CONF-771029--152

DEVELOPMENT OF LARGE INSULATOR RINGS FOR
THE TOKAMAK FUSION TEST REACTOR*

T. Brown and A. Tobin

Grumman Aerospace Corp.
Bethpage, N. Y. 11714

Summary

This paper discusses research and development leading to the manufacture of large ceramic insulator rings for the TFTR (TOKAMAK Fusion Test Reactor). Material applications, fabrication approach and testing activities are highlighted.

Introduction

Problems were expected in developing a large metal bellows (96-inch OD) sufficiently convoluted to provide the high electrical resistance required for the vacuum vessel of the TFTR. Consequently, a parallel research and development effort was begun to determine the feasibility of developing equally large ceramic insulator rings as an alternate to the bellows. The physical size of these rings greatly limited the selection of dielectric materials and the fabrication techniques. Preliminary evaluation resulted in choosing Corning Glass Works machinable glass ceramic MACOR as the baseline dielectric material for rings, to be made in segments, with titanium to serve as the metal sealing ring, based on its thermal expansion match to MACOR.

With further design refinements, the ability to fabricate a metal bellows proved feasible and subsequently the requirements for a parallel development of a ceramic insulator ring was not critical for this purpose. How-

ever, the design and fabrication techniques for large non-metallic insulators of approximately 48-inch OD were required for neutral beam and vacuum pump ducts. This smaller size opened the possibility of using a monolithic porcelain ring as the insulator material in addition to the segmented MACOR approach.

This paper emphasizes the design and fabrication techniques developed for making large segmented MACOR insulator rings and also discusses the techniques being used at Lawrence Livermore in making a 30-inch OD porcelain ring.

Design Configuration

Figure 1 illustrates a possible configuration of a 48-inch resistor element made up of either MACOR segments joined together with a high-temperature glass ceramic seal or a continuous porcelain ring. Using MACOR, a thin ring of stainless steel is sandwiched between two thicker rings of titanium located on each side of a MACOR ring. The symmetrical requirements of the SS/Ti rings are for isolating the MACOR against the thermal stresses which occur when the entire configuration is joined at the brazing temperature. When using a porcelain material, OFHC soft copper is sandwiched between the porcelain. Both arrangements are enclosed in a structural housing to isolate the insulator rings against operating and handling loads.

Fig. 1 Insulator Design Configuration

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, make any warranty, express or implied, or assumes any liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would infringe privately owned rights.

*This work was supported by United States Energy Research and Development Administration Contract E(11-1)-3073 with Princeton University, Princeton, New Jersey

1

MASTER

REAR

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Material Selection

High-alumina ceramic insulator rings and techniques for brazing them to metal are commercially available; however, the maximum size commercially fabricated is on the order of 22- to 30-inch OD. Princeton and Rutgers Universities jointly developed 40-inch diameter high-alumina ceramic rings for their PLT (Princeton Large Torus) machine. Unfortunately they developed leaks which were sealed with RTV. These results showed that further search into other possible materials was needed.

MACOR, a machinable glass ceramic, is a unique, partially-crystalline structure which can be machined to precise tolerances with ordinary metal-working tools; it requires no additional firing after the part has been made. This capability plus its relatively high use temperature (1000°C), low electrical conductivity and thermal coefficient of expansion compatibility with titanium, makes the material particularly attractive. Although there had been little development work in the area of high-temperature joining techniques with MACOR, it was anticipated that the brazing temperature required might be as high as 900°C. This was expected to complicate the development of a glass frit (required for joining MACOR segments into large rings) that will survive the brazing temperature. While Corning sought to determine the feasibility of providing high-temperature glass frits and developing techniques to join MACOR to MACOR, Grumman's efforts were aimed at developing a technique for making a leak-tight seal between titanium and MACOR.

MACOR-to-Titanium Seal

The approaches pursued in obtaining a brazed joint between MACOR and titanium were titanium hydride plus silver braze, metallized layer plus aluminum braze, and active metal braze.

Titanium Hydride Process

In the titanium hydride process the MACOR surfaces to be joined are painted with a slurry of titanium hydride powders suspended in an ethylene glycol carrier. The braze alloy foil (0.003 inch thick) is placed between the painted hydride layer and the titanium, and the assembly is clamped together. On heating in a vacuum (10^{-5} mm Hg) the carrier evaporates, leaving a titanium hydride deposit. At 350°C the hydride decomposes into a chemically active form of titanium and hydrogen gas. At the brazing temperature, the silver braze alloy reacts with the titanium to form an active metal braze which wets and bonds to the ceramic.

Three braze alloys that were considered for the titanium hydride and metallized layer process were: (1) silver-copper eutectic (CuSi1 - 860°C) (2) silver-palladium-gallium (Gapasil 9 - 920°C) and (3) commercially pure aluminum (Reynolds Wrap - 700°C).

