NIST

Fabrication of a Guarded-Hot-Plate Apparatus for Use over an Extended Temperature Range and in a Controlled Gas Atmosphere

R. R. Zarr, D. R. Flynn, J. W. Hettenhouser,
N. J. Brandenburg, W.M. Healy
National Institute of Standards and Technology

Presented to:
28th International Thermal Conductivity Conference
and 16th International Thermal Expansion Symposium
St. Andrews-by-the-Sea, New Brunswick, Canada
June 28, 2005

Outline of Talk

- Motivation
- Apparatus Design
- Fabrication of Hot Plate
- Summary
- Future Work

Motivation

- High-temperature thermal insulation reference materials are *unavailable.*
- High-temperature guarded-hot-plate apparatus comparisons:
  - 1988 – Hust and Smith: fibrous alumina silica, 60°C to 450°C, ±15%
  - 1988 – Hust and Smith: calcium silicate, 60°C to 450°C, ±16%
  - 2001 – Salmon and Tye: rockwool fibre, 100°C to 500°C, ±16%
  - 2002 – Albers: fibrous alumina silica, 0°C to 1000°C, ±24%
  - (Room temperature guarded-hot-plate comparisons ±2 % or better)
- Limitations for current standard test methods for guarded hot plates (ASTM C177 and ISO 8302)
  - Example – no comparison data (or statistics) for:
    - Cryogenic temperatures
    - Vacuum conditions

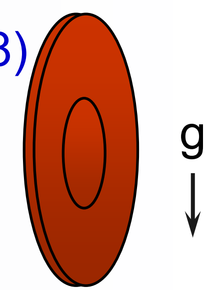
Apparatus Design

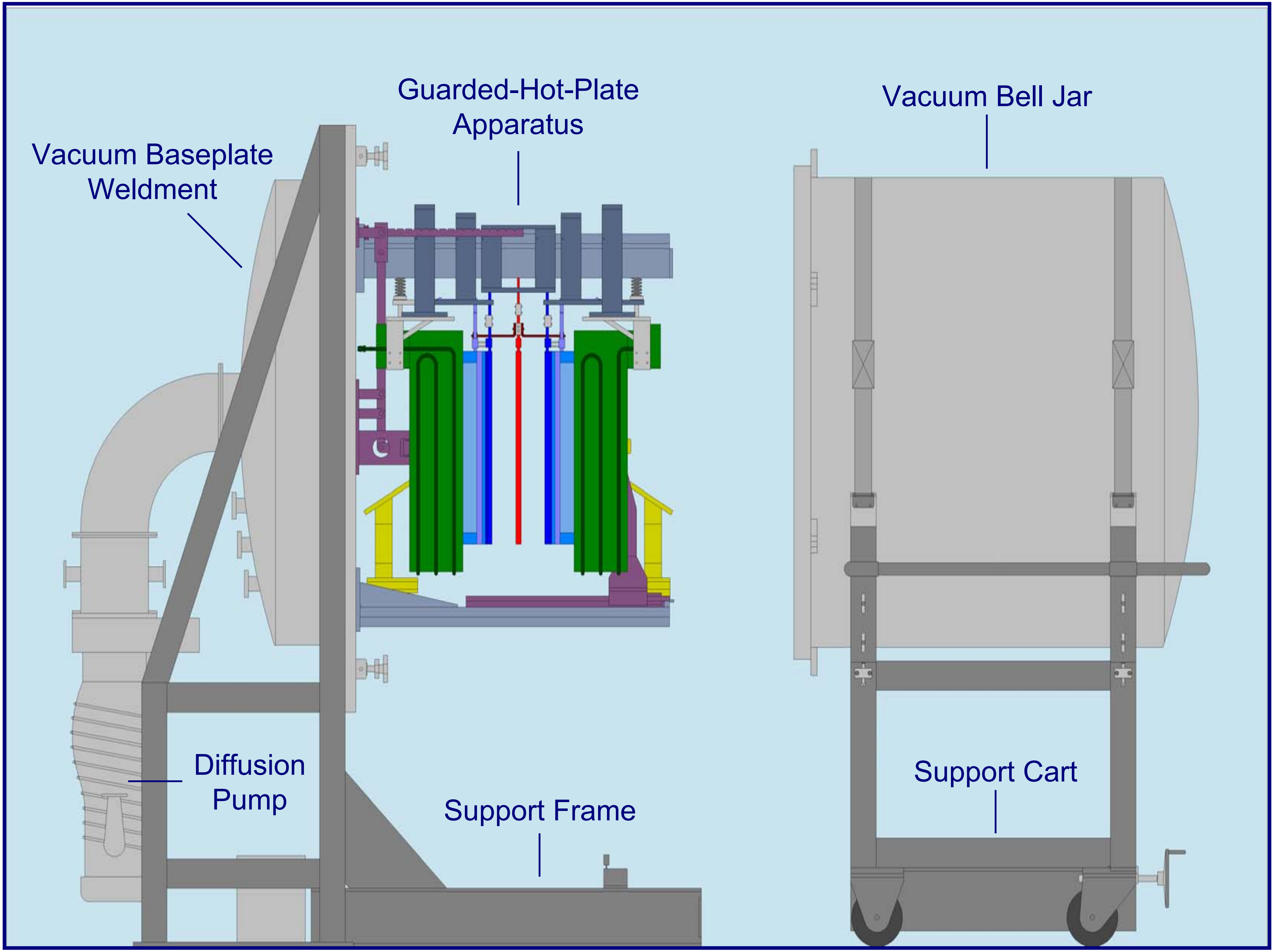
Requirements

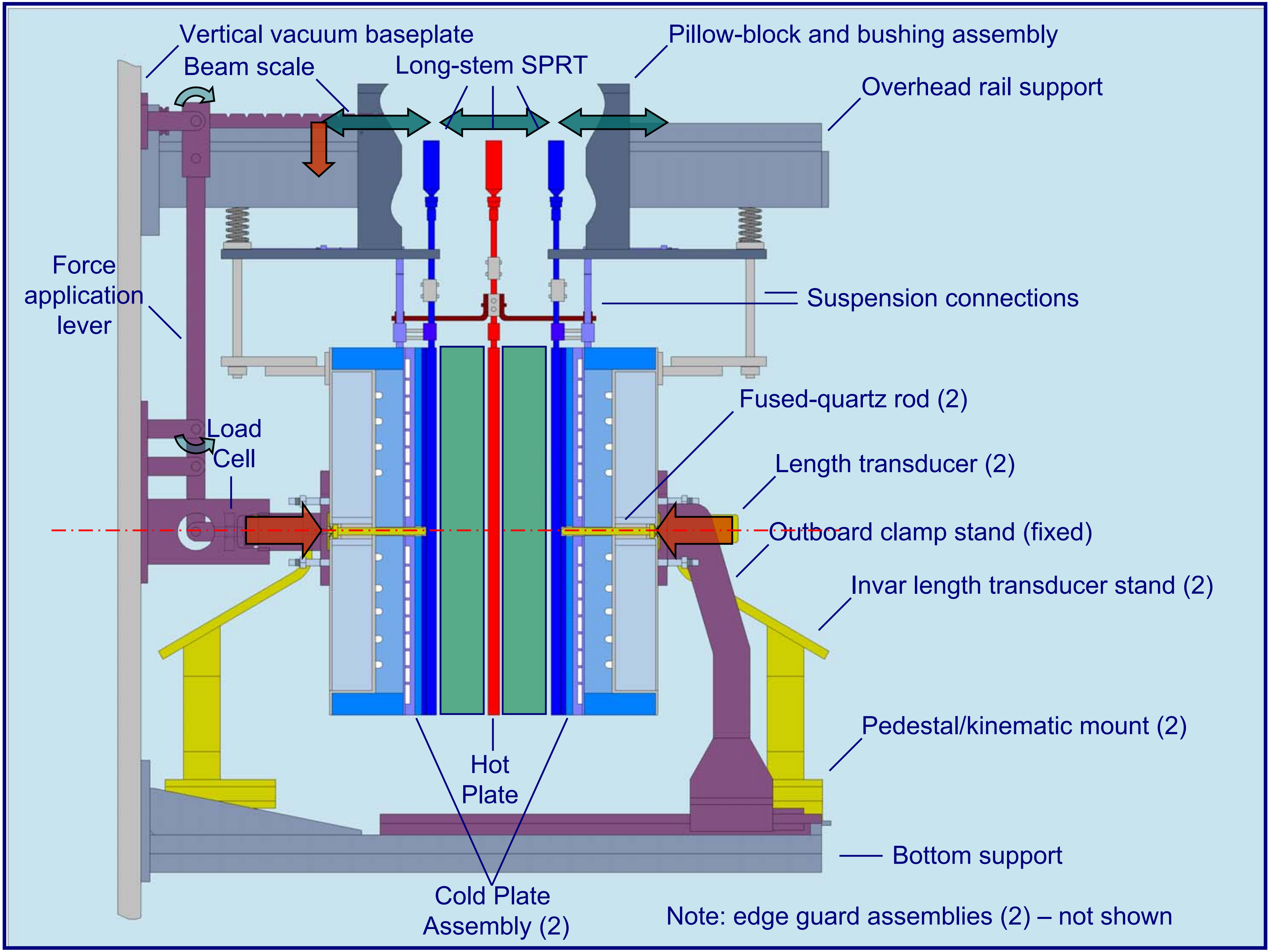
- Range of Operation
  - 90 K to 900 K
  - 10^{-5} kPa to 105 kPa
  - 13 mm to 100 mm
  - 2-sided mode (specimen pair)
  - Horizontal heat flow
- Desired Uncertainty
  - ±1 % at 300 K
  - ±3 % at 90 K or 900 K
- Heaters
  - Line-heat-source design
  - Rugged
- Integrated cold plates
- Integrated edge guards

