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Vacuum Brazing of Titanium/316L Stainless Steel Transition Joint for Application in Helium Vessel of Superconducting RF Cavities

P. Ganesh*, Abhay Kumar, S.C. Vishwakarma,Aniruudha Bose, Ram Kishor Gupta, B. Sindal, S. K. Rai, P. Chinna Rao, P. Ram Sankar, D. C. Nagpure, R. Kaul, G. Mundra and B. Singh
Raja Ramanna Centre for Advanced Technology, P.O.: CAT, Indore - 452 013
*Email: ganesh@rrcat.gov.in

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ABSTRACT

Superconducting radiofrequency (SRF) cavities would form important part of the proposed Indian Spallation Neutron Source and Accelerator Driven System. The high beta 650 MHz SRF cavity would be enclosed in a cylindrical vessel to hold liquid helium (LHe). Titanium (Ti) is being considered as the material of construction of helium vessel. The LHe inlet supply line and return helium gas pipe line of the helium vessel would be made of 316L stainless steel (SS). This requires a bi-metallic tubular transition joint between Ti and 316LSS, operating at LHe temperature. Vacuum brazing and explosive welding are potential processes for joining these two dissimilar metals, as fusion welding leads to extensive cracking.

In the present experimental study, vacuum brazing was performed to join Ti-pipe/316LSS flange with BVAg8 braze filler metal (BFM). Notable features of the process include use of (i) 5-10 μm nickel electro-plating as a diffusion barrier on SS part for preventing possible iron migration towards titanium while improving surface wettability for BFM and (ii) use of 304 SS plug, shrunk fit into Ti-pipe, for achieving dimensional and profile accuracy of Ti-pipe during machining while also controlling joint gap during brazing process. The brazed joint displayed uniform joint thickness and acceptable level of hermeticity (helium leak rate 2×10^{-10} mbar.lit/s) and also sustained six thermal cycles between 293 K and 77 K. Shear strength of Ti-SS brazed specimens, made in sandwich configuration, was found to be in the range of 50-60 MPa, with failure occurring at Ti/braze interface.

Keywords: Vacuum Brazing; Titanium, stainless steel 316L; BVAg8; Superconducting radiofrequency (SRF) cavities; intermetallic phases.

1.0 INTRODUCTION

Raja Ramanna Centre for Advanced Technology (RRCAT) is pursuing an extensive program on development of superconducting radiofrequency (SRF) technology for setting up Indian Spallation Neutron Source (ISNS) [1,2]. Superconducting radiofrequency cavities would form an important part of the proposed Indian Spallation Neutron Source and Accelerator Driven System. Major benefits offered by SRF cavities, made of niobium, include reduced power dissipation and possibility of operating with higher acceleration gradient. The high beta 650 MHz SRF cavity would be enclosed in a cylindrical vessel holding liquid helium (Lhe). Titanium (Ti) is being considered as the material of construction of helium vessel. The LHe inlet supply line and return helium gas pipe line of the helium vessel would be made of 316L stainless steel (SS). This requires a bi-metallic tubular transition joint between Ti and 316L SS, operating at Lhe temperature. Vacuum brazing and explosive welding are potential processes for joining these two dissimilar metals, as fusion welding leads to extensive cracking [3-5]. Brazing is one of the basic methods applied for joining titanium or its alloys with austenitic stainless steel in the industry including heat exchangers for chemical industry as well as subassemblies of nuclear reprocessing and accelerator component accessories [6]. In the case of brazing, the large mismatch in mean coefficients of thermal expansion (CTE) between Ti (8×10^{-6} µm/m.K) and 316L SS (16×10^{-6} µm/m.K), requires control of braze joint thickness at the brazing temperature through proper design and fabrication of fixtures for the given geometries of the components to be brazed. For its intended service, Ti-316L SS transition joints must not only be hermetically sealed (helium leak rate 2×10^{-10} mbar.lit/sec) but also strong enough to withstand multiple thermal cycles between room temperature (RT) and 2 K without any degradation in joint's hermeticity. Alexander E. Shapiroetal have reported an excellent review on braze joining of titanium with various other metallic alloys at temperatures below 800°C with silver-based and aluminum-based brazing filler metals [7]. It is reported that fast heating-cooling brazing cycle provides a significant gain in the strength of titanium joints brazed with low-melting silver filler metals [7]. Brazing temperature of conventional Ti-Cu-Ni, pure silver and Ti-Zr-Cu-Ni filler metals is usually above the β-transformation temperature of titanium base metals that adversely affects the mechanical properties of the base metal [7].

