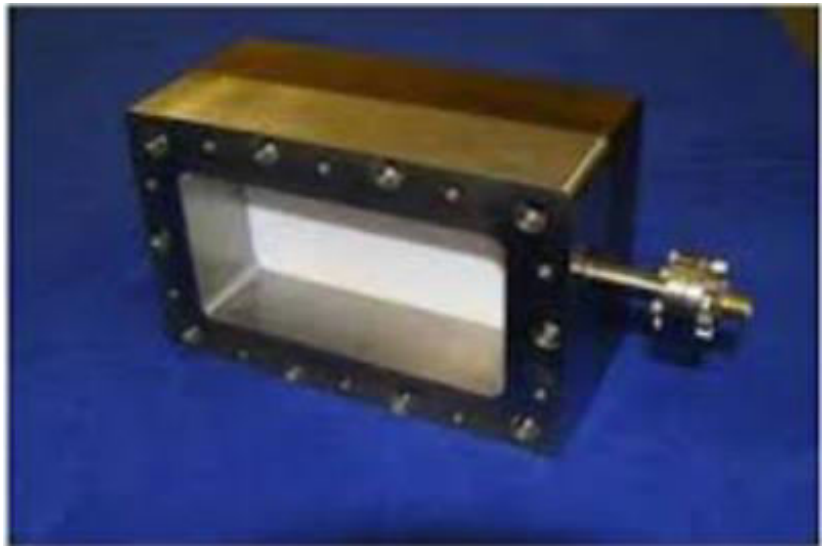


Diffusion brazing methods of difficult geometry dissimilar details.
Patent number – AM201453
Vardan Shavarsh Avagyan, Vahagn Vanik Vardanyan
Intellectual Property Agency of the Republic of Armenia