Specifications

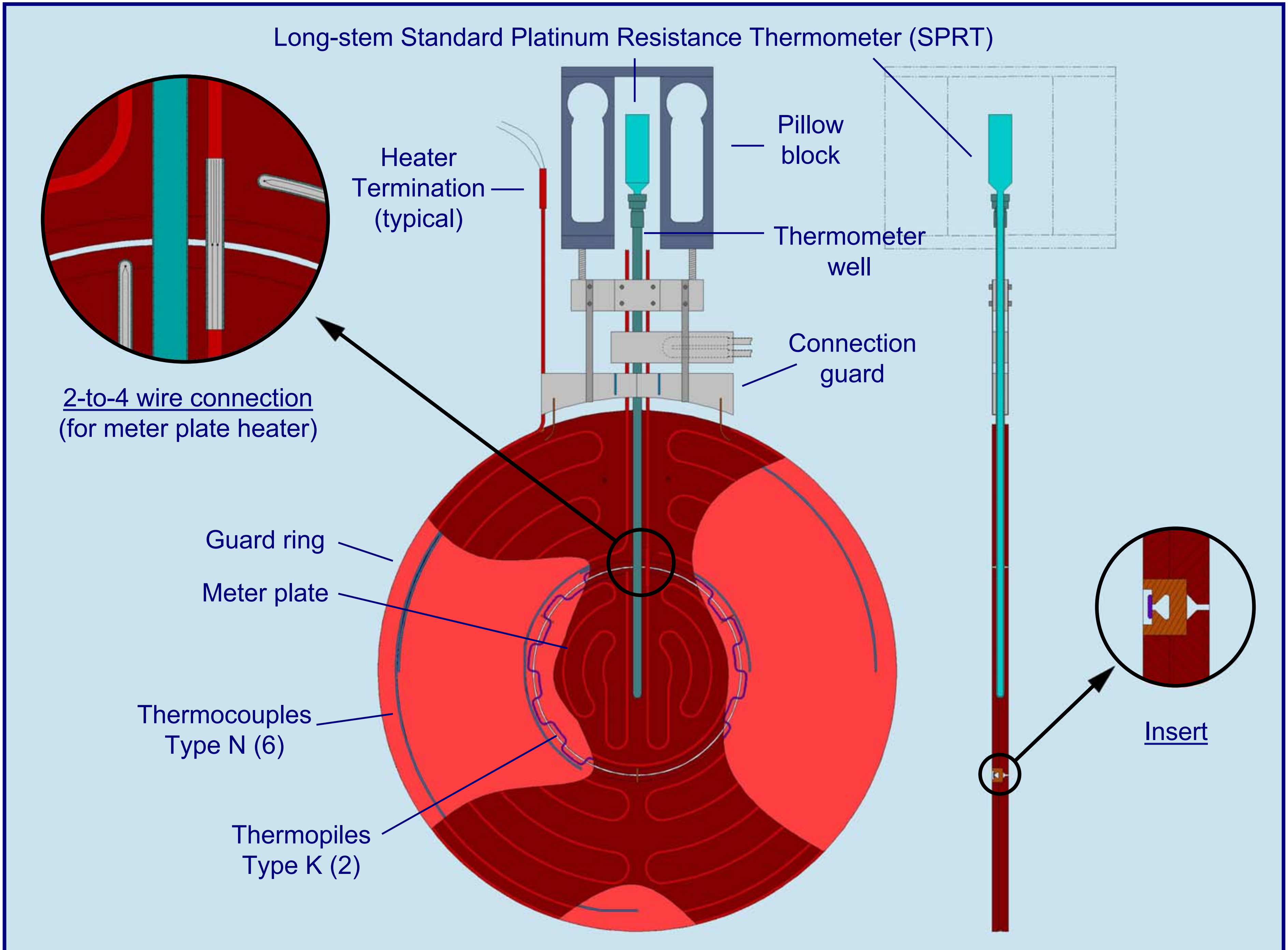
- Plates
  - Circular geometry
  - 500 mm diameter (ISO 8302)
  - Guard to meter ratio: 2.5
  - Material: nickel 201
  - Metal sheathed heaters, TC, TP
  - Long-stem SPRTs
  - Vacuum-brazed (one piece)
  - High-emittance coating (>0.8)
- Apparatus Arrangement
  - Vertical plates
  - Translate horizontally
  - Enclosed in a vacuum bell jar




Guarded-Hot-Plate
Apparatus
Vacuum Bell Jar
Vacuum Baseplate
Weldment
Diffusion
Pump
Support Frame
Support Cart



Vertical vacuum baseplate
Beam scale
Long-stem SPRT
Pillow-block and bushing assembly
Overhead rail support
Force application lever
Suspension connections
Fused-quartz rod (2)
Load Cell
Length transducer (2)
Outboard clamp stand (fixed)
Invar length transducer stand (2)
Pedestal/kinematic mount (2)
Hot Plate
Bottom support
Cold Plate Assembly (2)
Note: edge guard assemblies (2) – not shown



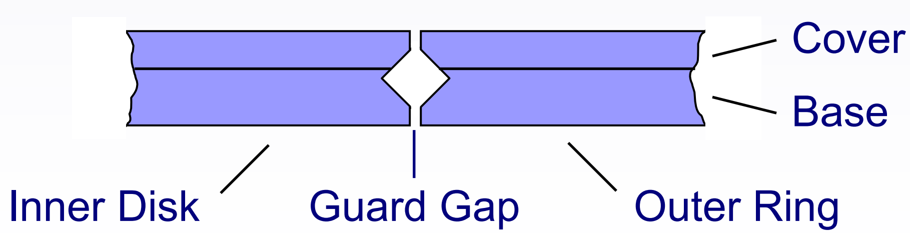
Long-stem Standard Platinum Resistance Thermometer (SPRT)
Pillow block
Heater Termination (typical)
Thermometer well
Connection guard
2-to-4 wire connection
(for meter plate heater)
Guard ring
Meter plate
Thermocouples Type N (6)
Thermopiles Type K (2)
Insert

Fabrication of Hot Plate

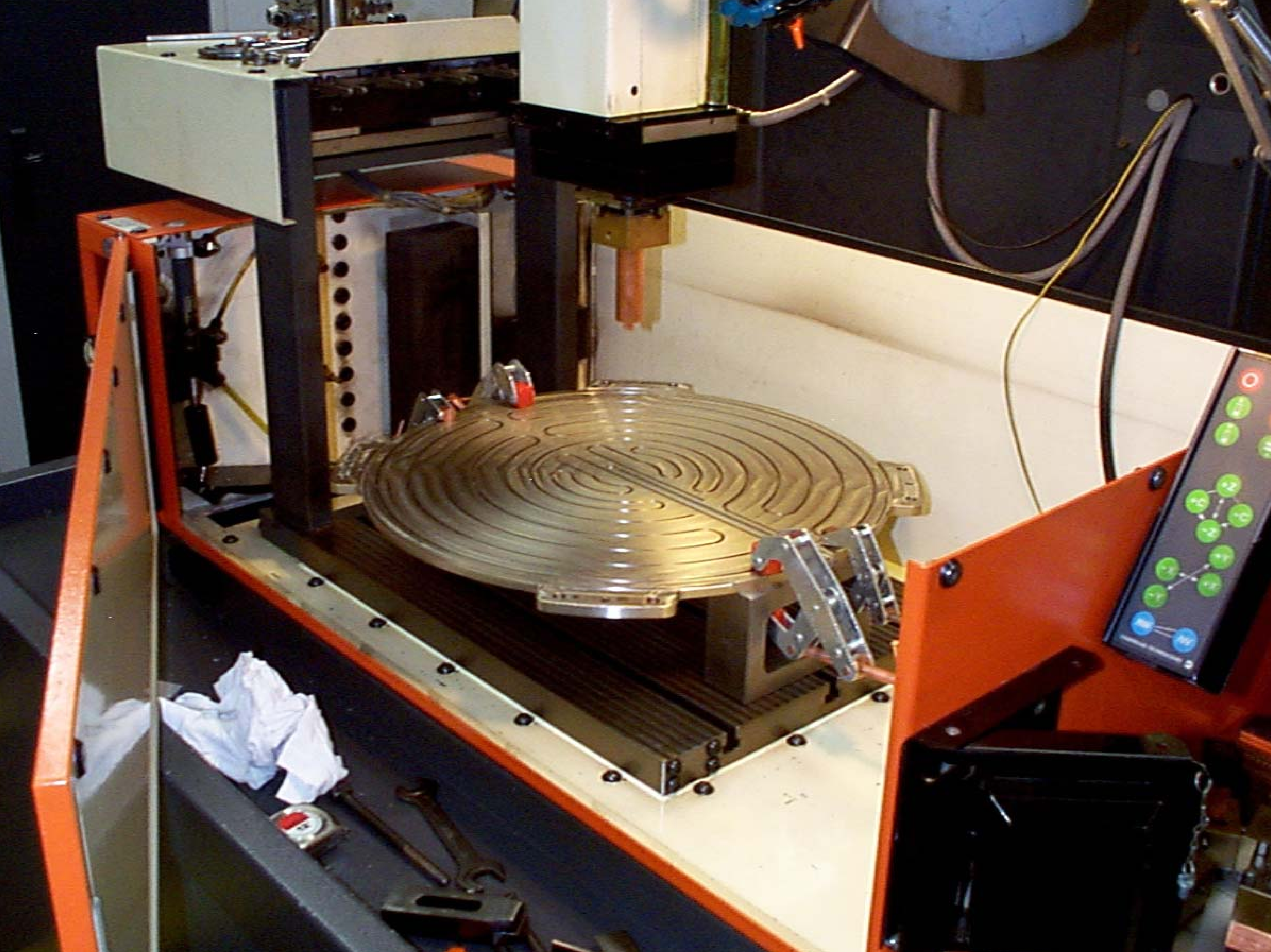
- Metal cutting ⇒ form components
- Vacuum brazing ⇒ assembly 925 °C
- Grinding ⇒ surface finish
- Ceramic coating ⇒ high emittance 510 °C
- Electrical connections 70 °C