Metallized Layer Process

In the metallized layer process a 1 to 2 micro-inch layer of nickel is sputtered on the MACOR surfaces. The braze filler material (either CuSi1, Gapasil or aluminum) is sandwiched between the nickel-sputtered MACOR and the titanium specimens. Upon heating to the braze temperature, the braze alloy wets and bonds to the metallized layer and the titanium. Joint integrity is determined by the adherence of the metallized layer to the ceramic.

Active Metal Braze

In the active metal braze process, powders of active metals (e.g., titanium, zirconium, beryllium, nickel and copper) are physically mixed in a blender for 24 hours. The powders are then mixed with an ethylene

glycol carrier and painted directly onto the MACOR surfaces which are clamped to the titanium and heated to the brazing temperature. The ethylene glycol evaporates and leaves no residue during the heating stage. The alloy powders sinter and fuse together to form a liquid active-metal braze which wets and bonds to both the ceramic and titanium surfaces. These braze powders showed excellent wetting characteristics to MACOR. As the active metal powders were not pre-alloyed, somewhat higher brazing temperatures were necessary to cause fusion and flow of the braze (920 - 1000°C). Because of its good flow characteristics and low melting point, a 35% Ti-35% Zr-30% Cu braze composition was chosen to fabricate titanium-MACOR seals.

Metal-to-Metal Seals

The ceramic insulator rings proposed must be joined to a stainless steel housing. This would require the fabrication of a titanium-stainless steel brazed joint in which there would be a large expansion mismatch; however the strength and ductility of the metals involved should accommodate the stresses developed by cool down from the brazing temperature.

The survey of available brazing materials indicated that the same three brazing materials used in the hydride and metallized layer processes for sealing MACOR to titanium offered the most promise for successfully joining 6AL4V titanium and 304 stainless steel.

Specimen Preparation and Assembly

As the quality of brazed joints is strongly dependent on surface preparation, all surfaces to be brazed were carefully cleaned prior to assembly. Titanium surfaces were degreased in acetone, cleaned in a hydrofluoric-nitric acid etching bath for 2 to 3 minutes, and then ultrasonically rinsed in distilled water for 5 minutes. Stainless steel surfaces were degreased in acetone, etched in a nitric acid-hydrochloric acid bath, and then ultrasonically rinsed in distilled water for 5 minutes. MACOR surfaces were degreased in acetone, rinsed in methanol and boiled in distilled water for 5 minutes. All surfaces were carefully wrapped in lens tissue prior to brazing and the braze alloy compositions were degreased in acetone prior to use.

Braze alloys in the form of 3-mil ribbon were cut and sandwiched between the surfaces to be joined. The number of braze strips used varied from one to three. The joint was held in place by either clamps or weights placed over the specimen.

Brazing Procedures

All brazing operations were performed in a water-cooled vacuum furnace. Specimens were placed on a mullite support pedestal in the center of the furnace. Tungsten elements heated the specimens by direct radiation with a usable hot zone of 6 x 6 x 6 inches. A

vacuum of 1 to 5×10^{-5} mm Hg was maintained during the brazing operation. A viewing port in the furnace permitted direct observation of the melting of the braze alloy. A thermocouple located near the specimen was used as a guide to the actual temperature; however, the braze alloy did not melt until the thermocouple recorded 20 to 40°C above its known melting point. Brazing time was counted from the time the braze was observed to melt until it flowed into the joint. After holding the specimen at the brazing temperature for 5 to 25 minutes the furnace was turned off and allowed to cool for 2 hours to about 300°C. Argon was then bled into the furnace and the specimens were removed.

Metallographic Examination of Brazed Joints

Each specimen was cut across the interface of the brazed joint, mounted and polished to permit metallo-

Fig. 2

graphic examination. In all cases, there was no significant penetration or degradation of the MACOR by the braze alloy; however, where the active metal braze was used, a distinct reaction layer was observed at the titanium surface. In some instances there was evidence of localized void formation near the interface of the active-metal braze and the titanium. Micrographs of the various interfaces are shown in Figure 2, views A through E.

Test Results

In order to screen the different braze materials and joining techniques, over one hundred coupon test specimens were joined and tested. A single lap shear joint was used to determine the shear strength between GALV titanium and 304 stainless steel using different braze alloys. A shear strength value of MACOR-to-titanium joints was obtained by loading each MACOR sample in compression as shown in Figure 3. The MACOR block was ground parallel to the lower surface of the titanium to eliminate any torsional stresses and the test fixture was used to support the bending loads generated by the eccentricity of the coupon.

Gapasil 9 and CuSil brazing alloys gave the best results in joining titanium to stainless steel, demonstrating an average shear value of 4500 psi. In the

single lap joint arrangement, distortion of the parts resulted from the mismatch in thermal expansion coefficients of titanium and stainless steel. This distortion can be minimized by making a symmetrical (Ti/SS/Ti) configuration; however a residual stress will remain within the joint upon cooling down from the brazing temperature.

