# Innovative Isostatic Processing Technologies for Defence and other Strategic Applications

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#### ABSTRACT

Powder Metallurgy (P/M) involving Cold Isostatic Pressing (CIP) and Hot Isostatic Pressing (HIP) techniques is the best suited processing route for manufacture of critical components from ceramics and highly complex metallic materials. The CIP followed by sintering route was adopted for the development of fused silica radomes for application in high-speed target seeking missiles. On the other hand, the HIP, a single-step processing technique has been used for the development of First-Wall (FW) of Test Blanket Modules (TBMs) from India Specific Reduced Activation Ferritic Martensitic (INRAFM) steel. The FW is an important component required for International Thermo-nuclear Experimental Reactor (ITER) application. The details of innovative processes which have been established to overcome the specific technical challenges for the development of CIP and HIP technologies for manufacture of silica radomes and INRAFM steel first-wall respectively for intended applications are highlighted in this paper.

Keywords: Powder metallurgy; Cold isostatic pressing; Hot isostatic pressing; Radome; TBMs

## 1. INTRODUCTION

In the spectrum of advances in materials processing, the near-net shape technologies are in the forefront, in view of the technical and economical advantages they offer over the conventional manufacturing methods. The Powder Metallurgy (P/M) is one of the most prominent near-net shape technologies finding rapidly increasing applications for manufacture of a wide-variety of critical components from complex materials<sup>1</sup>. The elemental or pre-alloyed powders with the particle size between 5-100 µm are normally used in conventional P/M for green compaction followed by sintering route to realize the components in desired shape, size and properties. The sintered products are subjected to nominal machining to obtain the final dimensions. However, the conventional P/M products usually have residual porosity and also are limited to low aspect ratio ( $\leq 2$ ). Therefore, the conventional P/M is not preferred for manufacture of critical rotating parts for long term applications.

In order to overcome this limitation, the P/M in combination with isostatic processing techniques such as Cold Isostatic Pressing (CIP) and Hot Isostatic Pressing (HIP) is adopted. In the case of CIP, the powder to be compacted is filled into the flexile rubber moulds of desired shape and subjected to high pressure (typically about 200 MPa) by using water as the pressure transmitting medium. Since, powder compaction takesplace at room temperature in CIP, the CIPed objects are subsequently sintered or hot isostatically pressed to obtain the desired density, microstructure and targeted mechanical properties<sup>2</sup>. On the other hand, the HIP is a most powerful

and versatile technique that involves simultaneous application of both high pressures (200 MPa) and high temperature of upto 2000 °C. The HIP offers near 100 % theoretical density of the material in a single- step. It has a greater potential for solid state diffusion bonding, defect healing of castings and consolidation of powders into near net shapes with complex geometry. The powders tobe HIPed are filled into pre-shaped containers fabricated from sheet metal and then evacuated sealed under dynamic vacuum conditions. After HIPing, the sheet metal encapsulation is removed by machining/chemical leaching to obtain the final product<sup>3-4</sup>.

The authors' laboratory (Defence Metallurgical Research Laboratory) is in the field of P/M and isostatic pressing technology since 1977 and over the years, the laboratory has developed considerable expertise in processing of different types of high temperature materials and manufacture of critical components for applications in defence, space and atomic energy sectors<sup>5</sup>. In the present context, it is aimed to bring out the innovative processes which have been established to overcome the challenges associated with the development of two prominent technologies viz., (i). Fused Silica radomes by cold isostatic pressing route for missile applications, (ii). First-wall (FW) of Test Blanket Modules (TBMs) from INRAFM steel through HIP route for ITER. The details if these technologies are presented in the following sections.

## 1.1 Development of Fused Silica Radomes by CIP Route for High Speed Target Seeking Missiles

Fused silica radomes are the most critical components of high speed target-seeking missiles. The radomes are integrated with seeker and positioned at the fore-front of the missile to

Received : 21 November 2022, Revised : 13 December 2022 Accepted : 02 February 2023, Online published : 06 April 2023

protect the navigation system from adverse environment. To enable the seeker to receive signals and track the target, the radomes have to be electromagnetically transparent while exhibiting required levels of physical, mechanical and thermal properties. Based on its excellent thermal shock resistance and low dielectric permissivity, fused silica is the preferred ceramic material for manufacture of radomes in different size and shape depending on the functional requirements of missiles<sup>6</sup>. Slip-casting or Gel- casting are the techniques commonly adopted for processing of fused silica radomes. However, the component rejection rate is very high due to prevalence of non-uniformly distributed porosity that leads to cracking of radomes either during machining to final profile or catastrophic failure in service. To overcome the above limitations, in the present study, Cold Isostatic Pressing (CIP) and sintering route for consolidation of fused silica powder has been adopted and successfully identified: (i) Powder characteristics (ii) Binders (iii) CIPing parameters (iv) Sintering reagents and (v) Sintering schedules to achieve the required microstructure and targeted properties for radome applications.7.

The potential of CIP processing route for manufacture of radomes conforming to different aspect ratios and profiles have been demonstrated successfully<sup>8-10</sup>. The CIPed silica radomes were subjected to various qualification tests and have been found acceptable for intended applications based on their excellent electromagnetic (EM) performance and thermostructural strength. The CIP technology is being adopted for large scale production of radomes for a wide-range of indigenously developed missiles. Manufacture of fused silica radomes through CIP technology has the advantage of high yield of component with isotropic properties. The CIP technology uses indigenously available low-cost raw- materials and hence, is cost effective. The technology also significantly contributes to self-reliance in missile defence systems, particularly for the seeker technology. The challenges in processing of silica with a special emphasis on development of radomes by CIP + sintering route are presented in this paper.

## 1.2 Development of First-Wall of Test Blanket Modules (TBMs) by Hot Isostatic Pressing Route for International Thermonuclear Experimental Reactor

International Thermonuclear Experimental Reactor (ITER) is planned to be constructed at Cadarache, France to demonstrate production of unlimited, clean and safe energy from atomic fusion for peaceful applications. The India Specific Reduced Activation Ferritic Martensitic (INRAFM) steel is selected as the candidate material for fabrication of First- Wall (FW) of Test Blanket Modules (TBMs) in fusion reactor due to its inherent beneficial properties at high temperature. The INRAFM steel contains alloying elements which do not become radioactive when irradiated, or if activated, the radioactivity decays quickly<sup>11</sup>. The use of such material would simplify the problem of after-service waste disposal of radioactive structures of fusion reactors. The firstwall of Test Blanket Modules (TBMs) is the crucial component of fusion reactor intended for shielding and heat extraction from thefusion reactor. The dimensions of the First-Wall (FW)



Figure 1. Dimensions of Indian LLCB-TBMs First-wall.

of blanket modules as per the Indian design are presented in Fig. 1. Helium is circulated through the 20x20 mm square channels at a temperature between 450-500 °C under a pressure of 8.0 MPa. Fabrication of First-wall (FW) of TBMs for fusion reactor is pronounced to be highly challenging and multi-disciplinary task due to its large size and complex shape.