Metal Cutting – NIST

- Main plate components: base and cover
  - Horizontal-spindle, high-speed milling machine: form and cut grooves (+0.05 mm tolerance)
  - Heater patterns based on FE analyses (Healy and Flynn)
- Guard gap: inner disk and outer ring
  - Electrical discharge machining: cut gap
  - Lathe V-grooves (diamond profile)




MACHINE
IN
USE

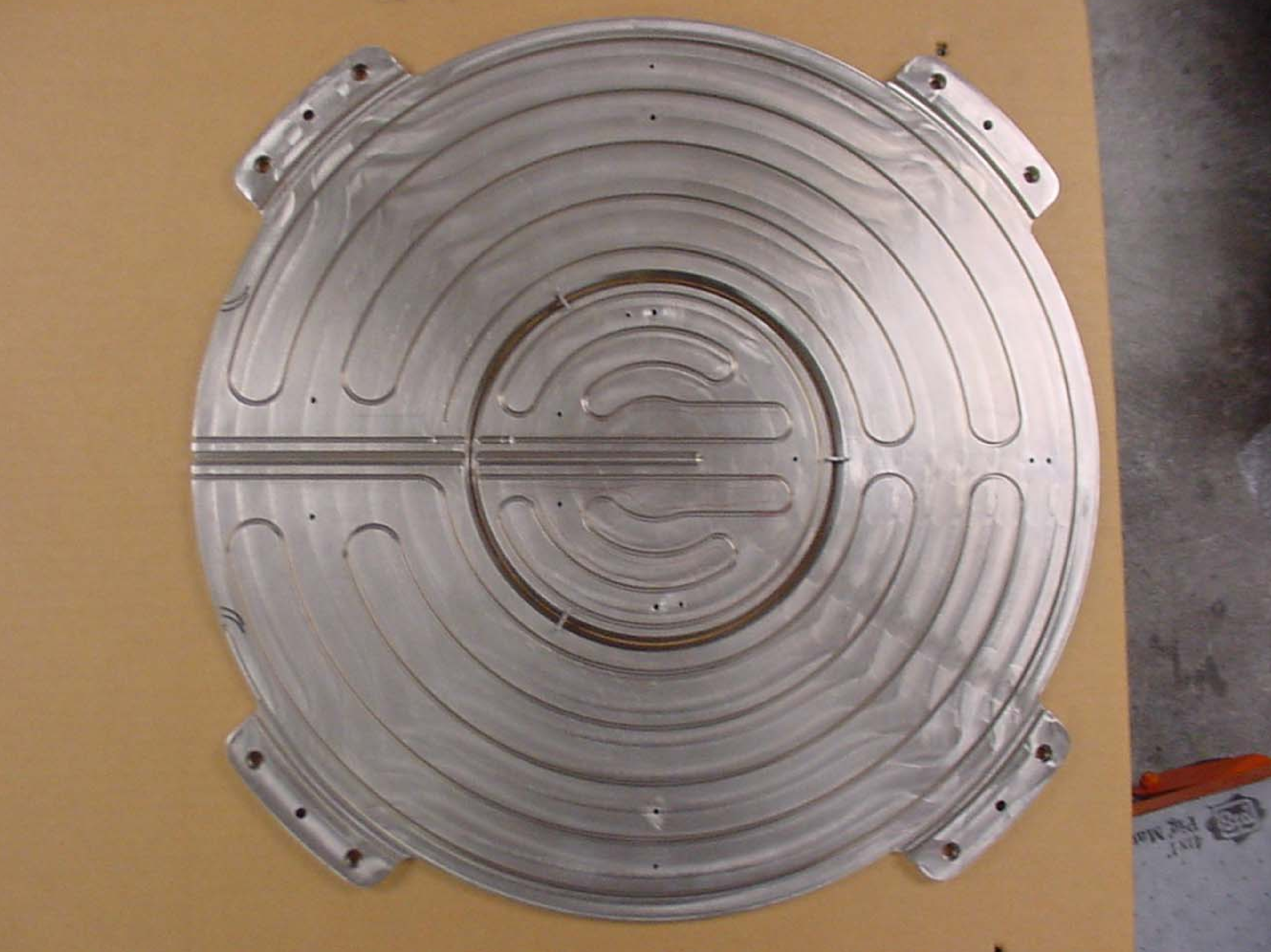


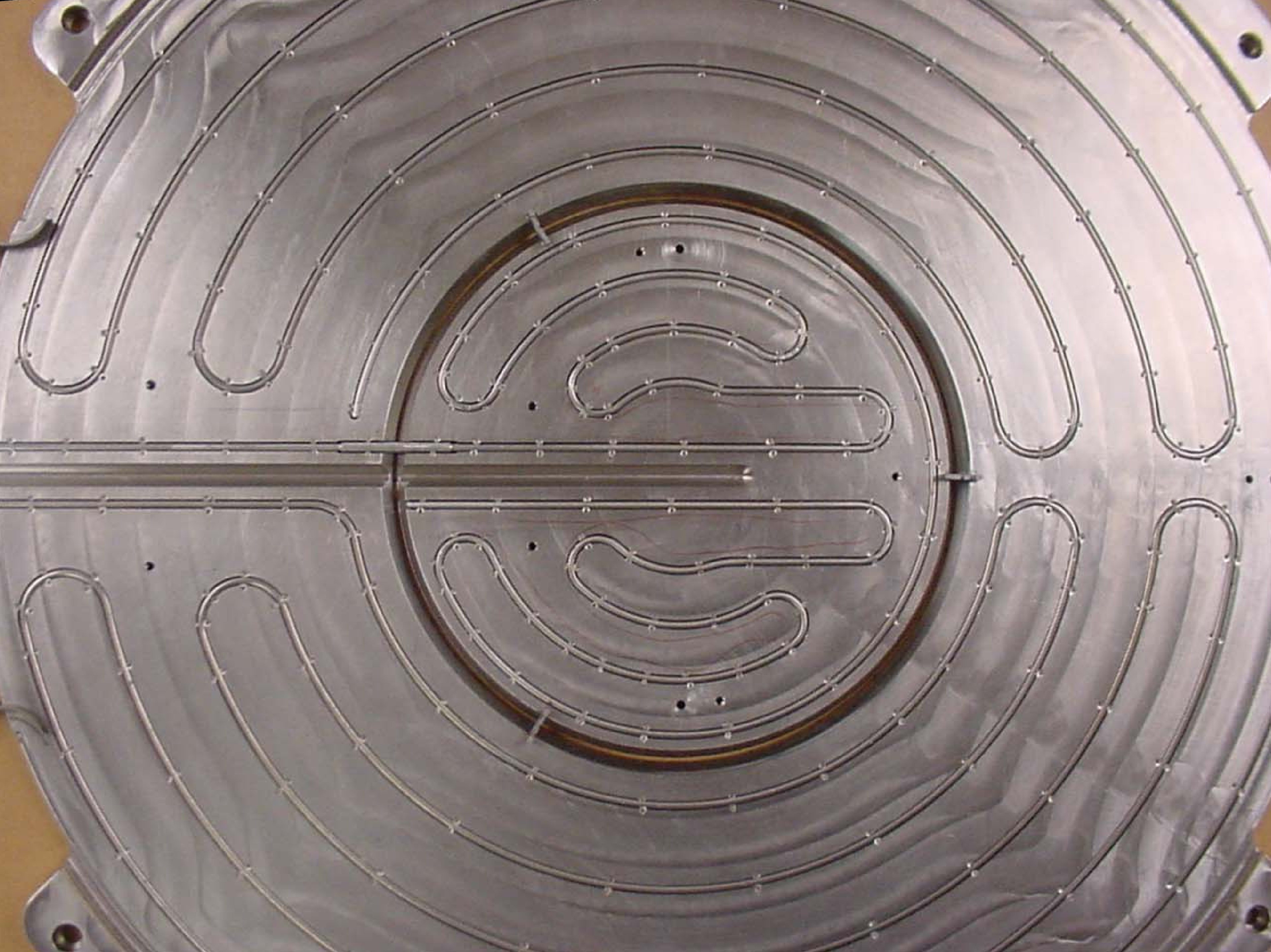
Fabrication of Hot Plate

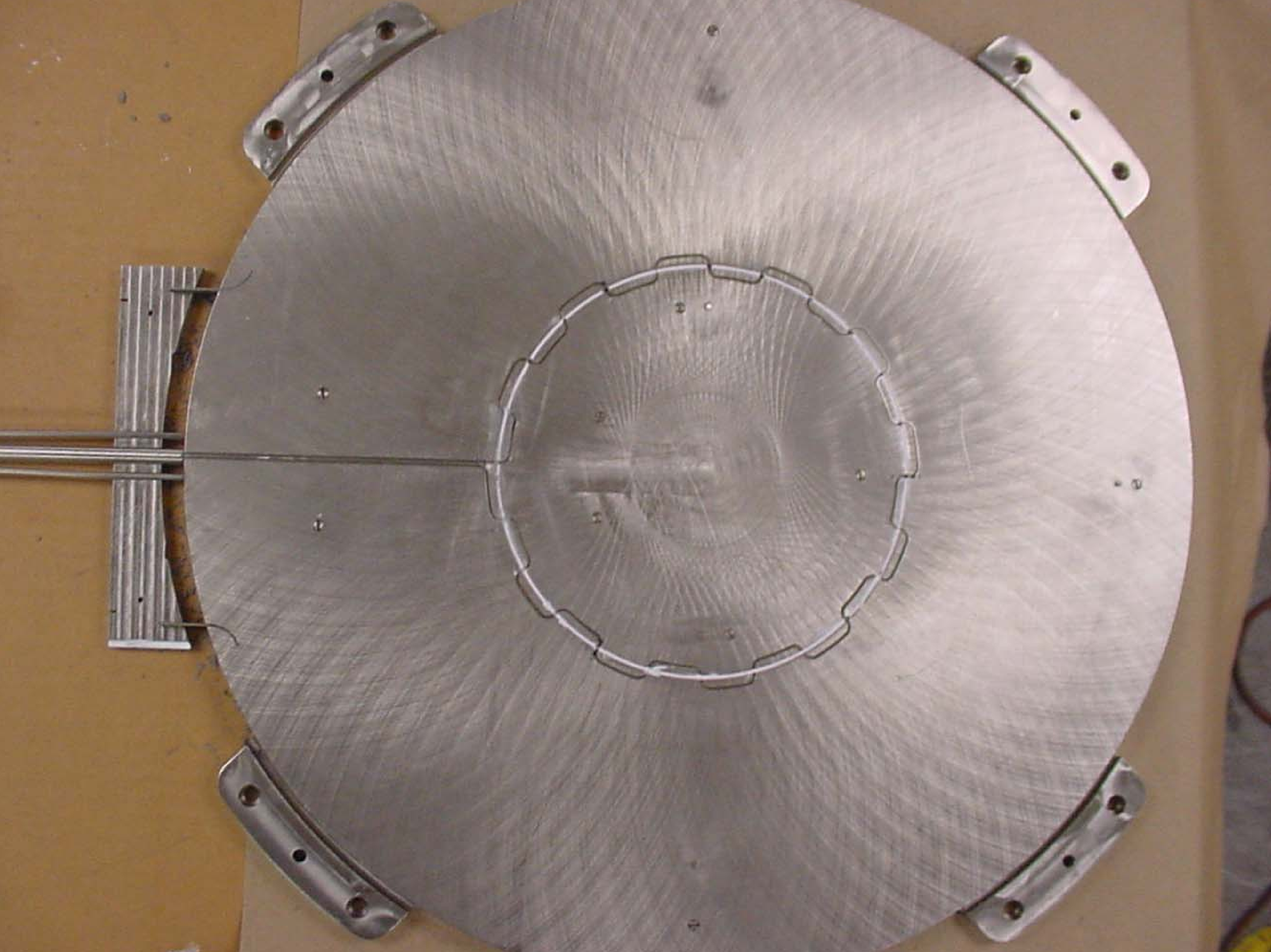
- Metal cutting ⇨ components
- Vacuum brazing ⇨ assembly
- Grinding ⇨ surface finish
- Ceramic coating ⇨ high emittance
- Electrical connections