RF Windows



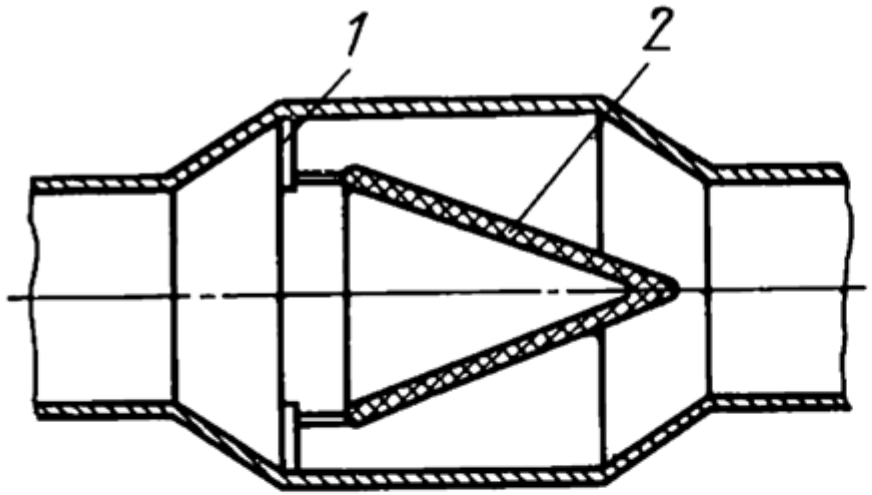


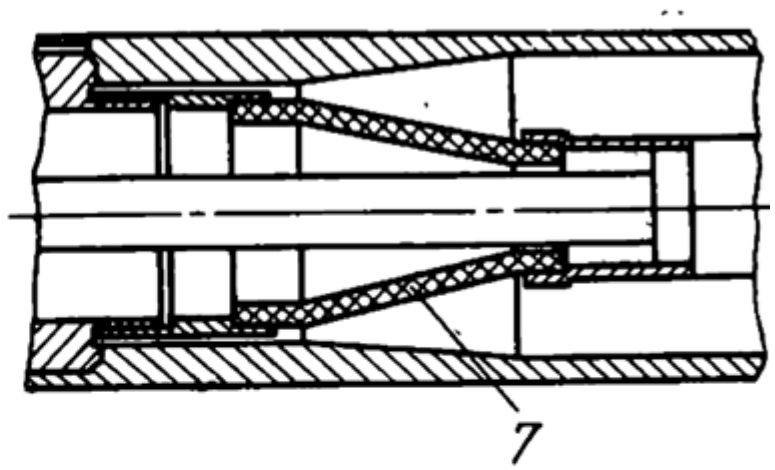


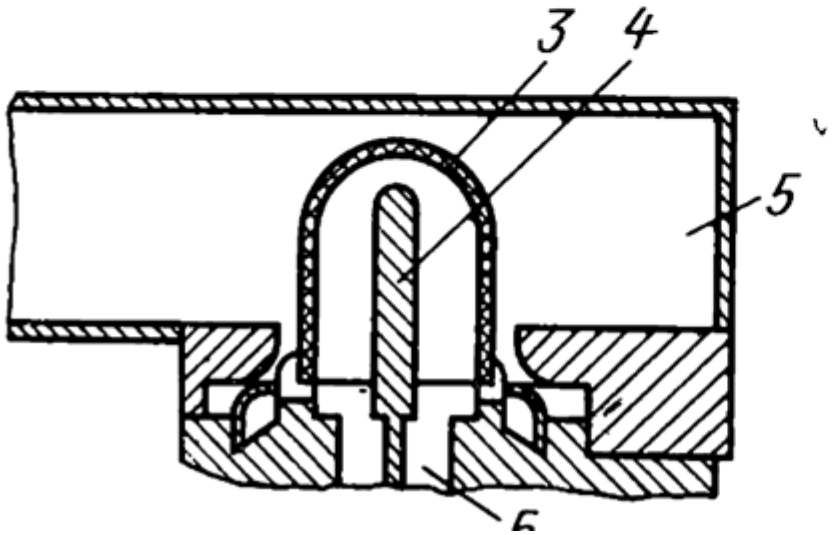
Main Requirements.

- High Mechanical strength,
- Outgassing Low Level,
- Reliable during long time,
- Thermal shock resistance,
- Necessary Electro – Magnetic Parameters,
- Low Material Penetration,
- Necessary Thermal Conductivity,
- Dimensional stability.