The conventional materials joining techniques such as welding and brazing etc; are not acceptable in the First-Wall fabrication, as it is a plasma facing structure. In order to overcome this problem, in the present study, Hot Isostatic Pressing (HIP) has been selected for the development of FW from INRAFM steel, as this technique has substantial potential for solid state diffusion bonding of highly complex materials without affecting the original microstructure<sup>12</sup>. Through sustained R&D efforts, the HIP diffusion bonding parameters were successfully established for INRAFM steel and then fabrication of scale-down version of First-Wall of TBMs with required channel size and configuration has been successfully demonstrated<sup>12-14</sup>.

The HIP technology for fabrication of FW of TBMs has been accomplished in a phased manner viz., (i) Establishment of HIP diffusion bonding parameters for INRAFM steel bars of 15 mm dia. x 40 mm length to realize butt joints with targeted strength levels/ bond integrity (ii) Diffusion bonding of plain steel plates of 100 x100 mm square and 15 mm thickness to achieve lap-joints (iii) Fabrication of three channel plate mockups with square channels and thin-wall ribs with the aid of specially developed high density non-reactive rigid ceramic inserts and finally (iv) development of sub-size First-Wall in U/C shape configuration with specified number of square channels and the important aspects of it are presented in this paper.

#### 2. EXPERIMENTAL WORK

### 2.1 Experimental Details on Development of Fused Silica Radomes

The fused silica powder used in the present study is sourced from M/s. Chettinadu Morimura Semiconductor, Pvt.

Table 1. Chemical composition of fused silica powd
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Silica content	Balance
$\overline{\text{Fe}_{2}\text{O}_{3}}$ content	< 35 ppm
Combined Alkali content	< 50 ppm

Table 2. Flysical properties of fused sinca powder	fable 2. Phys	sical properties	of fused s	silica powd	er
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Particle morphology	Irregular	SEM – Fig. 1	
Particle size range	0.1-200 mm	Laser beam	
Median particle size	10-20 mm	particle sizeanalyser	
Apparent density	30-40 % of TD	TT 11 (1 )	
Tap density	40-50 % of TD	Hall-flow meter	
termine and to take t			

\*TD. Theoretical Density of fused silica : 2.2 g/cc

Ltd, Chennai, India. The purity and physical properties such as particle shape, size, size-distribution, and median particle size as well as the amorphous structure of silica powder are very important for radome applications. In view of this the composition of as-received powder was verified through wet chemical analysis and is given in Table 1.

The powder morphology was examined using FEI make scanning electron microscope (SEM) and is shown in Fig. 2. On the other hand the physical properties were determined by their respective ASTM standard procedures and are listed in Table 2. The amorphous state of the powder was confirmed



Figure 2. Particle morphology of silica.

using the Philips make Xpert diffractometer with Cu-K  $\alpha$  radiation at a scan speed of 1° per min. between 2 $\theta$  of 10- 70°.

The silica powder after characterization was add mixed with suitable sintering reagent andbinder solution (about 1-2% w/v camphor-acetone in 16:3 ratio). The wet powder was dried for nearly 45 minutes. The dried powder was gently crushed and filled into cylindrical shape latex rubber moulds of 30-40 mm diameter and length up to 80-100 mm with 3 mm



Figure 3. Influence of sintering parameters on the density and phase transformation: (a) Density Vs. Temperature, (b) Density Vs. Sintering Time, (c) XRD- Profile on the sintered material with variation in time and temperature and the corresponding density values

wall thickness. The powder filled moulds were plugged with rubber bungs and top-covers/caps to prevent water penetration during compaction through Cold Isostatic Pressing. The CIP compaction of powder was performed using the CIP equipment manufactured by M/s. Autoclave Engineering, USA. The CIPing of powder was carried out by varying the pressure ranging from 150 to 200 MPa with a dwell period of 3-5 min. After CIPing, the rubber bungs and the top covers/ caps as well as the moulds are gently removed to realize the green compacts. To process the radomes of specific shape/ profile appropriately fabricated rubber moulds and mandrels were used for CIPing of silica powder. Preparation of CIP tooling such as patterns and mandrels was accomplished by accounting for the shrinkage characteristics of powder during CIPing (10-15 %) and sintering (2-6 %).

In order to achieve the required density and mechanical strength, the CIPed green compacts in cylindrical shape were subjected to sintering in ambient atmosphere using a front loading furnace manufactured and supplied by M/s SM Enterprise Pvt Ltd., Hyderabad. The sintering was carried out on the CIPed compacts at different temperatures ranging from 1050-1400°C with different soaking time intervals followed by furnace cooling to room temperature. The sintered material was tested for basic properties such as density and structure through XRD technique. The experimental details in sintering of CIPed materials and the corresponding information on density as well as transformation characteristics are graphically presented in Fig. 3. As depicted in Fig. 3(a) in one set of experiments, the soaking time of sintering was kept constant (3 hrs) and the temperature was varied between 1050 and 1400 °C.

In another set of experiments, the sintering temperature of 1200 °C remained unchanged but time of soaking at this temperature was varied between 2 and 12 hrs as described in Fig. 3(b). The resulting material was characterized for its density and transformation characteristics (amorphous/ crystalline). The XRD information on the CIPedsilica material subjected to sintering at different temperatures and soaking time periods together with the corresponding density is presented in Fig. 4. Based on the density and transformation characteristics of sintered material, efforts were made to establish powder conditioning schedules and suitable sintering reagents to achieve the desired combination of properties by sintering the



Figure 4. XRD pattern of CIP + sintered silica processed under optimised conditions showing amorphous state.

CIPed silica between 1100-1225 °C for soaking time of 2-4 hrs followed by furnace cooling to room temperature<sup>9</sup>.

The CIPed and sintered round bars processed under optimized conditions as shown in Fig. 5, were used for fabrication of test specimens for measurement of density and XRD analysis. Subsequently, the electro-magnetic properties such as die-electric constant (K) and loss tangent ( $\delta$ ), thermal



Figure 5. CIP and Sintered silica bars.



Figure 6. Microstructure of CIP + sintered silica.

properties and flexural strength (by three point bend test) at RT and at elevated temperature were evaluated.

The samples weighing 5-10 grams with regular geometry were prepared and the density was measured by Archimedes principle in accordance with ASTM B311-08. The structure of the sintered silica was verified through X-ray diffraction technique as per the test conditions mentioned earlier. The intensity-vs- Bragg angle (20) profile of sintered material is shown in Fig. 4. The CIPed and sintered silica samples were examined in optical microscope as well as scanning microscope (SEM). The microstructure of CIP+sintered silica is shown in Fig. 6.

It is seen that the sinteredmaterial is free from cluster of pores and contains fine and coarse silica grains. The electromagnetic properties (dielectric constant -K and tangent loss- $\delta$ ) of the CIPed and sintered silica were determined by using data network analyzer through wave guide method in the frequency range of 12-18 GHz in accordance with ASTM D5568-08/ASTM 2520. The thermal properties such as CTE, thermal diffusivity and Cp were evaluated by dilatometry, laser flash method and calorimetry respectively upto 600 °C in accordance with ASTM C1470-06. To evaluate the flexural strength, the test specimens of rectangular cross-section (4 x 3) mm and length of 50 mm were prepared. The flexural strength of the CIPed and sintered samples was evaluated by three point bend test using Instron 5500 R UTM with 50 kN load cell and cross head speed of 0.5 mm/minute at RT and at elevated temperature in accordance with ASTM C1161 – 02c and ASTM C1211 – 02 respectively.