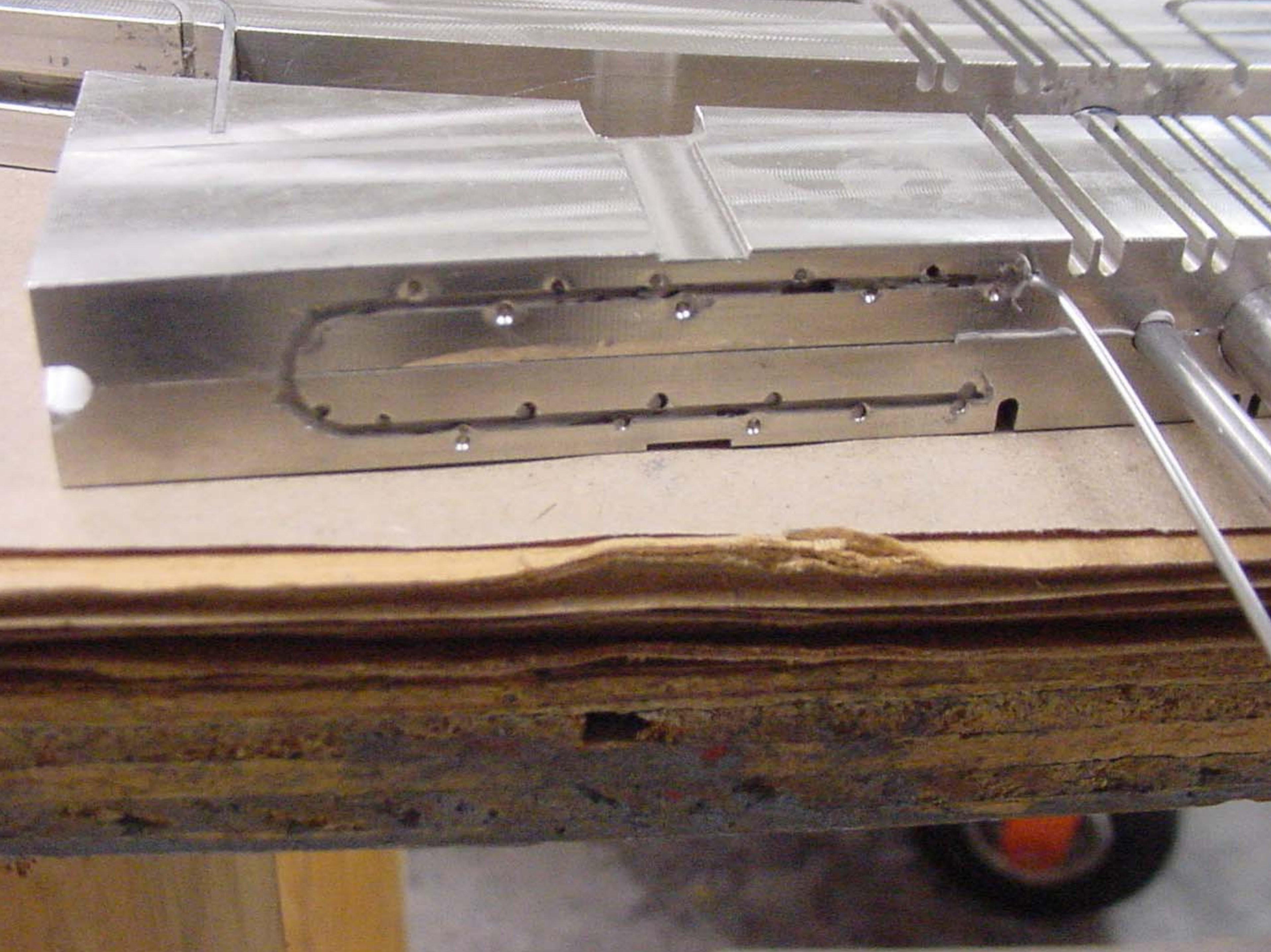
Vacuum Brazing – Vendor

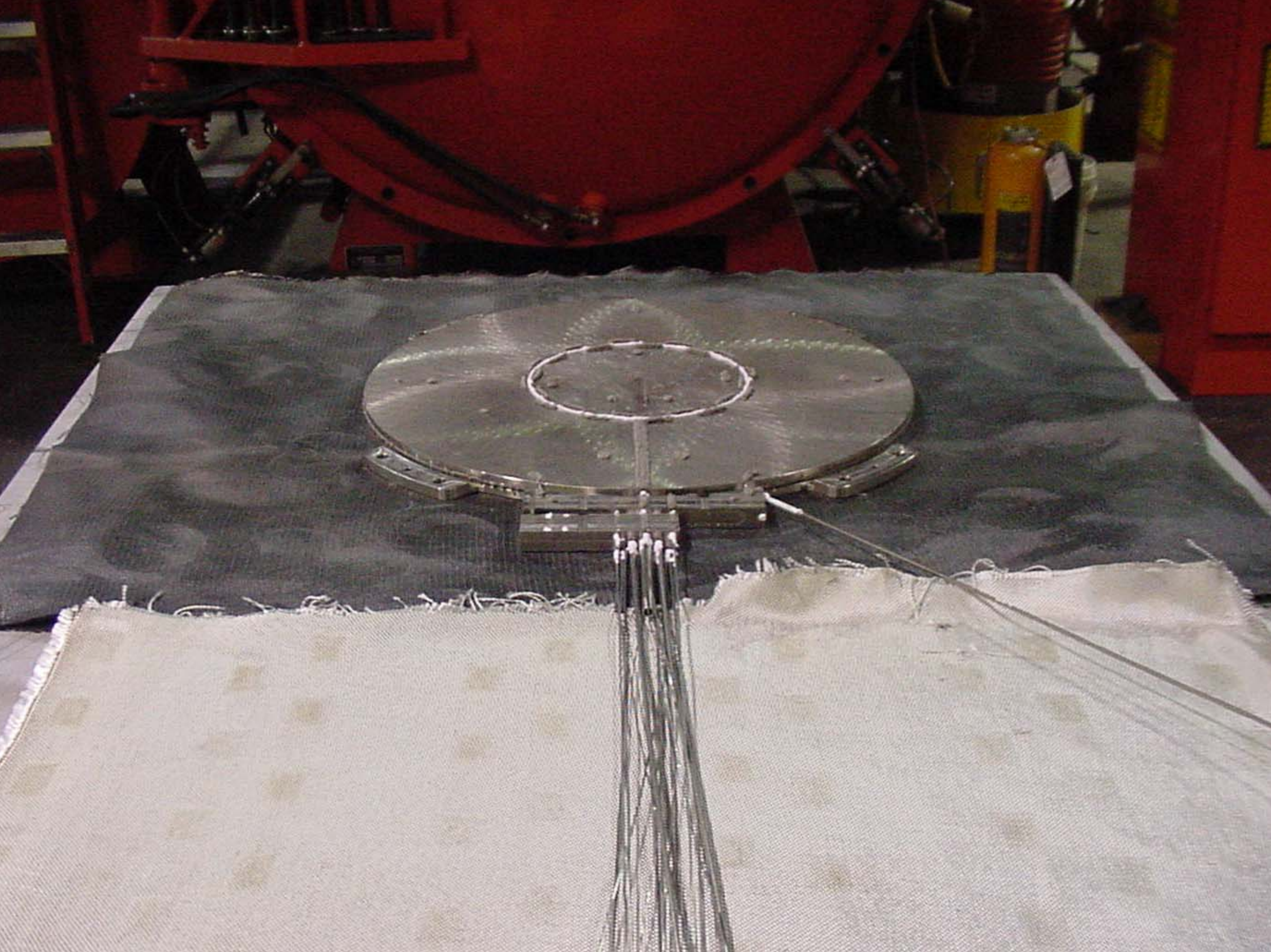
- Braze filler metal
  - Nickel-phosphorous (11 %) eutectic (nominal MT 875 °C)
  - Braze range: 925 °C to 1095 °C
- Assemble components
  - Grit blast surfaces, if necessary, to remove oxidation
  - Methodically “stake” pre-formed metal sheathed components (supplied by another vendor) into mating grooves
  - Apply measured water-based braze alloy (by syringe)
  - Apply water-based ceramic paste (as needed)
- Install in vacuum furnace
  - Assemble components on graphite sheet (weight)
  - Place in furnace and evacuate ≤ 10^{-5} torr
  - Heat and hold at 925 °C (~1.5 h) – braze alloy capillary action
  - Vacuum cool – establish metallurgical bonds
  - At 650 °C – quench with room-temperature nitrogen gas