Fig. 3 Macor Shear Test

Five types of failure modes were observed in the titanium-to-MACOR joints.

1. At the interface of the ceramic surface and metallized layer
2. Within the ceramic
3. At the active metal layer and braze interface
4. Within the braze
5. At the metal braze interface

Failure mode 1 resulted from a weak joint; however no clear-cut strength correlations were exhibited between failure modes 2 through 4. The metallized layer referred to in failure mode 1 was nickel sputtered on the MACOR surface. Braze joints using aluminum, CuSi1 and Gapasil 9 in conjunction with the nickel sputtered MACOR were so weak that they delaminated at the interface of the MACOR and metallized layer during handling. Further work with this approach was discontinued since it was felt that the adherence of the metallized sputtered layers could not be significantly improved. A more adherent metallized layer of either nickel or titanium can be obtained by ion plating than can be achieved by the sputtering method. Data on shear strength by this approach was not available at the time of writing this paper.

The strongest joints were obtained using a CuSi1 braze alloy with titanium hydride painted on the MACOR surface. This typically occurred when a high percentage of the area of failure was in the MACOR, indicating that the ceramic-metal joint strength was greater than the cohesive strength of the MACOR. Shear strength ranging from 2400 to 7000 psi was recorded; the latter value was obtained through refinements in the brazing techniques. The higher shear values were reached when a 3-mil sheet of CuSi1 and a thin layer of titanium hydride were applied to the MACOR surface.

MACOR-to-MACOR Seal

Corning Glass Works' efforts in developing high-temperature glass frits and techniques to join MACOR followed a survey of released (commercial) and unreleased (laboratory stage) glasses and glass-ceramics which might prove to be likely candidates. With feasibility substantiated and glass frit candidates chosen, Corning conducted coupon tests of mechanical strength, adhesion, wetting, surface tension, and thermal expansion compatibility between frits and MACOR. The most promising candidates were subjected to further mechanical, vacuum testing and joint configuration tests for modulus of rupture (MOR). The final phase will involve the fabrication of one or more full size test specimens made up of the best joint configuration and glass frit which meets the brazing temperature requirements.

Developing a high-temperature, vacuum-tight joint between MACOR segments has proved to be the most difficult task in achieving a segmented MACOR ring. Eight glass compositions showed promise at temperatures varying from 750 to 1000°C. All of the glasses produced seals, but small bubbles (0.0002 inch diam-

eter) were abundantly dispersed throughout the seal. To eliminate these bubbles candidate glasses were made into thin glass micro sheets, sandwiched between two MACOR segments, and cured under the combined effects of temperature-time-pressure and ultraviolet radiation. These seals have shown complete mechanical integrity, hermetic with an 8000 psi MOR after being subjected to an 860°C vacuum reheat cycle.

Lawrence Livermore Work

Recent work at Lawrence Livermore Laboratories has shown success in brazing two 30-inch diameter porcelain rings to a copper interlayer ring using a silver braze alloy and titanium hydride.

Although there is a large expansion coefficient mismatch between these two materials (9.8×10^{-6} for Cu vs 3.2×10^{-6} for the porcelain) the success of the seal depends on the low yield strength of the copper as well as the thickness of the copper interlayer. Presumably, the optimum thickness of copper (0.030 inch) allows complete yielding throughout the copper layer thus minimizing stress buildup in the porcelain on cool down from the braze temperature. Experience has shown that careful scrubbing of the ceramic surfaces and electropolishing the copper are important to effecting a good seal. Best results were obtained with a nickel modified silver-copper eutectic (Ni CuSi1 3) and TiH₂ active metal powders applied to the ceramic surface. A 5-psi stress was applied to the mating surfaces via dead weights.

Any variations in tolerance across the surfaces to be sealed are apparently overcome by flow of the glassy phase in the porcelain during the sealing operation due to combined effects of temperature time and stress. These techniques appear to be adaptable for scaling up to 48-inch porcelain-copper ring assemblies as would be required by the TFTR.

Acknowledgements

The authors wish to thank Mr. W. Poit of Grumman's Material Research Department who performed all the brazing operations.

References

1. G. Lewis and D. Mullaney, Fabrication of a 40 Inch Diameter Ceramic To Metal Steel For PLT, Proceedings of Sixth Symposium on Engineering Problems of Fusion Research San Diego, California, November, 1975.
2. W. Lamb, W. Dexter, T. Duffy and N. Land, Work performed at Lawrence Livermore (private communication).
3. Joining Ceramics and Graphite to Other Materials 1968 NASA Publication NASA SP-5052, Edited by H. Pattee, R. Evans, R. Monroe.
4. NASA Contribution to Metal Joints, 1967 NASA Publication NASA SP-5064, Edited by W. Reichenecker and J. Heuschkel.