Primary factors to be considered in designing brazed assemblies are the flow of the brazing alloy and soundness of the joints, with reference to joint gap dimensions, uniformity of the joint clearance, method of feeding the brazing filler, and means of ensuring the correct positioning of the brazed components relative to each other. These factors have a significant impact in the braze joining in tube to flange configuration. Brazed joints made in controlled atmosphere or vacuum furnace exhibit monotonic increase in strength with reduction in joint thickness [8]. Therefore, the lowest possible joint thickness may be preferred to obtain highest strength. However, designs for achieving very thin joints may result in zero joint thickness due to (i) lack of concentricity between male and female parts in the pre-braze assembly and (ii) distortions arising as a result of release of residual stresses during heating. Zero capillary gap may also result in unbrazed areas in cases where wettability of one of the faying surfaces is poor. Very thin joints also suffer from loss of ductility due to restricted scope for plastic deformation, where some plasticity is required to accommodate the local strain due to differential thermal expansion of materials being brazed.

The present study on vacuum brazing of Grade-2 Ti tube to Stainless 316 L flange was carried out with the following objectives: (i) obtaining a brazed joint with desired capillary gap and its uniformity with a low melting brazing filler (BvAg8) by designing the components of pre-braze assembly from the machining stage, (ii) qualification of the brazed joint in terms of its hermiticity for its service in ultra-high vacuum (UHV) (helium leak rate $\leqslant 1 \times 10^{-10}$mbar.lit/s), (iii) ability to withstand thermal cycling between room temperature (RT) and liquid nitrogen temperature (LN_2) without any degradation in joint's hermeticity. The paper presents the details of brazing recipe including design of the fixtures, components, sequence of machining operations and pre-braze assembly for obtaining acceptable transition joint for application in SRF cavity.

2.0 EXPERIMENTAL PROCEDURES

2.1 Experimental details:

In the present experimental study, vacuum brazing Ti pipe/316L SS flange was performed with BVAg8 (72% Ag and 28% Cu) braze filler metal (BFM). Details of dimensions of various components and pre-braze assembly are presented in **Fig. 1.** This joint configuration of Ti tube to SS 316L flange is a specific requirement for the SCRF cavity for inlet and return line