RF Windows



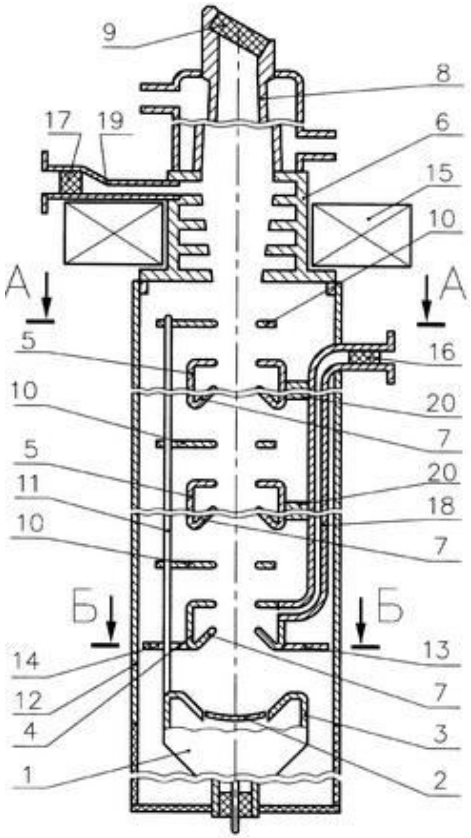




1. waveguide, 2. coaxial
3. Waveguide - coaxial

Parameters of ceramic for RF Windows

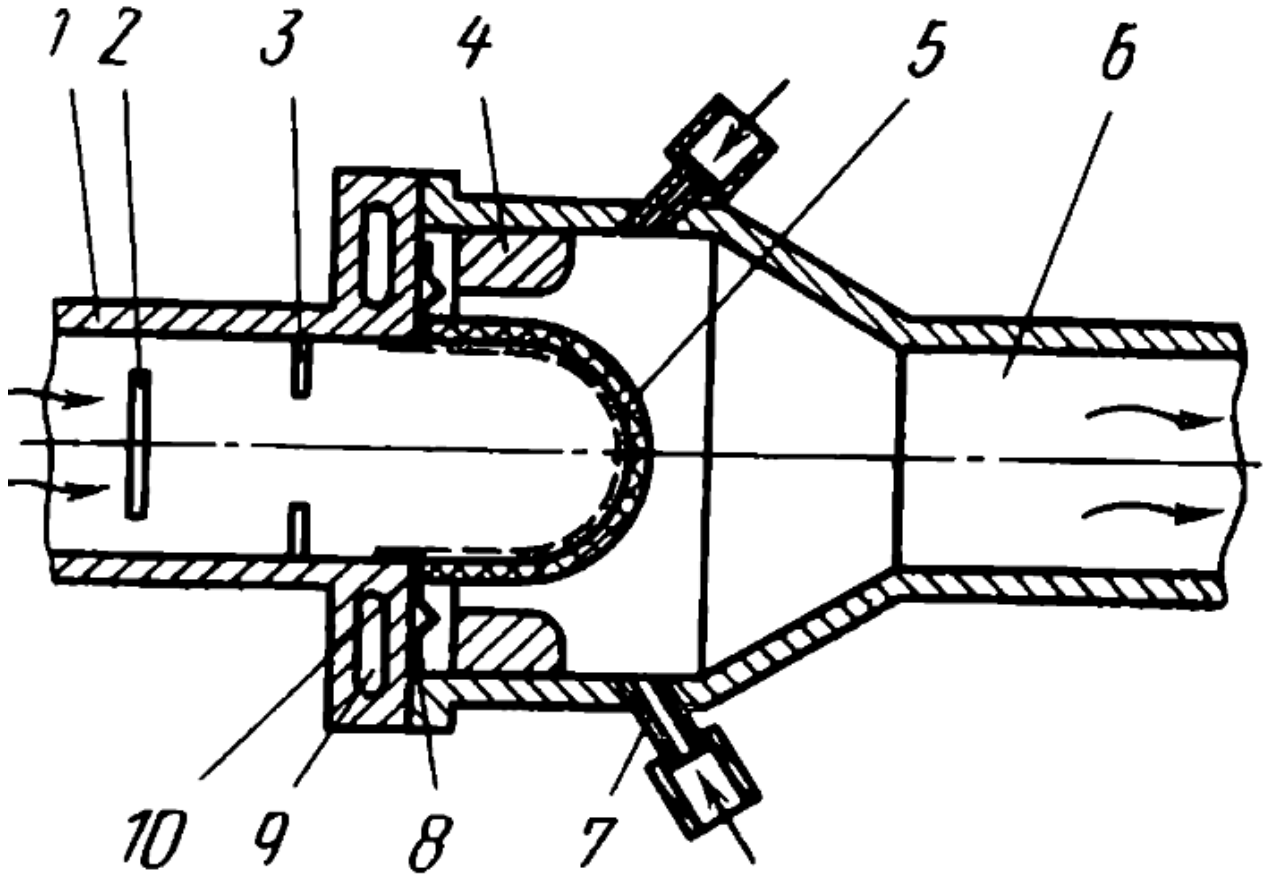
1. Vacuum Tight parameters at 10^{11}Pa and max. working temperature1600^{0}C,
2. low gas permeability 800 - 1000^{0}C – for 0_2, N_2, Ar, etc.,
3. Minimum dielectric loss, 10^9 – 10^{11} Gigahertz, 500- 700^{0}C,
4. High Electrical strength (vacuum, inert gas,),
5. High mechanical strength,
6. High thermal conductivity,
7. Multiple thermal shock,
8. Joining ability to metals,
9. Radiation durability,
10. High Purity.