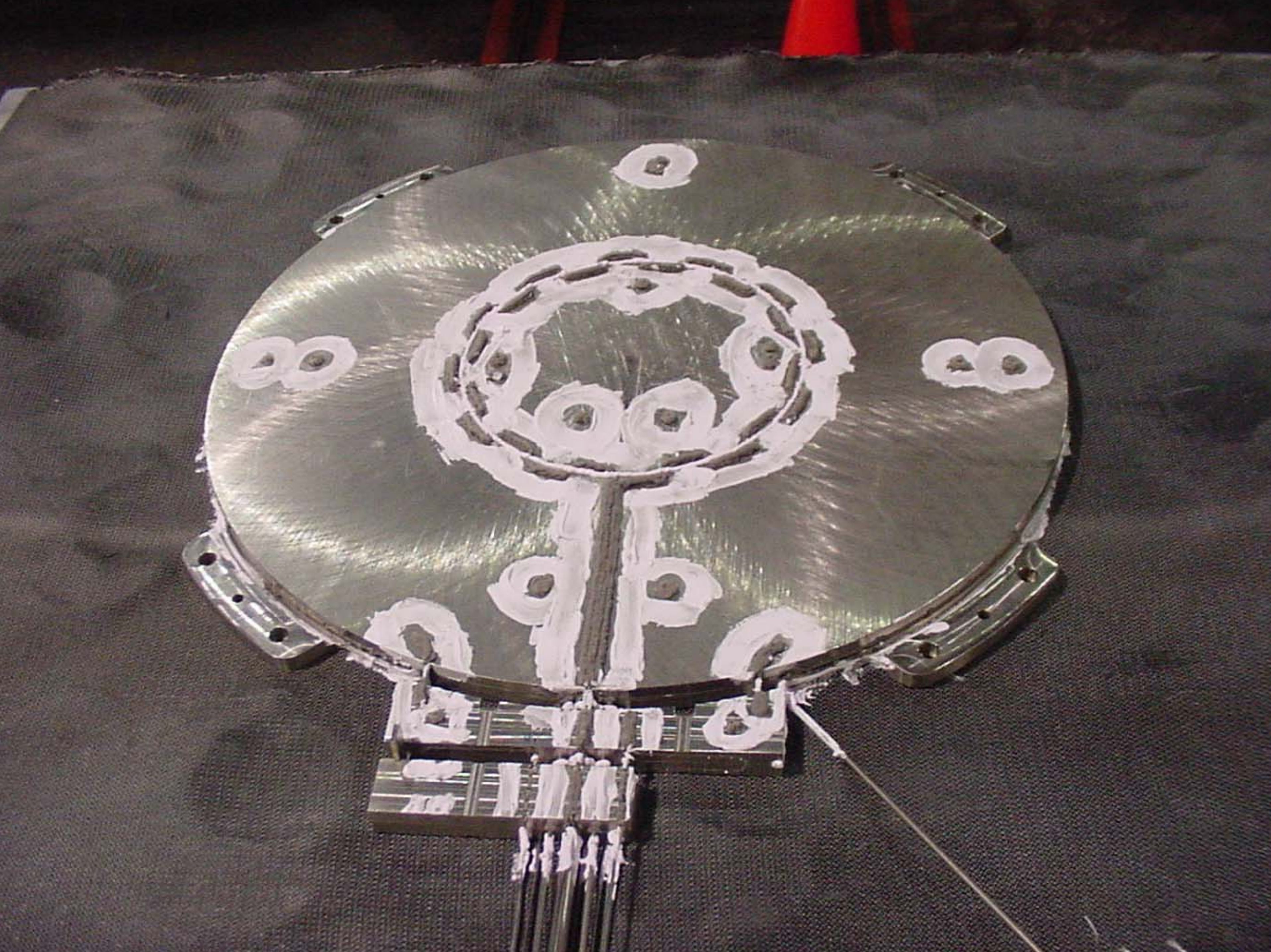


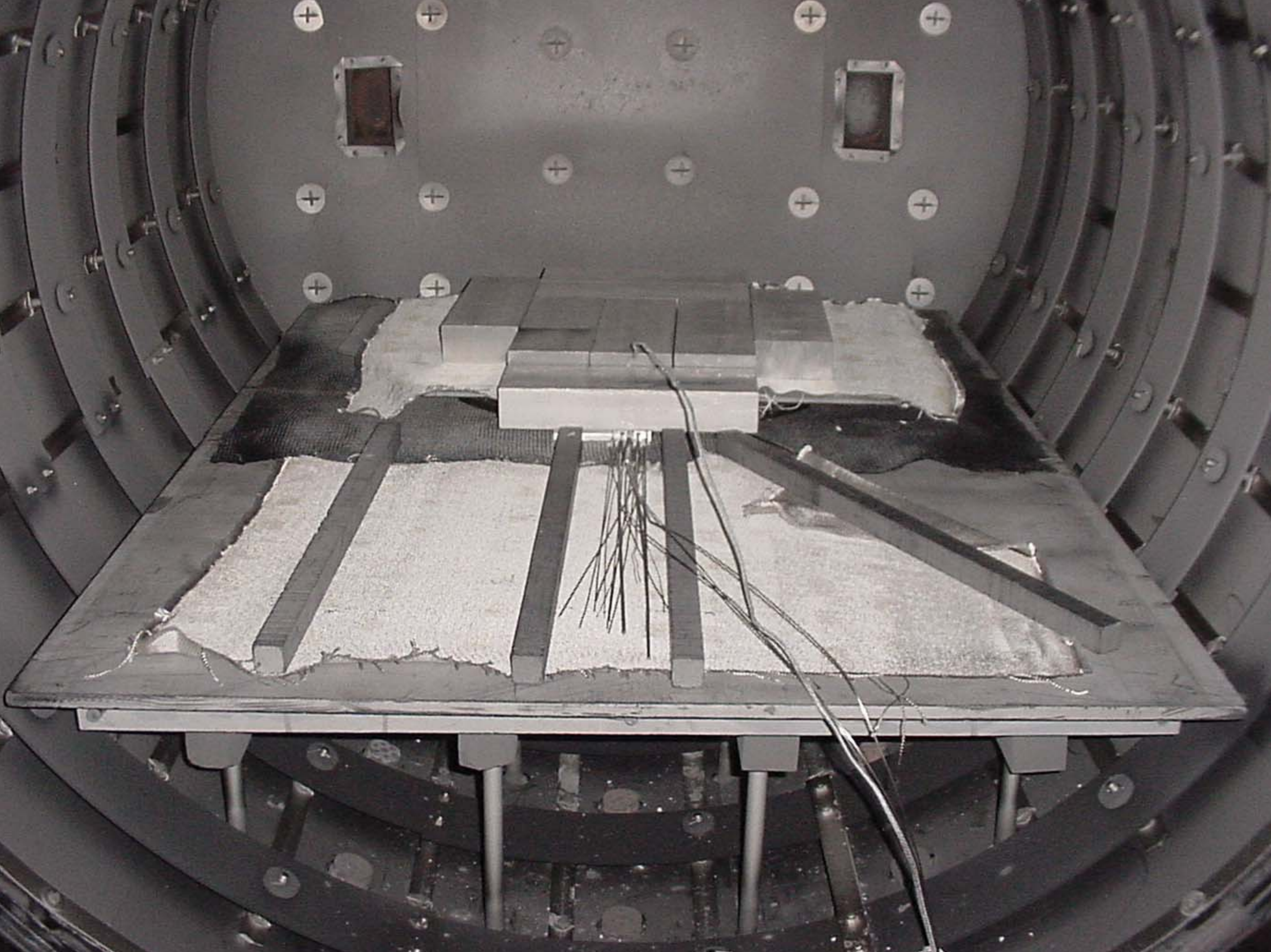










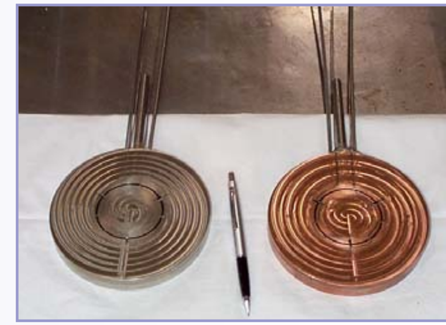


Fabrication of Hot Plate

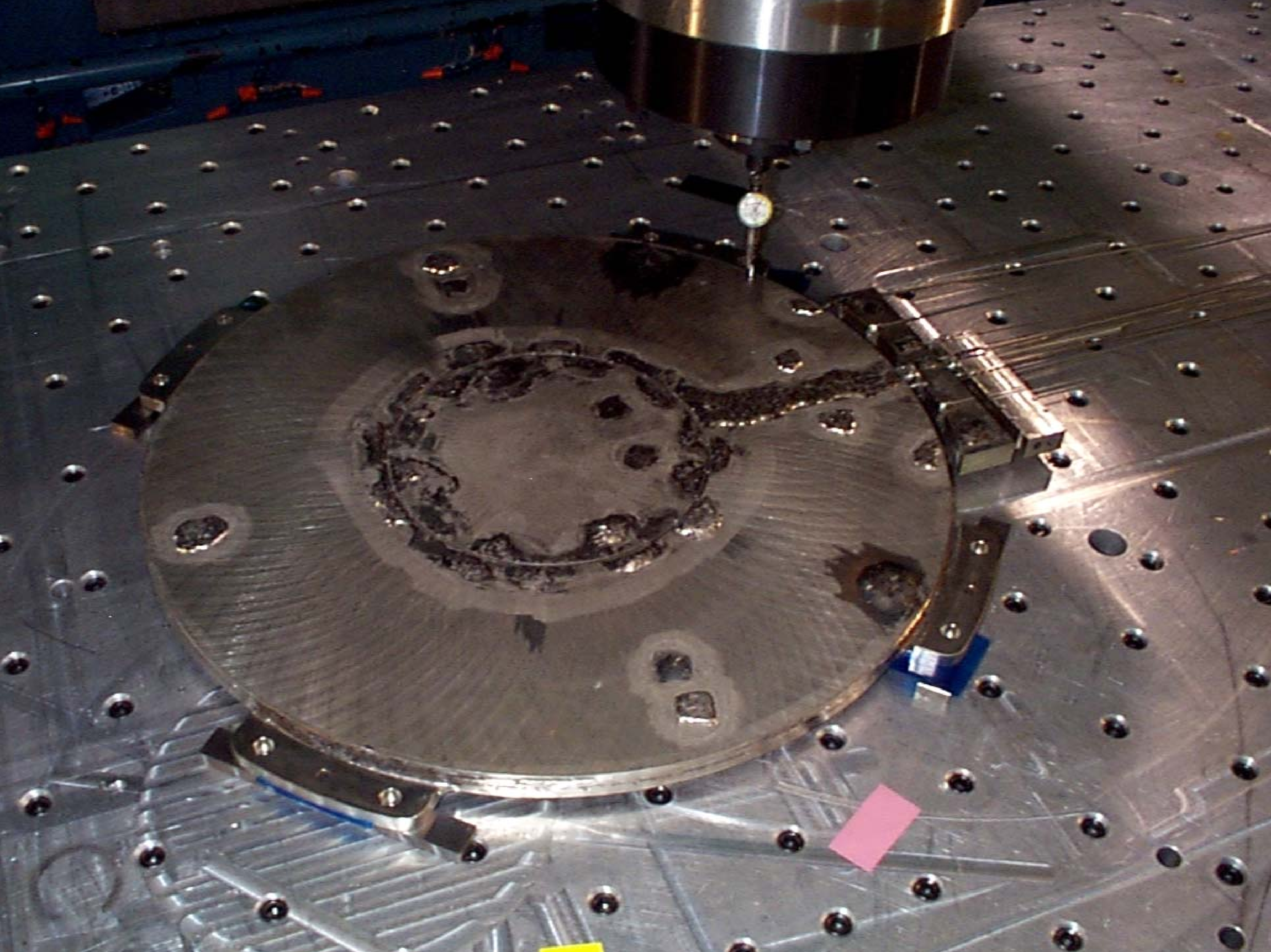
- Metal cutting ⇨ form components
- Vacuum brazing ⇨ assembly
- Grinding ⇨ surface finish
- Ceramic coating ⇨ high emittance
- Electrical connections