Upon achieving the required properties in test coupon level, processing of sub-size radomes followed by development of full-size radomes in required configurations wasperformed. The as-sintered radomes were visually examined to ensure that they are free from cracks and undesirable features and subjected to X-ray radiography using a 450 kv rated Philips made machine. The CMM measurements of the sintered radomes were performed and the resulting data were compared with actual radome profile and then released for machining. The CNC lathe machines were used for machining of sintered radomes by employing diamond tip tools to achieve the final profile and surface finish. The machined radomes (Fig. 7) were finally subjected to radiographic inspection and profile measurement to ensure that they are free from cracks and other defects and conforming to required profile.



Figure 7. CIPed and sintered silica radome after machining to final profile.

#### 2.2 Experimental Details on the Development of First-Wall of TBMs

In the present study, HIP diffusion-bonding experiments were carried out on India Specific Reduced Activation Ferritic Martensitic (INRAFM) steel developed and supplied by IGCAR, Kalpakkam<sup>13</sup> with an ultimate aim of development of first wall (FW) of Test blanket modules for ITER applications. The chemical composition of the steel is given in

Table 3. Chemical composition of INRAFM steel\*11

Elements	Cr	W	Mn	V	Та	С	Fe
Wt.%	8.9	1.4	0.54	0.22	0.06	0.08	Bal.
						0.0.7.	

\* Impurities (wt.%): Nb-0.001, Mo-0.001, Co-0.046, Si-0.056, Al-0.013, O-0.0057, Ni-0.002, Cu-0.006, Ti-0.005, B-0.005, P-0.002, S-0.002

Table 3. Establishment of HIP diffusion-bonding parameters for INRAFM steel has been an important aspect of this study to obtain the satisfactory level of bond strength. To begin with, round bars of about 15 mm diameter x 40 mm length were used for conducting lab- scale HIP bonding experiments. The round bars with different surface finish in the range of 1.5  $\mu$ m – 0.4  $\mu$ m, were encapsulated with 2.0 mm thick stainless steel capsules of suitable internal diameter and height of about 85 mm to achieve the butt joints. The encapsulated steel bars were evacuated at room temperature to attain the vacuum level of 6.6 x 10<sup>-3</sup> Pa. Subsequently, the capsules were introduced into a vertical furnace for vacuum hot degassing at a temperature of 500 °C - 800 °C for about 5-8 hrs and then crimp-sealed under dynamic vacuum level of 6.6 x 10<sup>-4</sup> Pa.

The vacuum sealed capsules containing the round steel bars were loaded into Lab-HIP of model EPSI-11144 (2003). HIPing cycles were carried out at different temperatures ranging from 980-1240 °C under varied pressures between 100-140 MPa for 3-5 hrs of sustaining time. It may be noted that the as-received INRAFM steel samples were also subjected to HIPing along with those capsules containing the steel-bars for characterization purpose both in HIPed and HIP bonded conditions. The HIP bonded samples were de- canned by mechanical machining and the selected sets of samples were given a heat treatment consisting of austenitizing treatment at 950 °C/2 h/air cooling (A/C) to room temperature (RT) followed by tempering at 750 °C/2 h/air cooling to room temperature. The HIPed joints were tested by various NDT techniques available in the laboratory including dye-penetrant test, X-Ray radiography and ultrasound testing.

The as-HIPed and HIP+heat treated samples were polished as per the standard metallographic techniques and etched with aqua regia solution, which is a mixture of nitric acid and hydrochloric acid in a molar ratio of 1:3. The as-HIPed and HIP+heat treated samples were examined in optical microscope to understand their microstructure at the joint interface both in as-polished and etched conditions.

Scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) were carried out on the selected samples. Line scanning and X-ray mapping techniques were used to identify the constituents of the microstructure at the joint interface and then to find out the extent of diffusion of different elements across the joint interface. Micro-hardness was measured across the joint interface of the HIPed INRAFM steel sample under an applied load of 10 kg to understand the approximate bond strength.

Further to evaluate the bond strength of the HIP bonded and HIP bonded + heat treated samples, the tensile test specimens were prepared in accordance with ASTM E-8 standard. A special care was exercised in machining of specimens to ensure that the joint interface lies well within the gauge length region of the test specimens. Tensile specimens were also prepared from the plain steel samples subjected to HIPing and heat treatment. Tensile tests were carried out on the as-HIP bonded and HIP bonded + heat treated specimens, as well as those of plain steels subjected to optimized HIPing and heat treatment at a cross-head speed of 1 mm per minute which results in a strain rate of  $1 \times 10^{-4} \text{ s}^{-1}$  for the test specimens having the gauge



Figure 8. Grooved INRAFM steel paltes.

length of 25 mm used in this study. The tensile properties of the HIP bonded materials after heat treatment were evaluated at room temperature and at 500 °C. Various experimental details stated above have been reported elsewhere<sup>12-14</sup>, however, the microstructure and tensile properties of HIP+heat treated materials together with some details on fabrication of channel plate mock-ups and sub-size first wall of TBMs are described in this paper.

To fabricate the channel plate mock-ups, the grooved plates are required; hence, the plates of size  $100x100 \text{ mm}^2$  were cut from as-received INRAFM steel plates of 16 mm thickness. Three grooves of size 10x20 mm with a rib thickness of 5.2 mm were made by CNC milling on a set of two plates



Figure 9. CIP+sintered high density silica cores.



Figure 10. HIPed channel plate made by use of dowel-pin and high density ceramic inserts concept.

(Fig. 8). Grinding operation was performed on the mating surfaces of the ribs to achieve the optimised surface finish of  $0.4 - 0.6 \ \mu m^{11-13}$ . Surface roughness measuring instrument of Talor Hobson, Model Talysurf Intra-II was used to measure the surface finish of the machined plates.

To prevent the channel collapse during HIPing, under optimized conditions of 1200-1240°C/120-140 MPa/ 4-6 hrs, non-reactive rigid inserts were needed; hence, extensive studies were carried out to establish such rigid inserts with rectangular cross-section. In this regard, a wide-range of materials were used to develop the inserts and the details of which have been reported elsewhere<sup>15</sup>. Among several approaches, the use of CIP+sintered silica cores with high density (> 90% of the theoretical density (TD) of silica) was found to be the best suited ones for HIPing of channel plates and bonding of the ribs effectively. The details of silica cores developed through CIP+sintering route have been described elsewhere<sup>15</sup>. A set of high density cores prepared by CIP+sintering route subjected to CNC milling to achieve the precise dimensions is shown in Fig. 9.

Square shape silica cores were inserted into the grooves of the channel plates and the two opposite sides of the plates parallel to the channels were TIG welded while the sides of the



Figure 11. Samples extracted from the HIPed channel plate mockup for preparation of micro-tensile specimens.