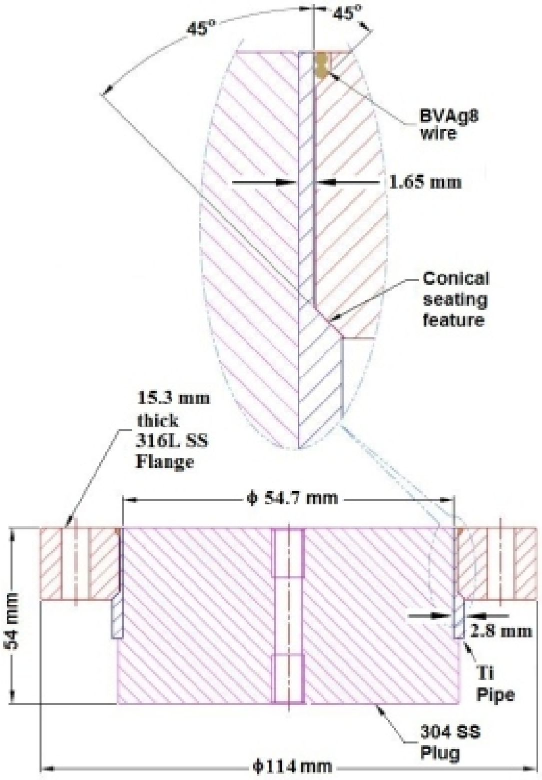

Fig. 1 : Schematic of the pre-braze assembly

of liquid helium. Two sets of joints were brazed under identical conditions for the sake of reproducibility. For evaluation of shear strength of the braze joint, 4 sets of Ti and SS specimens (arranged in the sandwich configuration with BVAg8 foil kept between Ti and SS specimens) were brazed along with the actual job. Notable features of the brazing process include use of (i) 5^{-10} μm thick nickel plating as a diffusion barrier on the faying surface of SS part for preventing possible iron migration towards titanium while improving surface wettability of SS for BVAg8 BFM and (ii) use of 304 SS plug, shrunk fit into Ti pipe, for achieving dimensional and profile accuracy of Ti pipe during machining while also controlling joint gap during brazing process. Vacuum brazing has been carried out in an indigenously built hydrocarbon-free vacuum furnace with chamber operating at a pressure of 2 X 10^{-5} mbar. The job temperature was measured by inserting two Inconelsheathed N-type job thermocouples inserted in thermos-wells provided in the sacrificial SS plug of the pipe-flange assembly. The thermal cycle used was heating from room temperature (RT)-523 K at 3 K/min, soaking at 523 K for 10 min; 523–823 K at 2 K/min, soaking at 823 K for 10 min; 823–1033 K at 3 K/min, soaking at 1033 K for 14 min; 1033–1073 K at 5 K/min, soaking at 1073 K for 1 min; followed by to RT. At the brazing temperature, the partial pressures of oxygen and water vapour were recorded as 2 x 10^{-9}mbar and 4 x 10^{-7} mbar, respectively. After brazing, the sacrificial SS plug was machined out before the brazed assembly was subjected to further characterization and testing.

The brazed jobs were characterized with respect to (i) microstructure and compositional details of the brazed joint, (ii) strength and fracture mode in shear tests, (iii) hermeticity and (iv) their capability to withstand thermal cycling between RT and LN_2 temperature (77 K). The characterization techniques employed for microstructural and compositional analysis included optical microscopy, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray florescence spectroscopy (XRF) and X-raydiffraction with CuKα characteristic radiation (λ= 1.54 ºA). The brazements were subjected to thermal cycling between RT and 77K. After each thermal cycle, the brazed joint's integrity was determined by subjecting it to Helium leak test (HLT). Thermal cycling test between 300 K and 77 K (in place of 2K) gives a preliminary validation of reliability of Ti-316L SS brazed joints against service-induced low cycle fatigue conditions. **Fig. 2** presents the Ti pipe/316L SS vacuum brazed component after removal of the 304 SS plug which is present inside Ti tube up to brazing cycle. The details of various procedures adopted and measures taken to obtain sound braze with uniformity of joint thickness in the entire circumference and length of the joint are described below.

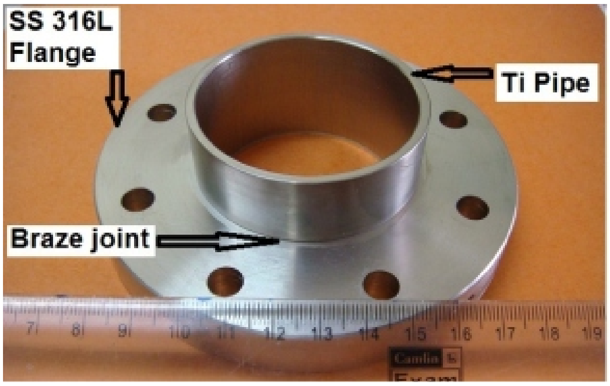

Fig. 2 : Vacuum brazed Ti-pipe/SS316L flange

2.2 Handling out of roundness errors in titanium pipe machining, during assembly stage

One of the major problems encountered in machining of Ti pipe with a wall thickness of about 3 mm was unacceptably high “out of roundness” errors (cylindricity and circularity) in the machined pipes. Based on the previous experience in developing the Niobium pipe to SS flange braze joint [9], the Ti pipe in the present study is machined precisely only on the internal surface on a lathe machine to remove the surface irregularities and an accurately machined SS304 plug is inserted into the Ti pipe after soaking it in liquid nitrogen with an interference fit. After this, the pipe-plug assembly is machined on the faying surface (OD) of the Ti pipe. The circularity and the cylindricity on the faying surface of the Ti pipe can now be controlled within 10 μm with the above process. The same plug is retained in place for controlling the capillary gap during subsequent brazing stage.