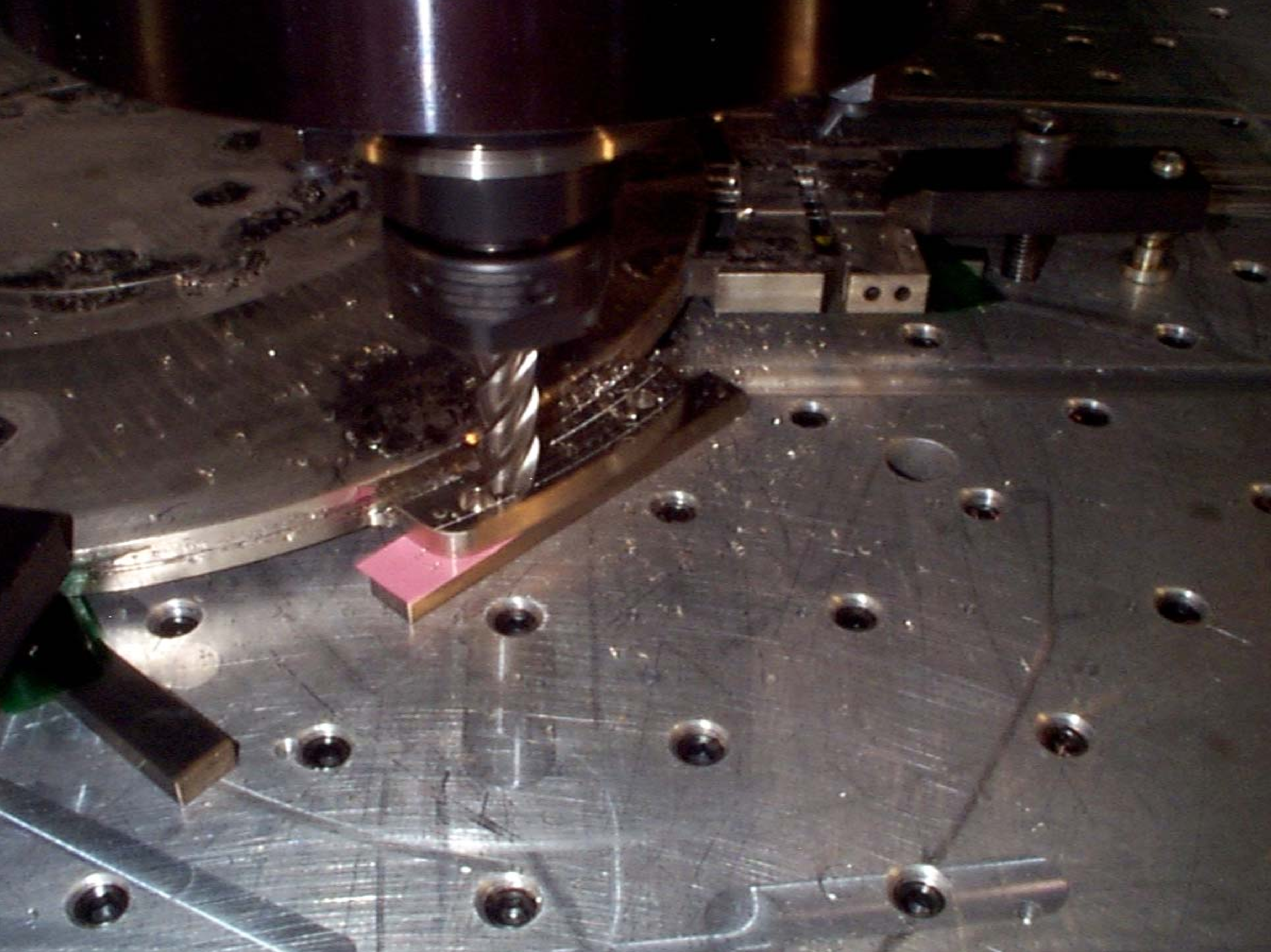
Grinding – NIST

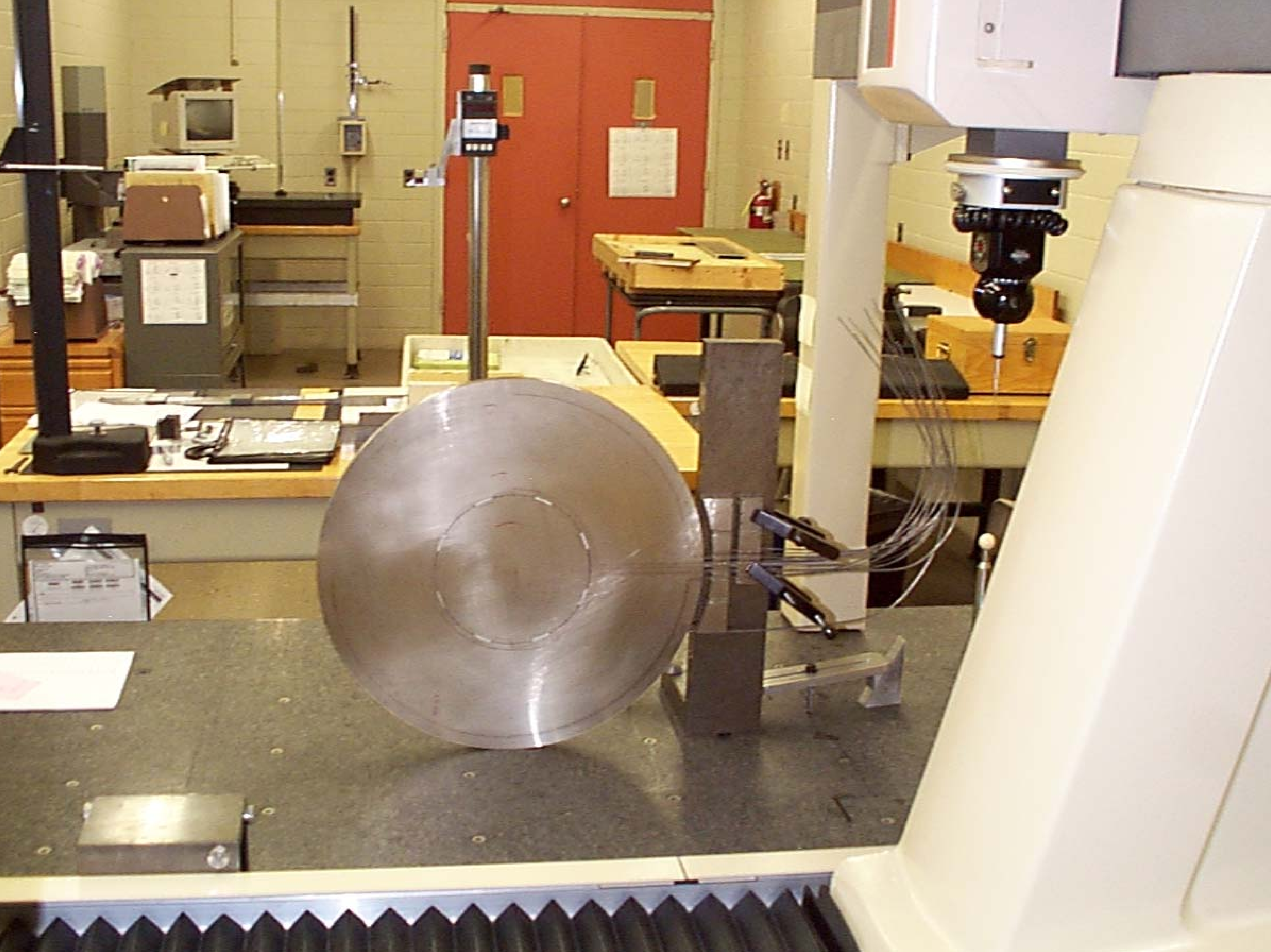
- Plate thickness oversized 0.5 mm (0.25 mm per face) – based on brazing results of small prototype plates
- Vertical machining center
  - Mount on pads (elevate to compensate for braze alloy puddles)
  - Indicate and establish parallel surface on pads
  - Re-mount and remove surface layers from plate
  - Excessive chatter noted in machining
- Blanchard rotary surface grinder
  - Segmented abrasives – aluminum oxide
  - Total removed 0.69 mm (0.027 inch)
  - Flat and parallel to 0.01 mm (to be verified)



Proto-type plates





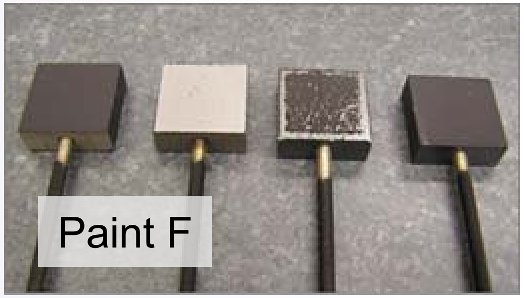


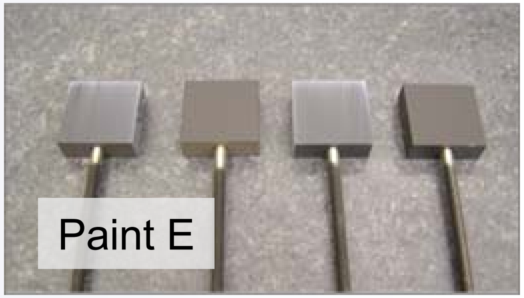
Fabrication of Hot Plate

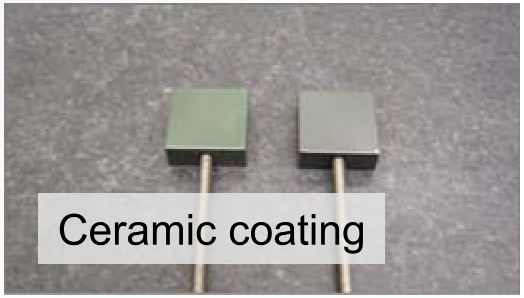
- Metal cutting ⇨ form components
- Vacuum brazing ⇨ assembly
- Grinding ⇨ surface finish
- Ceramic coating ⇨ high emittance
- Electrical connections

