Production process of alumina-ceramic vacuum chambers for J-PARC

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Abstract. In order to avoid the eddy-current effect due to a rapid-cycling magnetic field in the J-PARC proton synchrotron ring (348 m in circumference), several tens of ceramic chambers (3.5-m long and 500 mm in diameter, at their maximum) are to be installed. The 99.7% alumina ceramic ducts of sub-meter in length, having racetrack and circular cross-sections were sintered and jointed by braze. Several chambers of rectangular and racket-shape cross sections were also produced. A TiN film of 15 nm thick was formed inside the chambers for suppressing secondary electron emission, and electroformed copper stripes were plated outside for lowering the beam-impedance. The course of the production process, including polishing, metallizing and brazing to a titanium flange is reported. Outgassing rates were also measured for a few of the chambers by a conductance-modulation method.

1. Introduction
The Japan Proton Accelerator Research Complex, J-PARC, is to provide secondary beams, including neutrons, neutrinos and so forth, which are widely useful in scientific and engineering fields. The facility comprises a 400-MeV linac, a 3-GeV rapid-cycling synchrotron (RCS) and a 50-GeV synchrotron. The 3-GeV RCS is to be operated with a repetition rate of 25 Hz, in order to circulate a maximum average current of 333 µA and to provide a beam power of 1 MW to the experiment area. The chambers in the RCS are to be installed in a rapidly varying magnetic field. Alumina ceramic was chosen for use as the chamber material so as to avoid the eddy-current effect [1, 2, 3]. The alumina-ceramic chambers to be produced have the following characteristics to meet the RCS vacuum requirements:
(1) non-magnetic metal (Ti) flanges on both extremities (for Helico-flex delta-gaskets),
(2) adequate mechanical strength in ceramic-ceramic and ceramic-metal braze-joints,
(3) rf shield outside the chamber for lowering the impedance,
(4) TiN coatings inside the chamber for suppressing secondary electron emission, and
(5) low outgassing rate and high radiation resistance.
A total length of the ceramic chambers is about 200 m, while the circumference of the 3-GeV RCS is 348.3 m. In this report, the production process of the chambers is described.

2. Production process of alumina ceramic chamber
Several manufacturers sequentially carried out the processes as follows;
(1) sintering (1600˚C in air) a unit duct of 0.15-0.8 m long
(2) precise grinding both extremities, cleaning by acid solution and annealing (1300˚C, in air)
(3) metallizing (1400˚C wet hydrogen) layer of 7 to 10 µm thick by Mo-Mn compounds
(4) electroplating Ni of 2 to 5 µm thick on metallized surface, and then rinsing
(5) coating (260˚C in vac.) TiN film of 15 nm thick inside the unit duct
(6) brazing (820˚C in vac.) between the unit ducts, and also to a Ti sleeve by Cu-Ag eutectic alloy
(7) electroforming (26˚C sulfate electrolyte solution) high-purity copper stripes (rf shield) of 1.5 mm thick outside the chamber.

2.1. Sintering unit duct
The alumina ceramic of 99.7% purity with a grain size of 10 (std. dev=2) µm and with a specific gravity of 3.91 g cm⁻³, showing a flexural-strength of about 340 MPa (averaged), was chosen (ASS-S1, NIKKATO Co, Japan). The measured dielectric properties were ɛ=10.2, tan δ<10⁻⁴ at 3 GHz. The chambers produced are listed in Tables 1 and 2. Since it is difficult to sinter a large diameter chamber in length of 1 m or more without any deformation, segments of 0.5 to 0.8 m long are sintered as unit ducts, having circular and racetrack cross sections. For the chambers to be installed in the injection magnets, having racket-shape and racetrack cross sections, shorter unit ducts are necessary to suppress deformation. The moulded ducts were set vertically in the sintering furnace. In such configuration, the diameter in the bottom is liable to be larger than that in the top during sintering. Although the thicker wall mould gives a uniform shrinking ratio (15-20%), a thinner wall is preferable for obtaining a larger beam aperture. The optimized thicknesses of the circular ducts have been 7.5 to 8.5 mm: 9 to 10 mm for the ducts in injection magnets. From the statistical data of measured inner dimensions the deformation was within 2 mm (Table 1). Outside surface of some unit ducts was lapped after sintering, if necessary, so as to keep dimensions within tolerance.

| Table 1. Dimension of alumina ceramic chamber for dipole and quadrupole magnets [mm]. |
|-------------------------|---------|---------|------|------|------|------|------|
| magnet number of chamber | dipole racetrack | quadrupole circular |
| cross section          | 24      | 10      | 9    | 12   | 6    | 33   |
| outer dia.              | 261 - 203 | 394    | 314  | 314  | 314  | 274  |
| inner dia.              | 246 - 188 | 379    | 299  | 299  | 299  | 259  |
| length of chamber * 3540 (15°bend) | 1500 | 1600 | 740  | 1000 | 1300 |
| unit duct length        | 783.4   | 680.3   | 729.3 | 600.3 | 430.3 | 579.5 |
| unit ducts per chamber  | 4       | 2       | 2    | 1    | 2    | 2    |
| measured (unit duct)    |         |         |      |      |      |      |
| outer dia. (top/bot.)   | 264.7/266.5 - 202.9/202.9 | 397.1/397.7 | 315.1/316.6 | 274.5/275.1 |
| inner dia. (top/bot.)   | 246.9/248.6 - 188.4/188.4 | 380.3/382.0 | 300.1/301.7 | 259.6/260.3 |
| thickness (top/bot.)    | 8.4/8.4 - 7.2/7.3 | 8.4/7.9 | 7.5/7.5 | 7.4/7.4 |