2.2.1: Stress relieving of SS parts to avoid undesired dimensional changes during brazing

The mill practice after solution annealing of austenitic SS involves quenching of the material to safeguard against carbide precipitation. This practice introduces residual stresses which may be released during the brazing cycle, and causes unwanted distortions. Therefore, to control uniformity of joint thickness within a few micrometers, this issue was addressed by solution annealing of rough machined SS plug and SS flange at 1353 K for two hours followed by cooling in the vacuum furnace. The solution annealed SS plug and flange were subsequently subjected to final machining to obtain specified dimensions and geometrical tolerances. The introduction of solution annealing step after rough machining could prevent unwanted distortions during brazing.

2.2.2: The flange material is 316L SS whose coefficient of thermal expansion (CTE) is double than that of Ti (8 x 10^{-6} µm/m.K). This leads to a significant increase in the radial gap between SS Flange and Ti pipe at the brazing temperature. Increase in the radial gap can be brought down if the plug material has slightly higher mean CTE with respect to the flange material. 304 SS has a mean CTE of 19.5 µm/m.K (in 293K - 1073 K) which is slightly higher than that of 316 SS (19.0 µm/m.K). During brazing cycle, the 304SS plug kept inside the Ti pipe with an interference fit expands and makes the Ti pipe to yield and keeps the radial gap close to the desired value (refer **Fig. 1** for pre-braze assembly). Hence, 304 SS has been chosen as a material of sacrificial plug for better control of capillary gap at the brazing temperature.

2.3 Self-centering of niobium pipe in SS flange

The concentricity of male and female part is essential in achieving uniform joint thickness throughout the circumference. A simple way to achieve self-centering is to provide a conical seating of the flange on the pipe by providing matching conical surfaces. This feature has self-centering properties which does not permit eccentric placement of SS flange over the Ti pipe during pre-braze assembly or during the placement of the pre-braze assembly in the furnace. The vertex angle of 90° has been provided at the conical feature at the bottom end as well at the BFM wire groove at the top end. This feature not only provides conical seating for self-centering but also limits the excessive build-up of nickel during the electroplating process.

3.0 RESULTS AND DISCUSSIONS

Metallographic examination of transverse cross-section of vacuum brazed job, made between Ti pipe and nickel-plated 316L SS flange, revealed sound brazed joint. The joint exhibited a distinct layer (about 10-12 µm) at Ti/braze interface. The braze joint width was measured around the circumference and along full depth at four different locations in each quarter. The width of the braze joint was in the range of 50-60 µm throughout the joint length of 15 mm. **Fig. 3** presents the microstructure of the cross-section at various locations and the uniformity of the joint thickness along the length of the joint. The joint displayed microstructure, with Ag-rich bright and Cu-rich dark phases and a continuous layer of intermetallic at Ti/braze interface as shown in **Fig. 4**.