Figure 12. Micro-tensile samples fabricated from the HIP bonded IN RAFM steel plain mock-up (a) before testing (b) after testing.

plates perpendicular to channels were sealed with steel strips by TIG welding. Necessary provision was made onone of the steel strips for welding of a mild steel tube for evacuation and sealing under dynamic vacuum conditions. The channel plates having ceramic inserts were vacuum sealed and then subjected to HIPing as per optimised conditions mentioned earlier. The HIPed channel plate were machined to expose the silica core and then subjected to chemical leaching to dissolve the cores. A three channel plate mock-up prepared by use of CIP+sintered and then CNC machined silica cores as the inserts is shown in Fig. 10. The HIPed channel plates were heat treated as per the conditions stated earlier.

Round bars of 4 mm diameter were extracted from the rib portion of the HIPed channel plate (Fig. 11).

These samples were used for metallography and fabrication of micro-tensile test specimens as per the drawing prepared in accordance with ASTM standard (Fig.12).

Tensile tests were carried out at room temperature. The HIP-bonded plus standardheat treated specimens were tested at a cross-head speed of 1 mm per minute which results in a strain rate of 2 X  $10^{-3}$  s<sup>-1</sup> for the test specimens having the parallel length of 6.0 mm. After realizing the targeted properties in plain channel plate mock-ups, necessary studies have been carried out for fabrication of First-Wall of TBMs component in U/C- shape. Suitable concept was worked out and its sequence is presented in Fig. 13.

To implement this concept, the drawings were prepared by considering the dimensions of the as-received INRAFM steel plates of 25 mm thickness. These plates were subjected to hot bending to U/C-shape with the aid of specially designed and fabricated die-sets. Subsequently, to realize the channel structure on the bent steel plates, the grooves were made by CNC machining to realize the required dimensions and surface finish. For realizingthe channels in the scale-down version of



Figure 14. CIPed and sintered silica inserts after machining to net-shape.



Figure 13. Concept of for TBMs-FW mock-ups development by HIP route: (a) Milled INRAFM steel plate (b) Bending of milled INRAFM steel plate into C/U shape (c) Milling of grooves on the internal and external surfaces of the C/U sha plates (d) Assembly and HIPing of C/U shape grooved plates.

First-Wall of TBMs component, U-shape ceramic (silica) cores were prepared by CIPing followed by sintering as shown in Fig. 14.

Subsequently the near-net U-shape sintered cores were machined to net-shape by CNC milling/machining to easy fit into the grooves of the steel plate. The U-shape channel structures / First-wall was then welded and vacuum sealed as per the procedure described earlier and then subjected to HIPing under optimized conditions. The HIPed component was subjected to leaching to dissolve the ceramic inserts and realize the final component as shown in Fig. 15.



Figure 15. Sub-size U-shape channel component (FW).

## 3. RESULTS

### 3.1 Results on Development of Fused Silica Radomes

The powder composition and physical properties are very important to achieve the targeted properties in sintered material for radome application. The powder composition indicates the purity level of about 99.9 % and certain impurities such as  $Fe_2O_3$  and other combined alkalis which are well within the acceptable / tolerable limits. The powder had irregular shape particles with a wider size distribution between 0.1 and 150  $\mu$ m. These aspects are considered to be the important criteria for CIP compaction to realize the compacts with adequate handling strength and L/D ratio > 2. The powder had the tap density of about 45-50 % which decides the overall shrinkage during CIPing and based on that the dimensions of the patterns for rubber moulds and tooling for CIPing are worked out to realize near net shaped radomes.

The as-received silica powder was subjected to XRD to confirm the amorphous structure/state and the and the X-ray diffractograph of powder is presented in Fig. 16. The amorphous state of powder is highly required for processing of radomes from silica as it greatly influences the strength and EM properties of the sintered material.

If the powder has the crystalline content more than 5% to begin with, it will get transformed to crystalline state during sintering and that can cause cracking of material or



Figure 16. XRD pattern of as-received silica powder.

unacceptable EM properties. In the present case, the powder was found to show no major peaks related to crystobalite, hence found suitable for processing of radomes (Fig. 16). Filling of fine size powder into rubber moulds in dry condition is not advisable, hence, the powder was add mixed with acetone dissolved with camphor as the binder and then the powder in semi-wet condition was filled into CIP moulds. Secondly the binder contributes to an effective bonding among the individual particles, thus leading to sufficient green density/strength.

In order to understand the sintering behavior, the CIPed silica green compacts in cylindrical shape were subjected to sintering cycles by varying both temperature and soaking time. The influence of sintering parameters on the densification and transformation to crystalline phase in CIPed silica is presented in Fig. 3 (a) & (b). The extent of phase transformation in CIP + sintered silica was determined by XRD-analysis and the data are presented in Fig. 3(c). It is evident from the above data that sintering at a temperature less than 1225 °C/3 hrs retains amorphous structure, but the corresponding density is very low (75 % TD) as can be seen in Fig. 3(a). On the other hand, sintering at a temperature of 1400 °C/3 hrs results in about 80% density but formation of cristobalite occurs. Increase in soaking time upto 4 hours at lower temperature of 1200°C also results in amorphous structure but the corresponding density is only about 74.5 % of theoretical density (Fig. 3(b)). Further increase in sintering time at 1200 °C leads to formation of crystobalite that can cause cracks in compacted material. In order to overcome this problem, important powder characteristics, binders, sintering reagents as well as sintering parameters were established to realize the compacts of desired shape free from cracks and unacceptable defects<sup>7</sup>. The crack free silica compacts realized through optimized processing conditions are shown in Fig. 5.

The silica compacts processed under optimized conditions were tested for density, microstructure and XRD. The density was found to be 1.85-2.1 g/cc. The microstructure showed uniform distribution of coarse and fine silica grains and the presence of porosity (Fig. 6). The presence of porosity is desirable for EM performance particularly the dielectric constant (K) and improvement in strength at higher temperatures. The XRD pattern clearly shows amorphous state of sintered silica

Table 4.	Electromagnetic properties of CIP + sintered silica
	processed under optimized conditions

Property	Achieved
Dielectric constant	< 3.4
Tangent loss	< 0.008

 Table 5.
 Thermal properties of cold isostatically pressed and sintered silica

Property	Achieved			
	@ RT	5.5 X 10 <sup>-7</sup>		
CIE (om/om/V)	$RT - 320 \ ^\circ C$	1.6 X 10 <sup>-6</sup>		
(CIII/CIII/K)	$320-600\ ^{\circ}\mathrm{C}$	7.2 X 10 <sup>-7</sup>		
	@ RT	0.6		
Specific heat( J/g.K)	320 °C	1.0		
	600 °C	1.3		
	@ RT	0.49		
Thermal conductivity(W/m.K)	320 °C	0.69		
	600 °C	0.74		

	<b>Fable</b>	6.	Flexural	strength	of	CIPed	and	sintered	silica
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Test temp. (°C)	Flexural Strength (MPa)
RT	≥35
450	$\geq 50$

(Fig. 4) which is an important spect of the material for radome application.