Plate Coating - High Emittance Paints

- Part I – 14 nickel 201 coupons (650 °C)
  - 6 paints (A, B, C, D, E, and F)
  - Emittance (room temperature): 0.8 to 0.9
  - Poor durability noted for A, B, C, and D
  - Selected E and F for further study
- Part II – 24 nickel 201 coupons (650 °C)
  - 2 paints (E and F from Part I)
  - 1 ceramic coating (NiO · Cr_2O_3-SiO_2 spinel bond)
  - Metal coupon surface finish
    - High-speed mill
    - Bead blasted (improve mechanical adhesion)
    - Oxidation treatment (several days at 850 °C in air)
  - Paint F – failed (all coupons)
  - Paint E – gray discoloration
  - Ceramic coating: durable, emittance ~0.81





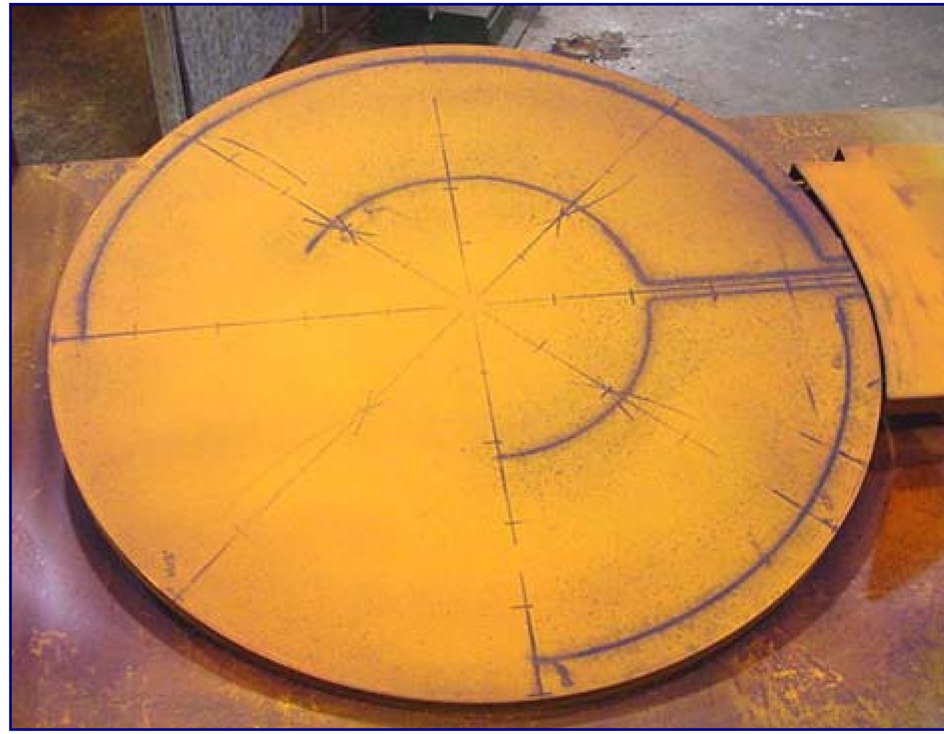


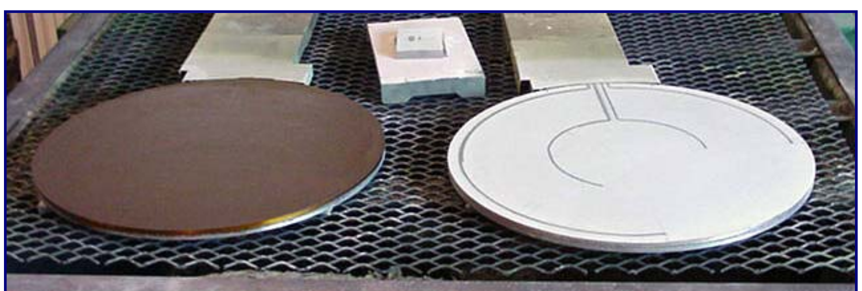
Ceramic Coating – Vendor

- Physical properties
  - Metal-ceramic bond (nickel, chromium, silicon oxides)
  - Thickness: 0.05 mm to 0.08 mm
  - Surface roughness: ~ 0.38 μm