Alumina Ceramic RF Windows

Temperature range -60 to +650^{0}C
Max pressure - 10kgf/cm^2

Types of RF Windows



loss of power

$$P \approx P_{\text{пад}} \alpha d, \ \text{вт},$$

X-Ray Windows

Beryllium Windows

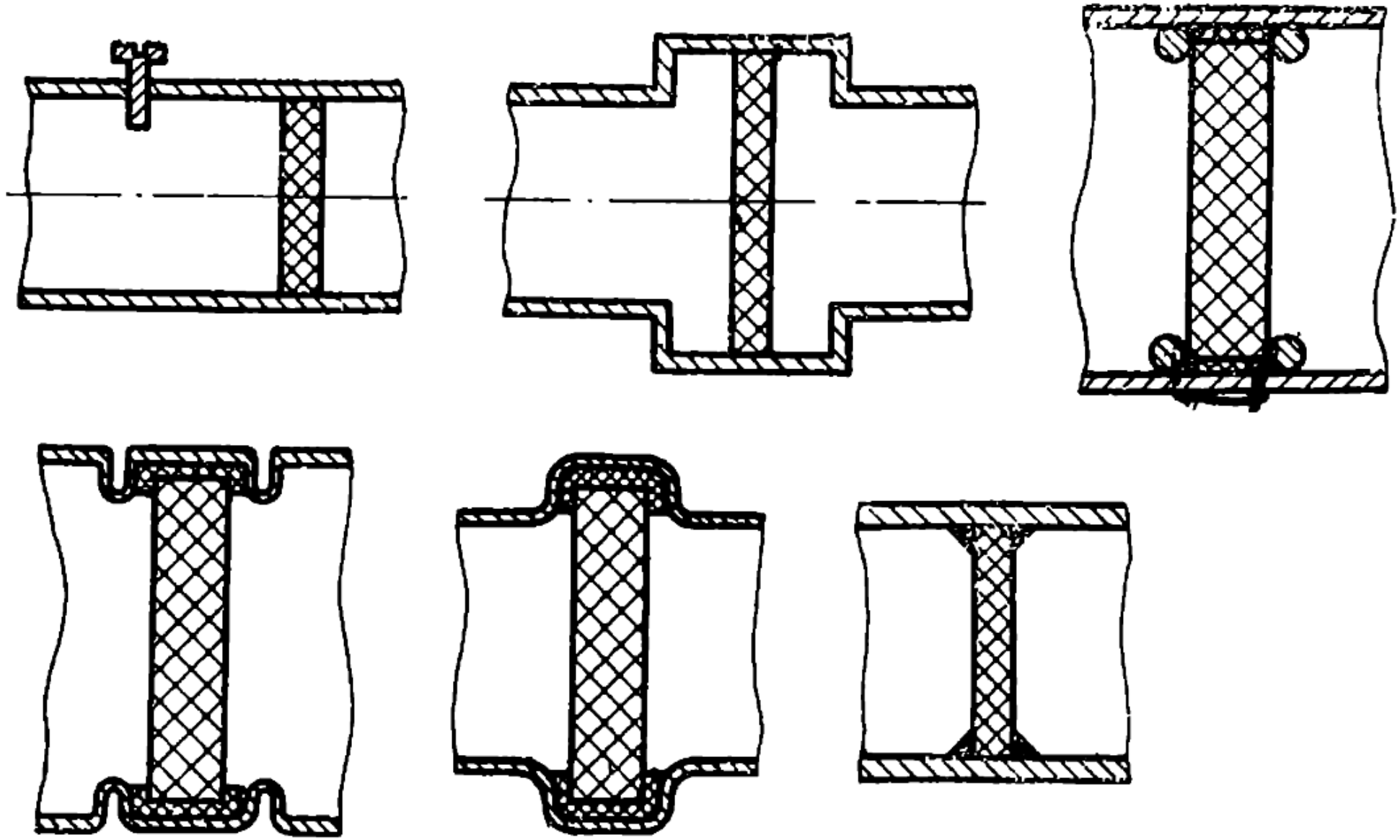
$P_{\text{пад}}$ — падающая мощность

d – толщина диэлектрика