The electro-magnetic (EM) properties such as dielectric constant– (K) and tangent loss- $(\delta)$  of the CIPed and sintered silica processed under optimized conditions were evaluated by using wave guide method in the frequency range of 12-18 GHz. The dielectric constant (K) and tangent loss ( $\delta$ ) of CIPed and sintered silica samples as listed in Table 4 are found suitable for radome application.

The thermal properties such as co-efficient of thermal expansion (CTE), thermal diffusivity and C<sub>p</sub> measured upto 600 °C are given in Table 5 are in agreement with the targeted requirement. The flexural strength of the silica material processed under optimized conditions (CIPed and sintered) was evaluated by using three point bend test. The data on flexural strength at room temperature (RT) and at elevated temperatures are given in Table 6. It can be seen that the strength of the material increases with increasing the temperature and this characteristic feature of silica is found to be beneficial for radome application.

It may be noted that the as-sintered radomes shall have extra material on the profile. Hence machining is needed on the radomes to obtain the desired dimensions and wall thickness. However, machining of sintered silica radomes is a highly challenging task and very important as far as radome performance is concerned. The critical parameters like bore sight error and insertion losses are of utmost important. These are influenced by the material properties, profile and surface finish of the radomes. It is observed that radome holding on the lathe machine, truing and balancing are the critical steps. Cracking of radomes and high rejection rates are normally experienced due to lack of appropriate facilities and technical knowledge in machining of thin-wall ceramic components of high L/D ratio. A lathe machine of bed-length: two times more than the height of the radomes is needed for internal machining. The radomes to final dimension through mechanical methods are subjected to non-destructive testing (NDT) by X-Ray radiography in accordance with ASTM E94 – 04 thereby ensuring that they are free from unacceptable defects. CMM profile measurement is also carried out on the machined radome before taking up performance evaluation/qualification testing.

In order to evaluate the EM performance, structural integrity/load bearing capacity, vibration resistance, thermalcycle and lightening resistance and other relevant functional capabilities, the machined radomes are attached to metallic bulkhead. The EM performance of full size radome is evaluated by using Ku-band ensuring that the radome has minimum insertion losses and bore sight error. The structural tests at different temperatures were conducted as per the standard procedures and based on the results it was found that the CIP+sintered radomes are acceptable for the intended applications.

#### 3.2 Development of First-Wall of TBMs

The chemical composition of as-received INRAFM steel plates is given in Table 3.

Apart from the desirable elements, the levels of impurities of elements are also presented in the table. During the course of present study, it is observed that diffusion bonding of INRAFM steel is a difficult task due to the presence of W and Ta with sluggish diffusion kinetics. Therefore, HIPing a very high temperature of 1200-1240 °C under high pressure of 120-140 MPa for more than 5 h of sustaining time was found to be essential to obtain the good bond strength (> 80% of the parent material). It was also observed that the surface finish of the mating surfaces of the steel plays an important role as the effective bonding could only be achieved with the surface finish in the range of 0.4-0.6 microns<sup>13</sup>. The second important aspect in the development of channel structure from INRAFM steel is the adaptation of highdensity non-reactive rigid ceramic cores. Without ceramic cores, the ribs bulged and collapse of channels occurred. Based on the encouraging results obtained in test coupon level bonded samples in terms of microstructure and tensile properties, the sub-size first wall fabrication was taken up and the results of it are present below.

In order to prepare the U-shape inner and outer segments the required INRAFM steel plates of 25 mm thickness were bent to realize two U-shape plates (Inner and outer plate). Subsequently, rectangular grooves were made on both inner and outer bent steel plates using vertical CNC machine. The concept of U-shape inner and outer steel plates with grooves are presented in Fig. 13. As the ceramic cores are essential to avoid the collapse of channels during HIP-processing, accordingly U-shape ceramic cores were developed by CIP forming technique where in ultra thin rubber moulds were employed (Fig. 14).

Uniform contact and alignment of ribs in two U-shape plates and assembly of U-shape coreswithin the grooves are the challenging tasks. This was accomplished by use of dowel-pins and specially fabricated tools and fixtures. After assembly of inner and outer segments of grooved plate as well as the cores, TIG welding was performed to ensure leak proof during HIP and a provision was made to evacuate the plates assembly and sealed under dynamic vacuum conditions. The welded TBMs component was then subjected to HIPing under optimized conditions. The HIPed sub-size U-shape first-wall of TBMs component is presented in Fig. 15. In order to carry out the cutup test and property evaluation, a three channel plate mockup was prepared and has been processed along with the sub-size first-wall of TBMs. One of the HIP three channel plate mockup is shown in Fig. 10. The test samples were extracted from the rib portion (Fig. 11) of the channel plate for microstructural characterisation and mechanical property evaluation.



Figure 17. Microstructure of HIP-bonded INRAFM steel by using silica inserts.

 
 Table 7.
 Tensile properties of HIP+heat treated INRAFM steel at room temperature

Properties	Plain steel	HIP bonded steel	Remarks
YS (MPa)	470±5	555±5	
UTS (MPa)	620±5	670±5	Fracture occurred
% EL.	20±2	20±2	away from the joint
% RA.	74±2	74±2	interface

Table 8. Tensile properties of HIP+heat treated INRAFM steel at 500 °C

Properties	Plain steel	HIP bonded steel	Remarks
YS (MPa)	380±5	440±5	
UTS (MPa)	425±2	475±5	Fracture occurred away
%EL.	15±2	20±2	from the jointinterface
% RA.	75±1	80±1	

 
 Table 9.
 Tensile properties of HIP+heat treated INRAFM steel at room temperature

Properties	Plain steel	HIP-bonded steel
YS (MPa)	520±5	510±5
UTS (MPa)	600±5	650±5
%EL.	22±2	40±2
% RA.	80±1	60±1
Specimen geometry	Gauge length : 20 mm Gauge diameter: 4 mm	Gauge length: 6.0 mm Gauge diameter: 2.3 mm

The microstructure of HIP-bonded INRAFM steel plate shown in Fig. 17 reveals the joint interface and considerable grain boundary migration across the joint interface can also be seen in the micrograph.

The samples extracted from rib portion were heat treated as per the standard schedule of 900 °C/2h/AC + 700°C/2/ AC. The microstructure of theheat treated material presented in Fig. 17 shows the joint interface is free from the undesirable phases but contains fine grains and tempered martensite. Round bars of 4.0 mm diameter test coupons were extracted from the rib portion (Fig. 11) and then the micro-tensile test specimens were fabricated from it as per the drawing prepared in accordance with ASTM standard. Tensile tests were carried out at room temperature on theHIP-bonded plus standard heat treated specimens (Fig. 12).