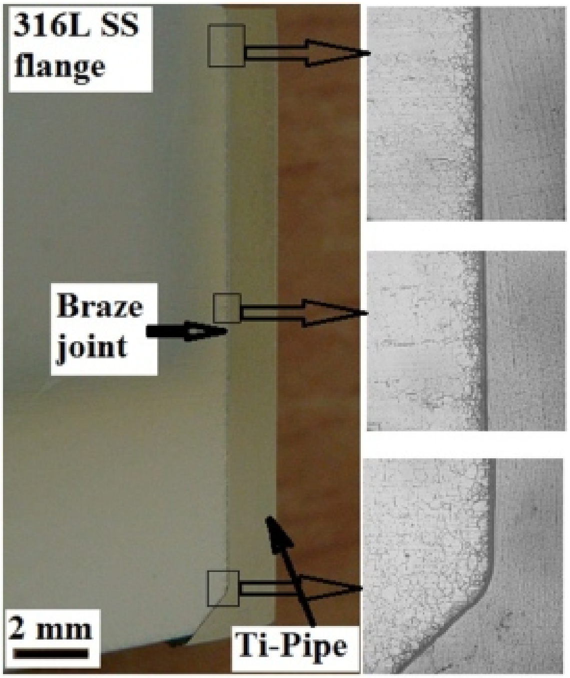

Fig. 3 : Macro and microstructure of the cross-section at various locations of braze joint

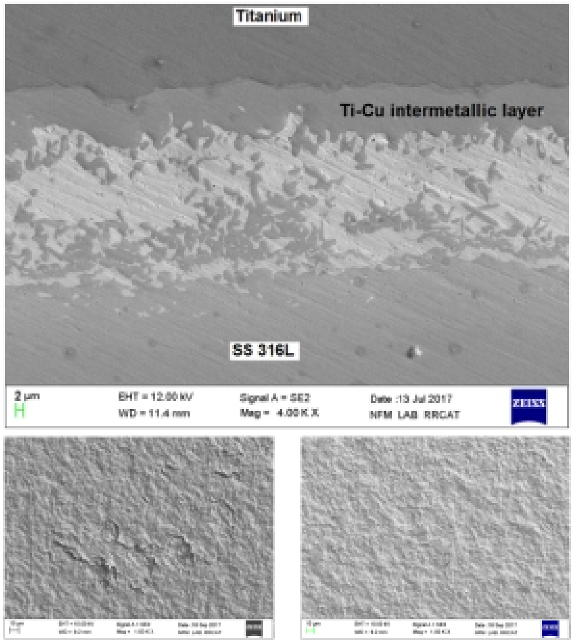

Fig. 4 : Top: Microstructure of the braze joint with intermetallic layer at Ti-braze interface. Bottom : Fractographs after shear testing.

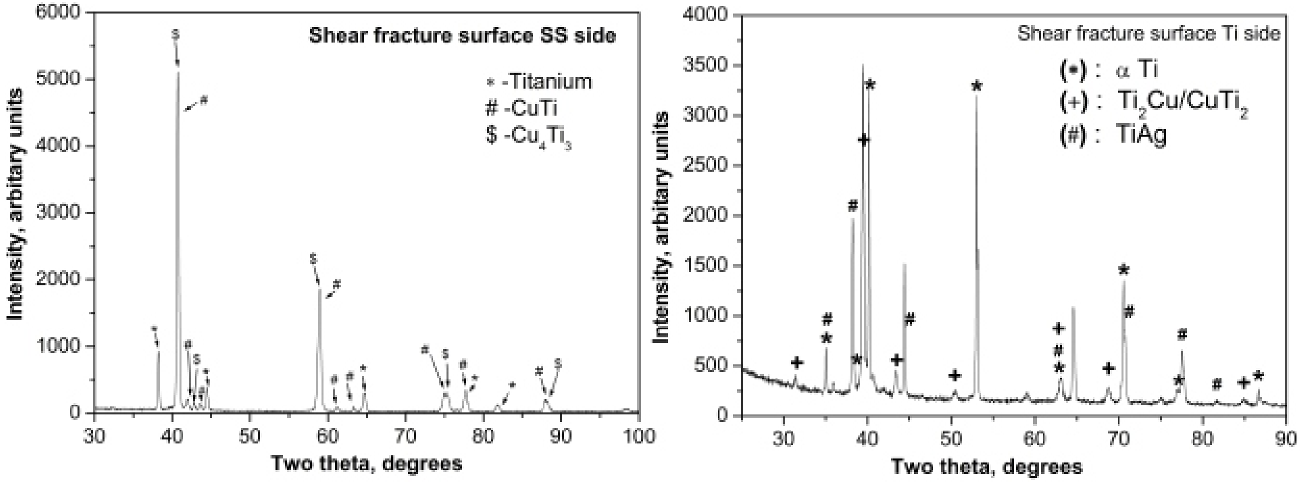

Fig.5 : X-ray diffraction plots of shear fracture surfaces of BVAg8 brazed Ti-316L SS specimen.