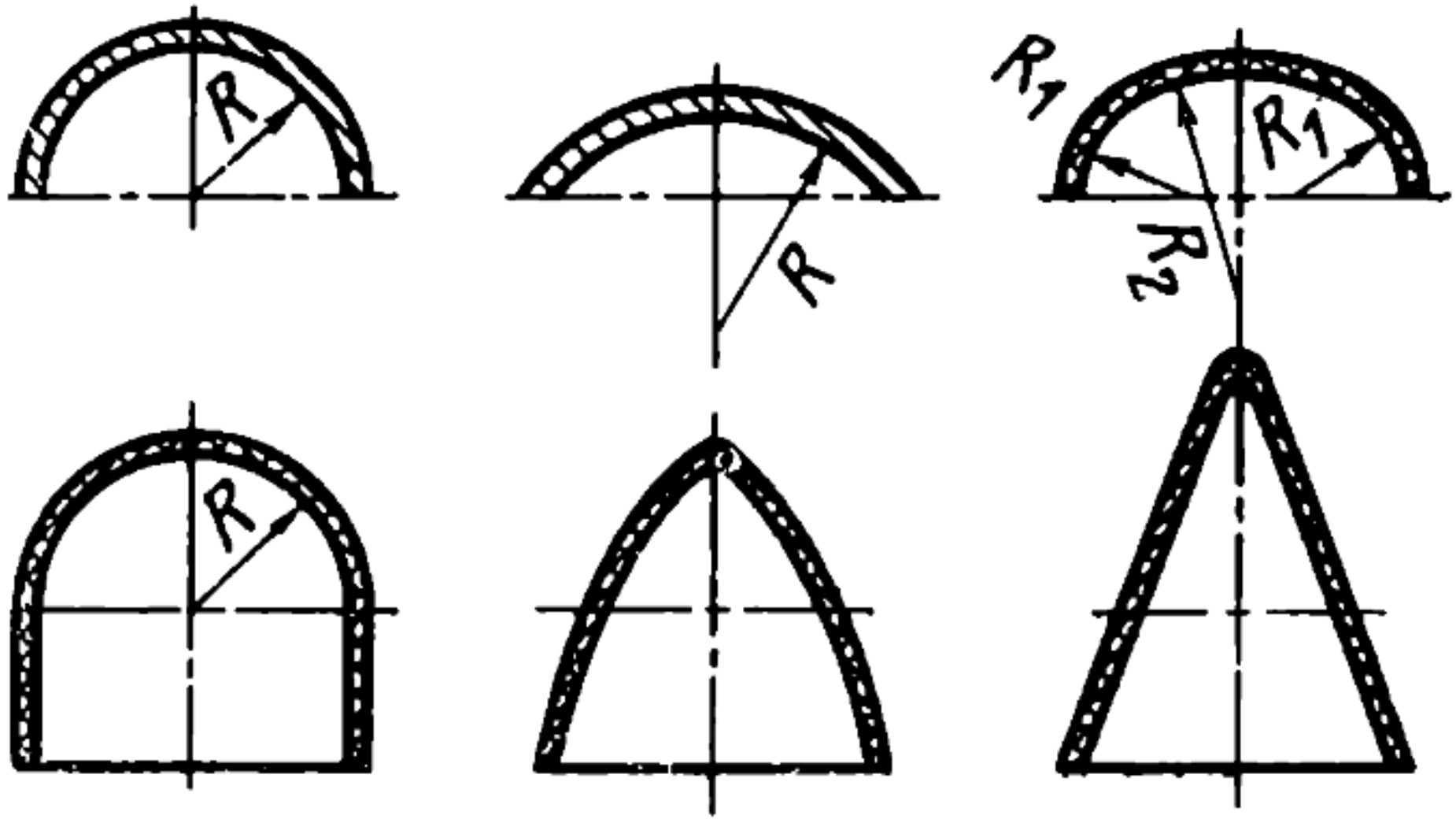
$\alpha = 1/2 \operatorname{tg} \delta \sqrt{\varepsilon} K$ (здесь $K = 2\pi/\lambda_0$)

λ_0 — длина волны в свободном пространстве

Diffusion Brazing and Welding for RF Windows



Types of RF Windows

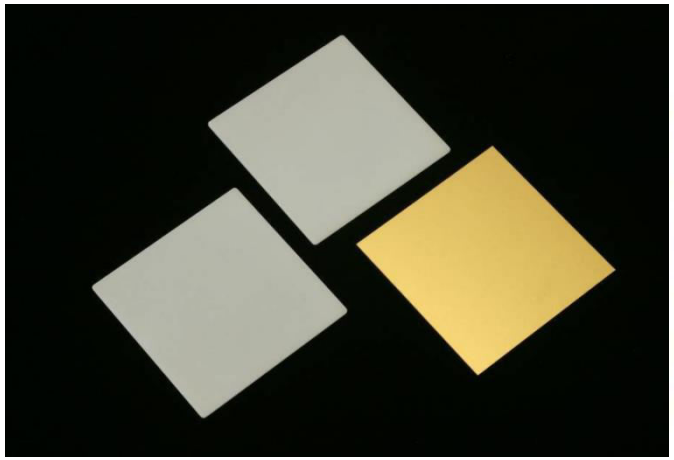


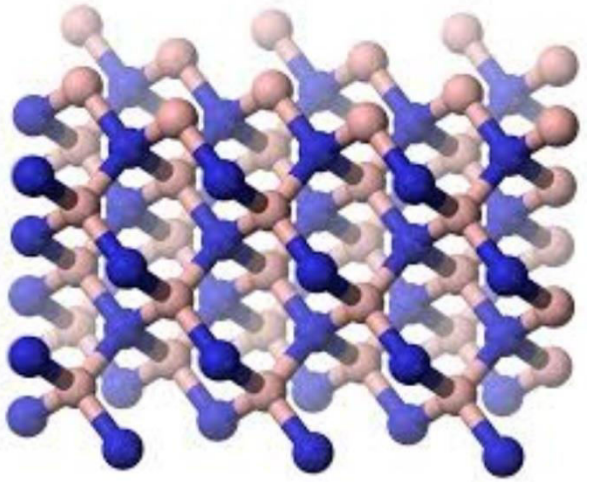
Parameters of RF Ceramic.