The tensile properties of HIP-bonded + heat treated samples (but joints) at RT and at 500 °C are presented in Table 7 and Table 8 respectively. For the purpose of comparison, the tensile properties of the plain steel with same processing history (HIP followed by standard heat treatment) are also included in the tables. It can be seen in the Table 7 and 8 that the YS and UTS of the HIP-bonded steel are comparable to those of the parent material both at RT and 500 °C. On the other hand, the tensile properties of the HIP- bonded + standard heat treated material extracted from the rib portion have revealed strength properties (YS and UTS) comparable to those of the parent material, the results of which are presented in Table 9. Also, the ductility (% EL of 41.5 and % RA of 60) for the HIP bonded steel are comparable to that of the plain steel. A comprehensive information on the microstructure and tensile data of HIP bonded plain channel mock-ups have been reported elsewhere14.

#### 4. **DISCUSSION**

## 4.1 Development of Fused Silica Radomes

The radar-dome (radome) is a protective dielectric housing for a microwave or millimeter- wave antenna. The functions of the radome are to protect the antenna from adverse environment in ground-based, airborne and aerospace applications while having insignificant effect on the electromagnetic performance of the antenna<sup>15</sup>. In order to fulfill the above demands, the radome is required to satisfy functional as well as structural requirements. The functional properties of the radome material directly influence the radarefficiency; consequently a radome material should be transparent to the working frequencies, stable low dielectric permissivity (K) and low loss tangent ( $\delta$ ) On the other hand, the structural properties of a radome material are closely related to the speed and aerodynamic loads encountered by the missile; hence, it should have light weight, high strength at ambient and at elevated temperatures, high elastic modulus (to avoid the buckling of thin wall radome), good thermal shock resistance to sustain high heating rates at the surface and all weather capability (rain erosion and particle impact resistance), etc.,

Depending on the functional and structural requirements of missiles including loads, temperature and flight duration etc., radomes of varied size and shape (hemispherical, cone, tangent Ogive, power law and hybrid complex profiles) are used. The radomes are generally fabricated from polymers and ceramic materials. Polymers like Kevlar and high density polyethylene were the most commonly used ones due to their low dielectric constant, low loss tangent combined with good fabricability and serviceability. However, application of the polymer based radomes is limited to applications wherein the surface temperature of about 150 °C. On the other hand, the radomes intended for high speed missiles are fabricated from high temperature ceramics such as fused silica, silicon nitride, Alumina and other composite materials with a proven capability to withstand higher skin temperatures experienced by the missiles during service.

Among the ceramics, fused silica  $(SiO_2)$  is found to be the most commonly used one for radome applications based on its attractive properties. Silica in its amorphous form exhibits low thermal expansion coefficient and low thermal conductivity. It has low dielectric constant, low loss tangent and offers exceptional thermal shock resistance. Silica is considered to be the ideal material for radome applications because most of its properties do not vary much with increase in temperature up to about 1000 °C. It is evidentfrom the literature that silica radomes are generally processed through slip casting followed by solidstate sintering route<sup>16</sup>. In slip casting, powder compaction to the required shape (green compaction) is achieved under atmospheric pressure<sup>17</sup>. This causes insufficient green strength and prevalence of non-uniformly distributed porosity in the sintered radomes. As a result, the radomes are prone to cracking either during machining to final profile (leading to high component rejection rate) or catastrophic failure during service.

Gel-casting is a variant of slip casting process, finding applications in making the silica radomes<sup>18</sup>. This technique relies on green compaction of powder mixture/slurry under higher than atmospheric pressure. It is known that this process involves hazardous chemicals as the binders (monomers) for powder compaction to the required shape. Due to lack of sufficient strength in as-cast material, handling of greenradomes is found to be the major problem encountered in this process and this finally leads to high rejection rate of component during the subsequent stages of processing like sintering and machining.

As stated above, silica is a prominent material for fabrication of radomes. However, powder compaction, sintering and machining to the final dimensions are the most challenging aspects of the development of radomes. Development of silica radome greatly relies on solid state sintering process to achieve the required physical, mechanical and electromagnetic properties. However, solid state sintering of fused silica requires very high temperatures (>1400 °C) and longer periods to achieve 80 % of its theoretical density (2.2 g/cc of SiO<sub>2</sub>). Such high temperature sintering leads to formation of Cristobalite, which is a crystalline phase, affect adversely the strength and electro-magnetic properties. On the other hand, sintering at lower temperatures for shorter duration though retains the amorphous structure of the silica but does not confer adequate density and correspondingly the required level of mechanical strength. Also, the presence of non- uniformly distributed residual-porosity which is inherent to the solid state sintered products causes a large scatter in the mechanical properties.

In order to overcome the problems associated with conventional processing of silica- radomes, Cold Isostatic Pressing (CIP) has been used for consolidation of silica powder for the development of radomes for missile application. In CIPing, the pressure is highly uniform due to its hydrostatic nature and absence of die-wall friction and hence, green density is much higher that usually achieved by slip-casting/ gel-casting. Since the initial green density is 65-70 % of T.D (2.2 g/cc of Silica) in CIPed material, it requires lower sintering temperature to achieve the required density level as compared to that for slip-cast material as shown in Fig. 3. Since the initial particle size is irregular, under atmospheric pressure compaction, the pores will have irregular shape in slip-cast and sintered material. This would lead tocracking of thin wall section of radomes during machining. In contrary to this, the pores though present in CIP+sintered material, will have round shape with more or less distributed uniformly as depicted in Fig. 6. Presence of such pores will act as the sinks for crack propagation and beneficial for good performance of radomes.

It is to note that optimization of sintering parameters has been accomplished through extensive sintering experiments on CIPed compacts by varying both sintering temperature and soaking time schedules. As can be seen in Fig. 3(a) that, the CIPed silica compacts sintered at 1225 °C for 3h have retained the amorphous structure but acquired the density of 75 % of theoretical density (T.D) of Silica (2.2 g/cc). It can also be seen in the figure that with increasing the sintering temperature, the density has increased and had attained about 79 % of T.D at a sintering temperature of 1400 °C for 3 h. This is still much lower than the targeted minimum density of 85% of T.D. Further, it can be seen in Fig. 3(c), that an increase in sintering temperature to 1230 °C has caused transformation of material into crystalline phase (cristobalite) with no much improvement in density. The presence of cristobalite causes cracking of material and leading to inferior strength as well as unacceptable EM properties required for radome application. In view of this it was resorted to investigate and understand the effect of soaking time on the CIPed silica compacts at a lower sintering temperature of 1200 °C. It was observed that sintering at 1200 °C for 5 h, initiation of devitrification occurs and the corresponding density is only about 75 % of T.D (Fig. 3(c)) and is not adequate to realize desired strength. Based on the above, it can be inferred that sintering at lower

temperature though retains the amorphous structure but the material do not get densified to the required level and hence required strength is not achieved. On the other hand, higher sintering temperature/ longer duration of soaking time though improve the density marginally, however, undesirable phase transformation is prevalent and that affects the EM and mechanical properties adversely. In view of this, instead of varying the sintering time and temperature, it was resorted to improve the powder properties and employ suitable sintering reagents to densify the material at lower temperature with no much scope for transformation of material into cristobalite or in other words keep the material in amorphous state to achieve good combination of EM and mechanical properties.