* total length of transitions and flanges is 406 mm as compared with 140 mm in other chambers:
   the longer sleeve is chosen for the long and bending chamber with a racetrack cross section so that unnecessary stress in the braze joint of Ti-ceramic is avoided upon installation.

| Table 2. Dimension of alumina ceramic chamber for injection magnets [mm]. |
|-------------------------|---------|---------|---------|---------|---------|---------|
| magnet number of chamber | dipole rectangular | shift bump rectangular | injection-Q racket | bending racetrack | v-painting bump racetrack |
| cross section outer      | 318 - 268 | 479 - 289 | 502 - 117 | 233 - 146 | 130 - 114 |
| inner                   | 300 - 250 | 461 - 271 | 482 - 97 | 217 - 130 | 80 - 69 |
| length of chamber 770 | 1085 | 1340 | 1500 | 660 | 790 |
| unit duct length 315 | 275 | 120 | 155 | 60 | 520 |
| unit ducts per chamber 2 | 3 | 4 | 1 | 8 | 2 |
| thickness 9 | 9 | 10 | 8 | 8 |
2.2. TiN coating inside duct
A film of electrically conductive material, such as TiN, to be formed inside the duct should be thick enough so to suppress secondary electron emission, but thin enough to prevent any excessive heating due to eddy current effect. A secondary electron yield $\delta$ from the coated alumina can be described, by film thickness $t$, as,

$$\delta = B \exp \left( -\frac{t}{\lambda_{\text{film}}} \right) \times \int \frac{dE}{\beta \ dz} \exp \left( -\frac{z}{\lambda_{\text{alumina}}} \right) dz$$

(1)

where $\lambda$s are mean-free-paths of electron in the film and alumina, and $B$, $E$ and $\beta$ are an escape probability from the surface, a primary particle energy and an energy required for secondary electron generation. Since $\lambda$ in TiN film is estimated as 0.5 nm [4] while 50 to 100 nm in alumina [5], the thickness of 1 nm or more is enough to suppress secondary electrons. Eddy current loss becomes unacceptable, when the thickness exceeds 100 nm. The TiN film of 15 nm thick has been coated in every unit duct by a hollow-cathode-discharge method. In order to let plasma enter the duct from both apertures, the duct is rotated vertically in the coating chamber. Thickness deviation was within 3 nm and measured value of stoichiometry was 0.92 of N/Ti.

2.3. Brazing ceramics to titanium
Figure 1 shows the chamber for Q-injection magnet, consisting of ten of unit ducts, two of Ti sleeves (at both extremities) and flanges after braze process. Since, in the range from 600°C down to 400°C, thermal expansion of Ti is larger than that of alumina ceramics, residual stress thermally generated in the joint, should be reduced by a buck-up ring of alumina ceramic (figure 2). From the calculation based on a finite-element analysis, the thermal residual-stress on the alumina ceramic surface can be reduced to 200 MPa by the ring, indicating a margin of 140 MPa: indeed, the measured strength of test sample was 100 MPa or more.

The joint strength depends not only on the thermal stress but also on the mechanically introduced defects during grinding. The defects may cause failure of metallize layer and also induce degradation of sintering additives of CaO and MgO. As surface finishing, precise grinding of 2 µm deep, being shorter than grain size, has been adopted so as not to introduce grain damage, and 2 µm deep by 50 times was performed in order to remove the residual stress previously introduced by coarse grinding: usually, the depth of the stress in the coarsely ground ceramic surface is several tens µm. Sufficient wetting between titanium and braze i is also important. A thick oxide layer on titanium is to be removed by chemical polishing. The AES signal of Ti reaches 90% of that in bulk at 7 nm deep when the machined surface of 1 µm thick has been removed, while 17 to 24 nm deep when not removed.

![Figure 1. Ceramic chamber of racket-shape cross section.]( inserted image)

![Figure 2. Structure of a back-up ring.]( inserted image)
2.4. Outgassing measurement of the ceramic chambers

Since an alumina ceramic is sintered at high temperature and, originally, chemically stable oxide-compounds, the surface is considered to have a lower adsorption-energy for water molecules than those of metals. Further, a TiN film is effective to reduce the water-molecule adsorption due to its dense microstructure and chemical stability. The chamber is, therefore, expected to have a low outgassing rate, if the production process is carefully controlled so as not to introduce any excessive contaminations; in an electroforming process of copper-stripes, the chamber is sealed off from the electrolytic solutions by blank flanges. An outgassing measurement for several numbers of ceramic chambers was carried out by a conductance-modulation method. Figure 3 shows the outgassing rate measured for a ceramic chamber of 1 m long and 299 mm in diameter. After pumping for 50 hours, the rate decreases down to $10^{-8}$ Pa m$^3$ s$^{-1}$ m$^{-2}$, which is close to that for a surface-processed stainless-steel chamber (electrochemically buffed followed by baking) of 1 m long and 150 mm in diameter.

3. Summary

Long, large diameter, vacuum chambers made of alumina ceramic have been produced for use in proton accelerators. The wall thickness should be optimized for preventing deformation during sintering. The surface should be controlled so as to avoid residual stress and micro cracks, especially when a grinding process is necessary. Thermal stresses induced at a braze joint between titanium and ceramic can be reduced by using a backup-ring configuration. The measured outgassing rate of the chamber is low enough for accelerator use.

References

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