Ceramic Type	L·mm · mmHg cm/cm^2 $\times 10^3$	Gas type %			
		H_2	N_2+CO	CO_2	H_2O
22XC	1.2	55	40	3	2
22X	0.6	55	40	3	2
A-995	1.9	47	41	10	2
M-7	3.8	52	43	3	2
102	2.0	57	31	10	2
Ф-17	3.0	48	44	6	2
C-14	3.6	50	42	5	3

- High Mechanical strength,
- Outgassing Low Level,
- Reliable during long time,
- Thermal shock resistance,
- Electro – Magnetic Parameters,
- Low Material Penetration,
- Corrosion resistance,
- Dimensional stability.

Nitrid Bore



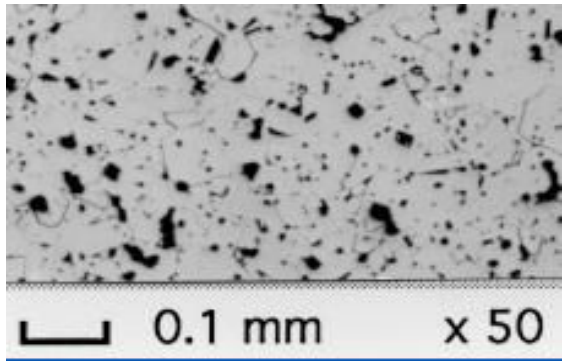


- Necessary RF parameters,
- Mechanical and thermal parameters

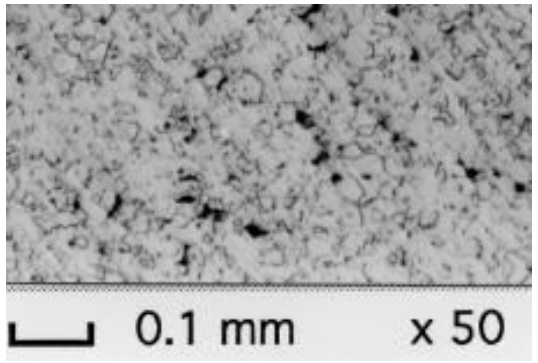
RF Windows

Various Ceramics for RF Window

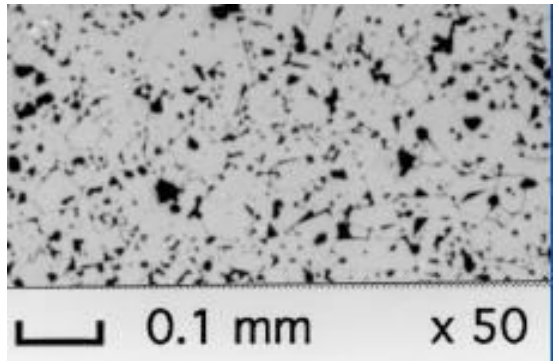
WESGO A L300



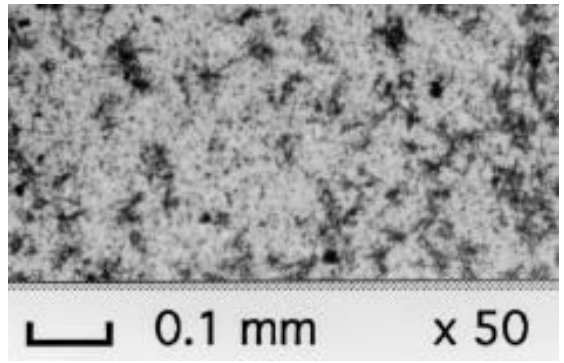
COORS AD995



KYOCERA 476SS



NTK UHA99



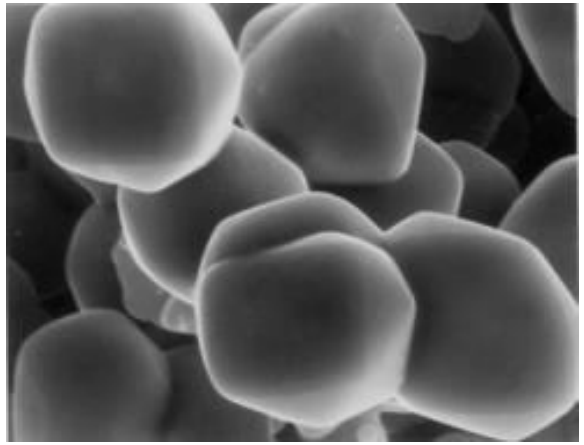
Metallizing for High Purity Ceramic

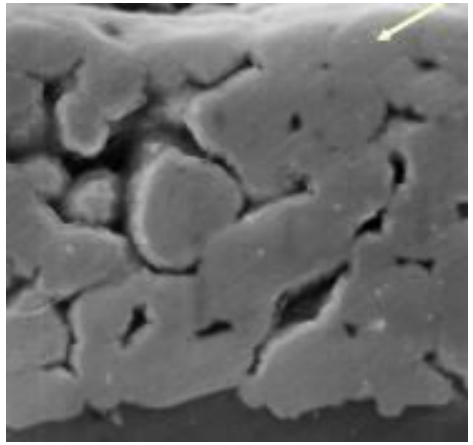
Metalize:

- Mo (85%) + Mn (10%) + Glass (3-7%) for 99.5% ceramic
- Mo (85%) + Mn (10%) + Ti + Glass (3-7%) for 99.9% ceramic
- Thickness: 15~20 μm
- Sintering Temperature: 1350 °C