The CIP + Sintered material has exhibited a good combination of electromagnetic and mechanical strength properties (Tables 4 and 6). It is observed that the EM properties are closely related to material purity, amorphous structure and porosity levels. The dielectric constant and loss tangent are usually affected by the alkali content and crystalline phases of the material. On the other hand, the mechanical strength of the sintered silica depends on density and amorphous structure. It may be noted that high density material offers high strength but in the case of silica, high density can only offer high strength provided the amorphous structure is maintained. The CIP+sintered material processed under optimized conditions did not have major peaks conforming to cristobalite and had the density  $\geq 1.85$  g/cc and consequently the material had exhibited the flexural strength of  $\geq 35$  MPa and  $\geq 50$  MPa at room temperature and at 450°C respectively (Table-6).

Apart from the properties, achievement of desired dimensions and profile is an important aspect of radome development. In this regard, the shrinkage of powder mass is one of the important parameters that is to be considered during processing of full size radomes. As mentioned earlier that the CIPed radome exhibited shrinkage of 10-15 % during CIPing and 2-6 % during sintering under optimized temperature and soaking time. In order to achieve the targeted dimensions, these shrinkage allowances were considered and accordingly the CIP mandrels and rubber moulds were prepared. Secondly, it was also ensured that the sintered radome had sufficient positive material and that conforms to its near net shape which has been machined to achieve the net-shape and final dimensions.

The sintered full-size radomes did not have the cracking tendency during machining to final profile and has been attributed to the uniform density and consistent strength. It is evident from the unpublished work of Mangels et al. that one of the leading radome manufacturing companies elsewhere produces radome blanks from fused silica powder by adopting an advanced ceramic processing technique<sup>19</sup>. However, exact details of their processing technique have not been revealed. It is also stated that the fused silica blanks are subjected to sintering at very high temperature to obtain the desired properties. The sintered blanks are then diamond machined using the latest CNC, ID and OD grinders to produce finished radomes. The radomes are inspected using automated coordinate measuring machines (CMMs) to verify dimensional accuracy. The radomes are then calibrated using 'bore sighting' to ensure electrical behaviour of the radome. It can be visualized that machining

of radomes from sintered blanks may be a good option for realizing the dimensions as per the requirement but it becomes highly expensive and time consuming because of the technical constraints associated with the machining of high density silica compacts. The performance of full-size radomes processed through CIP+ sintering route was found to be acceptable in terms of EM performance (> 85 % transparency) and load bearing capabilities i.e. 240 % above the design strength at RT and 400 % above the design strength at 400 °C. Based on the excellent EM and mechanical strength, CIP+sintered full-size radomes are being manufactured on a large scale to meet the specific requirements.

#### 4.2 Development of First-Wall of TBMs

The First-Wall (FW) is one of the crucial components of test blanket modules (TBMs) intended for shielding and heat extraction from the fusion reactor. Fabrication of FW is highly challenging and multi-disciplinary task due to its large size and complex shape and moreover, the conventional welding is not acceptable for its fabrication, as it is a plasma facing structure. Solid state joining is the key technology that determines the mechanical properties of the FW of TBMs to ensure reliable performance in nuclear environment. Hot Isostatic Pressing (HIP) has the potential for joining of materials without destroying the original microstructure<sup>3,20</sup>. Therefore, HIP route was adopted for joining of plain and grooved plates of India Specific Reduced Activation Ferritic Martensitic (INRAFM) steel to achieve the required bond strength. The FW of TBMs shall be subjected to thermal and mechanical stresses during service, hence, it is utmost important to achieve the joints with strength comparable to that of the parent steel ( joints free from porosity and related defects to realize the joints with a minimum strength of about 80 % of the parent material). In order to accomplish this requirement, various processing parameters such as (i) Mean surface roughness of the steel plates to be joined, (ii) Plates alignment concept, (iii) Capsule welding and vacuum sealing, (iv) Hot Isostatic Pressing and (v) Heat treatment schedules have been established through sustained R&D efforts and the details have been reported elsewhere<sup>13,14</sup>.

HIPing is usually conducted at a temperature greater than 0.7  $T_m$  (where  $T_m$  is the melting temperature of the material in Kelvin). Secondly, the pressure of HIPing is selected in such a way that it will be very close to or higher than the yield strength of the material at HIPing temperature. On the other hand, the HIPing time is selected based on the diffusion characteristics of the material as well as the mass/volume of the material to be HIPed. However, the HIPing conditions can be varied depending upon the microstructure and mechanical property requirements accordingly.

As stated earlier, that in the present case, the HIPing cycles were carried out at different temperatures ranging from 980-1240°C under varied pressures between 100-140 MPa for 3-5 hrs of sustaining time. An elevated temperature aids interdiffusion and assists microplastic deformation<sup>4</sup>. Whereas, a high pressure aids deformation of surface asperities and to close up all pores. In addition, the pressure helps to breakup surface oxide films which tend to inhibit diffusion. The time at the sustained temperature should be appropriate both for

reasons of cost and also to avoid potential deleterious effects such as formation of brittle intermetallics, excessive grain growth and secondary recrystallization. As the INRAFM steel consists of W and Ta with sluggish diffusion kinetics, the bonded joints free from porosity and undesirable phases could only be achieved when the material was subjected to HIPing at 1200-1240 °C/120-140 MPa/ 4-6 h of sustaining time. Hence, these set of conditions have been considered as the optimised HIPing parameters.

The HIPing conditions optimised in this study ensured significant grain boundary mobility across the joint with adequate diffusion of various elements. In the as-HIPed condition, the grains were coarse and coarse martensite lathes were present in the steel (Fig. 8). However, the HIPed steel subjected to heat treatment showed the joints with fine size tempered martensite over the fine prior austenite grains (Fig. 18).



Figure 18. Microstructure of HIP-bonded INRAFM steel by using silica inserts subjected to heat treatment.

The strength of HIP bonded joints (but joints) after heat treatment was found to be better than that of the base material i.e. INRAFM steel at room temperature and at 500 °C (Tables 7 and 8). This could be due to the fact that in HIP bonded version, the steel is encapsulated and then subjected to vacuum degassing at elevated temperature and finally HIPed. Due to the encapsulation, the steel will be HIPed more effectively as compared to the steel with no encapsulation during HIPing.

Any smaller internal defect shall be totally eliminated during HIPing due to the encapsulation and evacuation. Secondly, the encapsulated steel does not come in contact with pressure transmitting medium (Ar gas) during HIPing which will have a small amount of oxygen (usually upto 2 vpm) and hence, the surface contamination/grain boundary oxidation/ embrittlement is avoided as compared to plain steel (with no encapsulation).

The marginal reduction in ductility with increase in testing temperature to 500  $^{\circ}$ C in HIP bonded steel is presumably due to slight oxidation of the steel during testing. On the other hand, the improvement in reduction of area at 500  $^{\circ}$ C could be due to the effect of additional tempering of steel that can take



Figure 19. Full size tensile tested samples before and after testing. The joint interface is away from the fractured region.

place at this temperature. Moreover, during the tensile testing of samples, fracture occurred away from the joint interface. Though this failure location is not very easily visible in case of micro tensile specimens but, when full-size tensile test specimens fabricated separately from HIP bonded material and tested, have revealed the failure location and the joint interface very clearly in support of this statement, the relevant photographs of the tested specimens showing joint interface and fractured location are given in Fig. 19 and these findings have been elaborated elsewhere<sup>10</sup>.