- Application process
  - Initial coating
    - Grit blast (fine alumina grit) surfaces to be coated => brush clean
    - Spray apply initial coat (water-based slurry) => air dry
    - Fire in oven at 510 °C (air atmosphere)
  - Densification
    - Apply chromate solution at room temperature
    - Fire in oven at 510 °C (air atmosphere) => repeat 14 applications
    - Hand polish => 2 applications

Initial coating





Cold plate #1
End – Coating Process

Cold plate #2
Start – Coating Process

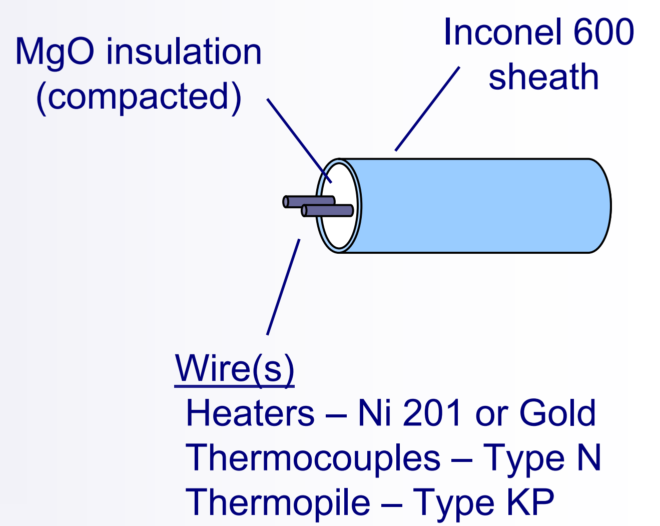
Hand polishing coating during densification process

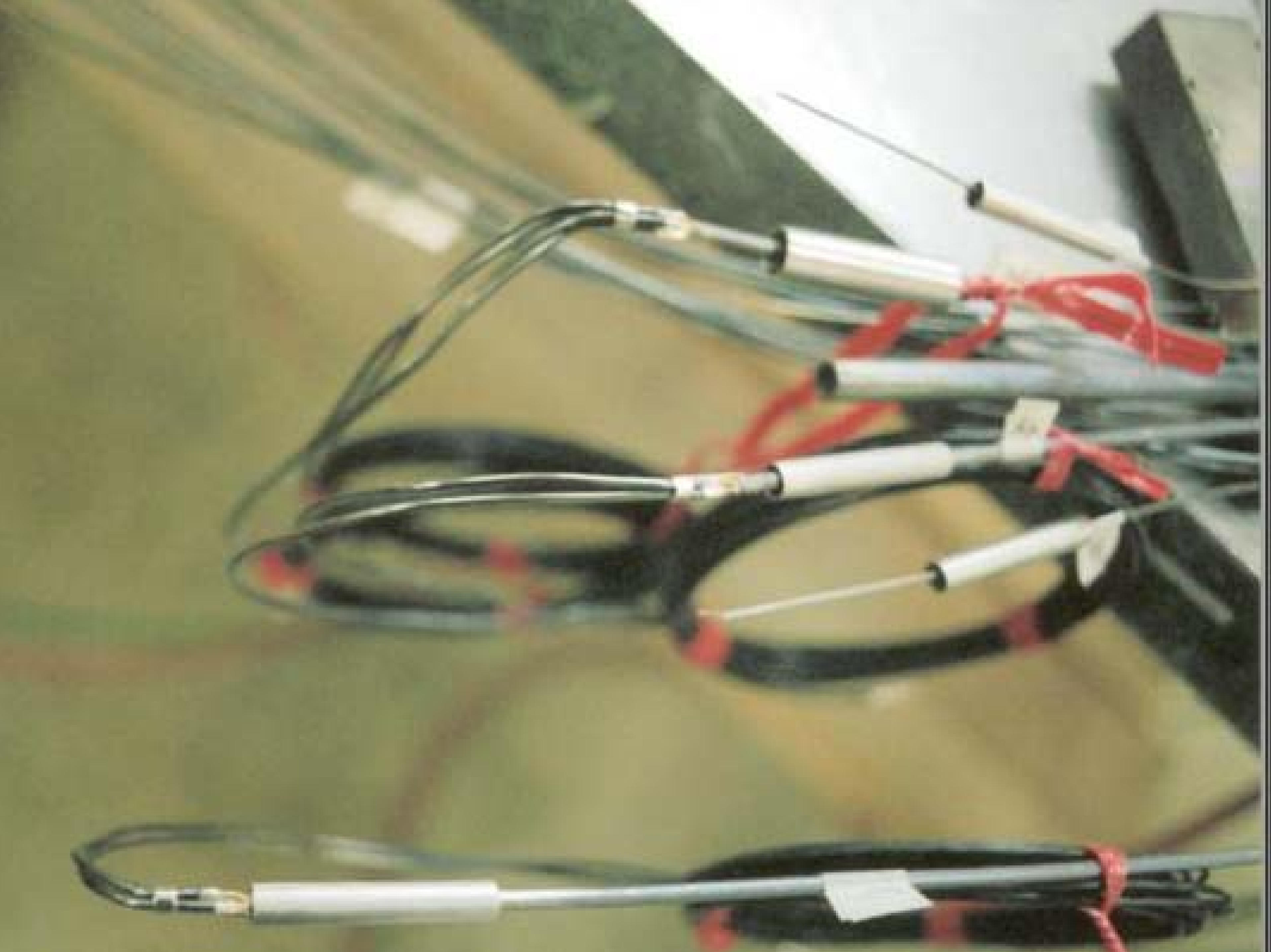
Fabrication of Hot Plate

- Metal cutting ⇨ form components
- Vacuum brazing ⇨ assembly
- Grinding ⇨ surface finish
- Ceramic coating ⇨ high emittance
- Electrical connections

Electrical Connections – Vendor

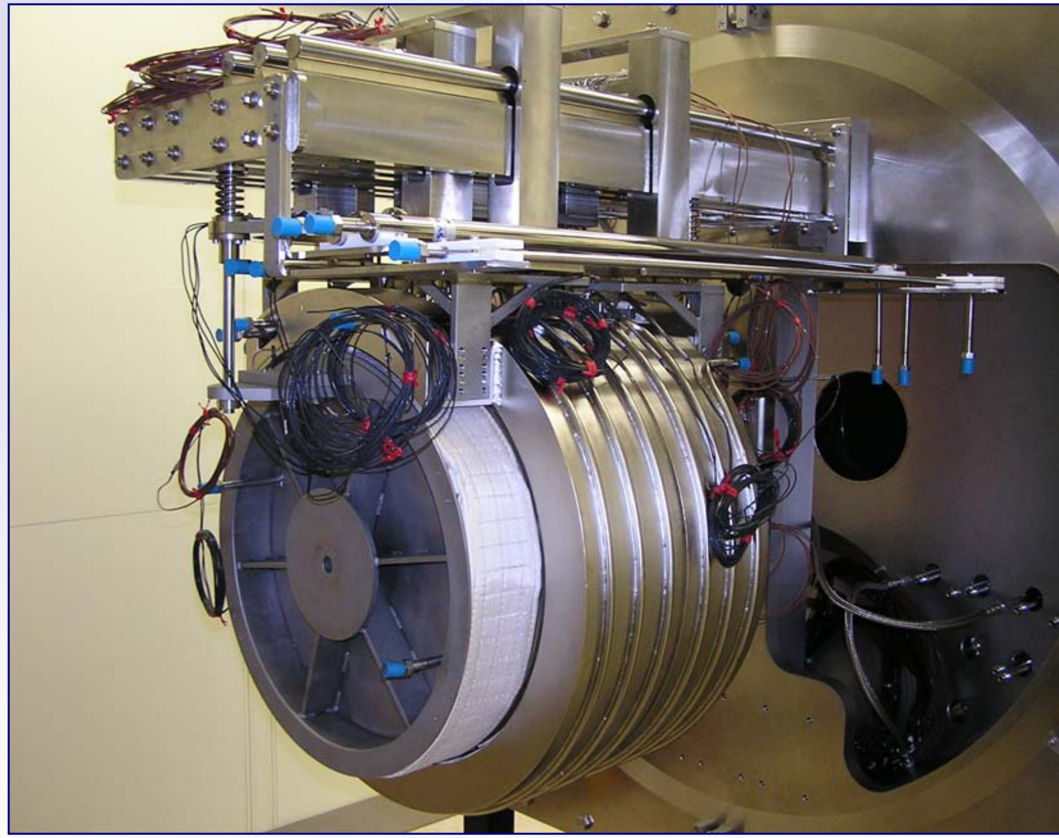
- Preparation
  - Cut sheathed cables to length
  - Remove metal sheath and MgO insulation
  - Expose wires and weld electrical terminations for extension wires
- Pot adapter
  - Slide tube over welds
  - Fill tube with moisture-resistant epoxy
- Check electrical continuity





Summary – Current Status

Front of apparatus



Installation of edge-guard assemblies, cold-plate assemblies, and hot plate

Future Work

- Remove and replace existing thermopiles (2) with new Type K thermopliles
- Dimensional metrology
  - Measure meter-plate area and uncertainty
  - Measure surface flatness and parallelism
- Complete data acquisition computer program
- Complete operational checkout
- Comparisons
  - Internal (NIST 1016-meter guarded-hot-plate apparatus)
  - External