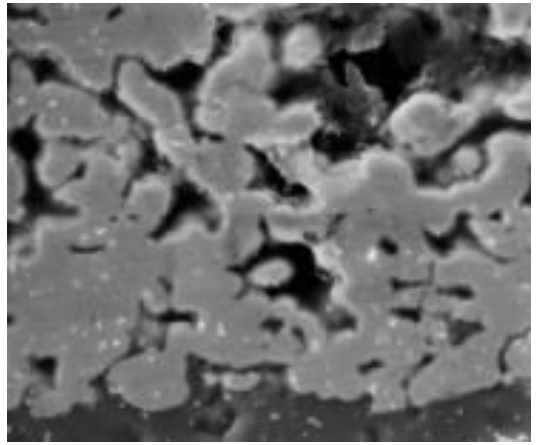
For Brazing

Use activate type brazing material: Au + Cu + Ti (1~2%)
Temperature: ~900 °C in vacuum

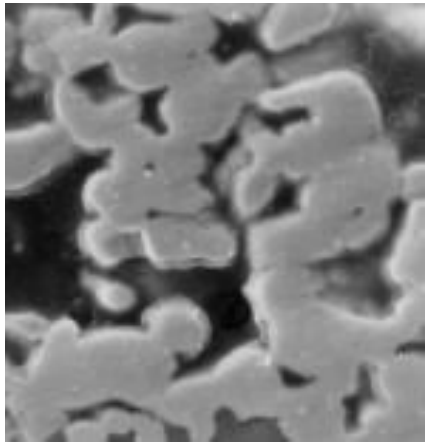




Glass 3%



Glass 5%



Glass 7%

	EIRO-XFEL & ILC (35 MV/m)	ICL (45 MV/m)
1) RF feed system:	no change	need study (use jB generator)
2) Coupler rf structure:	no change	need study (no tapering section, try to omit warm side window)
3) Alumina powder :	fine alumina (0.1-0.5 μm diameter)	←
4) Alumina purity:	>99.5% (~0.06% of MgO)	←
5) Void population:	~0.8% (no need HIPs)	←
6) Metalize ink:	Mo+Mn+Ti, size: Ø4.8-6 μm, Glass: 5%	←
7) Brazing material:	Au+Cu+Ti, in vacuum	←
8) Structure at brazing:	need study (less micro-clacks, structure simple)	←
9) Surface coating:	need study (diamond like etc)	←

UHV Vacuum Test Stend

Residual gas analysis in high vacuum
Outgassing
Etc.

Software

Pro|ENGINEER

PTC creo™

Molflow+

A Monte-Carlo Simulator package developed at CERN

NATIONAL INSTRUMENTS LabVIEW

SOLID EDGE

COMSOL

SolidWorks

Mathcad®

NX SIEMENS

AutoCAD

SynRad+


Thank You!