This substantiates that the joints are stronger than parent material. The higher strength of HIP bonded joints could be attributed to the observed ultrafine round shape oxide particles discretely distributed at the joint interface that can contribute to dispersion strengthening. The HIPing parameters such as temperature, pressure and sustaining time optimized in this study for joining of INRAFM steel are much higher than those reported for EUROFER steel<sup>20</sup>, but it is inevitable to have such a high temperature for the present steel to achieve the satisfactory bonding. During HIPing, an excessive grain growth occurred in the base material with which the average grain size of the steel was found to be 60  $\mu$ m. However, after heat treatment grain refinement has taken place and the average grain size was found to be 20  $\mu$ m and is responsible for improved and realization of acceptable properties.

The first-wall fabrication tried elsewhere envisages the use of two-step HIPing, single high pressure HIPing of rectangular tube methods<sup>21</sup>. It is understood from the data available in the literature that these processes are time consuming and involves a wide-variety of materials with different thickness. Although, the two-step process was tried initially in this study<sup>13</sup>, it is found that this technique is not suitable for joining of INRAFM steel; hence, it has been decided to adopt the high temperature / high pressure conditions. It may also be noted that the HIPing at high temperature and high pressure would lead to enhanced bond strength and consistency in properties. To prevent the channel collapse, thin wall tubes of circular or rectangular cross-section are used elsewhere<sup>22</sup>. However, it involves extensive welding and multiple weld joints and this ultimately would lead to leaking of capsules during HIPing. In view of this, non-reactive rigid ceramic cores were used in this study.

Since the required metallurgical properties could be obtained in HIP bonded plain INRAFM steel samples with butt joints, subsequently, efforts were put in for joining of 10x20 mm grooved plates with ribs of 5.2 mm thickness. HIPing cycles of different kind such as two- stage HIPing, HIPforming and single-stage HIPing were investigated to realize the 20x20 mm channels in U/C shape configuration<sup>10-12</sup>. Based on the results of these experiments, it was resorted to adopt the single-stage HIPing at high temperature under high pressure for longer sustaining time. To prevent the channel collapse and bulging of ribs, non- reactive and rigid high density ceramic cores of U-shape configuration which are chemically leachable have been developed by a special technique based on Cold Isostatic Pressing (CIP) and sintering processes (Fig. 14). The application of ceramic core as the insert material does not require total encapsulation of grooved plates to be joined by HIPing. The cores also immensely protect the internal surface of the channels from oxidation during post-HIP heat treatment. By use of such ceramic cores together with optimized HIPing parameters, a sub-size First-Wall of TBMs from INRAFM steel with well bonded ribs has been successfully developed as shown in Fig. 15.

The critical processing steps established to overcome the specific technical challenges during the development of HIP technology for First-Wall of TBMs include: (i) hot bending of 20mm thick INRAFM steel plates to U-shape configuration. This could be successfully achieved with the aid of specially designed die-set and adaptation of suitable temperature and strain rate parameters, (ii) Precision milling of grooves of size: 20 mm width x 10 mm height with rib thickness of 5.2 mm on internal and external surfaces of the U-shape segments conforming to a mean surface finish of 0.4-0.6 µm is another important aspect of TBMs development that could be successfully achieved by use of 5-AXIS vertical CNC milling machine and adopting innovative engineering techniques and (iii) Assembly and alignment of U-shape grooved segments of INRAFM steel with ceramic inserts is also a critical step and this could be accomplished by use of specially fabricated fixtures and clamps for foolproof welding of capsule.

## 5. CONCLUSIONS

## 5.1 CIP Technology for Fused Silica Radomes

- In view of the strategic nature and long-term application, an innovation driven R&D was carried out at DMRL and established an indigenous technology based on Cold Isostatic Pressing (CIP) and sintering route for manufacture of silica radomes of different configuration/ profiles for application in target seeking missiles
- The silica compacts realized under the CIPing pressure ranging from 150 200 MPa subjected to sintering at temperature ranging between 1100-1225 °C for 2-4 hrs of soaking time have exhibited acceptable physical, mechanical and electromagnetic properties suitable for radome applications.

• A large number of CIPed radomes integrated to different missiles have made the series of flight release tests highly successful. The CIP technology offers high yield of radomes with low cost and has been found to be fillip to the home-grown missile defence systems and self-reliance in seeker technology.

# 5.2 HIP Technology for First-Wall of TBMs

- In the present study, hot isostatic pressing route was adopted for diffusion bonding of INRAFM steel to realize butt and lap joints with the mechanical properties comparable to those of the parent material
- The crucial steps established in HIP technology include

   (a) mean surface roughness (0.4-0.6 µm) of the mating surfaces of the material to be bonded (b) bending of thick steel plates in U/C shape and milling of channels on the internal/external surfaces (c) alignment of grooved plates with thin wall-ribs (c) precision welding and vacuum sealing of capsules and (d) hot isostatic pressing of grooved plates at 1200-1240°C/120-140 MPa/ 4-6 h of sustaining time with non-reactive rigid leachable ceramic inserts. This is followed by processing of plain-channel plate mock-ups and then fabrication of sub-size First Wall.
- Indigenously developed sub-size First-Wall of TBMs conforming to U/C configuration with 20 x 20 mm square channels, with the strength and ductility comparable to that of parent material substantiates the significance of HIP technology for fabrication of fusion reactor systems with enhanced quality and reliability over the conventional fabrication methods.

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## ACKNOWLEDGEMENT

The authors would like to thank DRDO for providing required financial support for the development of CIP and HIP technologies for manufacture of fused silica radomes and First-Wall of TBMs for missiles and ITER applications respectively. The financial support of IGCAR, Kalpakkam for TBMs activity is also duly acknowledged. The financial support of IGCAR, Kalpakkam for TBMs activity is also duly acknowledged.

The support extended by Dr K. Bhanu Sankara Rao, Dr T. Jayakumar and Dr Shaju Albert from IGCAR and Shri E. Rajendrakumar, IPR, Ahmendabad in the development of TBMs is duly acknowledged. The authors gratefully acknowledge Dr G. Madhusudhan Reddy, OS and Director, DMRL for his encouragement to publish this paper. The support of Dr SV. Kamat, DS and Secretary, DD (R&D) and Chairman, DRDO and Dr. G. Malakondiah, DS and former CC R&D (HR & TM), is gratefully acknowledged. The encouragement extended by the former Directors of DMRL is duly acknowledged. The authors would like to thank Dr. TK Nandy, OS and AD, PMMC for his encouragement for this work. The technical support of members from Powder Processing Group in particular Shri P. John Sree, TO 'D' (Rtd.) and those from the other groups in DMRL warrants special thanks.

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Contribution in the current study: He had contributed to the experimentation, analysis, material characterisation and co-ordination of relevant activities for realization of components.