Shear testing of four numbers of Ti-SS specimens brazed in sandwich configuration and also four specimens extracted from the brazed job with capillary based brazed filling exhibited joint strength of 40 and 50 MPa, with site of failure again falling in the braze metal and Ti interface. Detailed fractographic examination of sheared fracture surface revealed presence of feature less flat regions representing brittle mode of fracture. **Fig. 4** presents fractographs showing fracture surface of shear tested brazed Ti-316L SS specimen. X-raydiffraction of sheared fracture surfaces of Ti/316L SS brazed specimens exhibited presence of Ti and Cu-Ti, and Ti-Ag based intermetallic phases. **Fig. 5** presents X-ray diffraction plot of sheared fracture surface of Ti- 316L SS specimen.

The average chemical composition (in wt%) of the shear fracture surface of SS and Ti side as analyzed by XRF spectrometer/probe (Olympus make) are presented in **Table 1**. The distribution of the elements and various phases on the cross-section of the braze joint as analyzed by SIMS (Secondary Ion Mass Spectroscope) are presented in **Fig. 6**. The brazed joint cross-section displayed noticeable enrichment of Ti-Cu phase adjacent to Ti-side and a Nickel rich layer on the SS side. **Fig. 6** presents the representative micrograph and associated EDS compositional mapping of Ag, Fe, Ti, Cu, Ni across braze joint.

The flat featureless fracture surface (**Ref. Fig. 4**) of the shear tested specimens and the presence of continuous layer of Ti-Cu phase adjacent to Ti-side as seen in the SIMS EDS elemental mapping (**Ref. Fig. 5**), indicate that the failure of the specimen took place at the Ti/Ti-Cu intermetallic layer of the braze joint. The average chemical composition of the shear

Table 1 : Chemical composition of the shear fracture surface obtained by XRF spectroscopy

Location of measurement/Element	**Titanium (Wt.%)**	**Copper (Wt.%)**	**Silver (Wt.%)**	**Nickel (Wt.%)**
SS Side fracture surface	37.47	33.52	28.55	0.35/0.56
	38.1	33.11	28	1.68
	36.12	31.47	25.52	1.58
Ti Side fracture surface	99.53	0.52	-	
	96.2/96.86	2.34	-	0.11/0.59
	86	10.1		2.24

fracture surfaces on Ti side and SS side as analyzed by XRF probe also strongly compliment the above inference that the failure was at the interface of the Ti/braze intermetallic layer (**Ref. Table 1**). Braze joining of titanium and its alloys with stainless steel with various Ag, Cu Sn, Al, Ni alloys was reported by Winiowsk [10]. A continuous layer of Ti-Cu intermetallic was invariably present in all the cases. In the present study, shear strength of Ti-SS brazed specimen, made in sandwich configuration, was found to be in the range of 50 - 60 MPa, with failure occurring at Ti/braze interface. Although, open literature [7, 10] reports shear strength values in the range of 140-160 MPa, the braze joint involved was between Ti-6Al-4V and the filler metals Ag Cu Al Sn related materials. The reported shear strength between Ti (CP Titanium) and stainless steel with BVAg8 filler by Shapiro [7] was 47 MPa which is close to the result obtained in the present study. Uniformity of joint thickness and ultra high vacuum (UHV) compatibility are not addressed in previous studies. In the present study the brazed assembly displayed acceptable level of hermeticity (helium leak rate 2 x 10^{-10}mbar.lit/sec) and also successfully sustained six thermal cycles between room temperature and liquid nitrogen temperature (77 K). It should be noted that selection of test temperature of 77 K (in place of 2 K) was based on the fact that most of the materials' contraction occurs between300 to 77 K and hence the test provides validation of reliability of Ti/316L SS transition joints against service-induced low cycle fatigue conditions.

4.0 CONCLUSIONS

The results of the present investigation have demonstrated that vacuum brazing of titanium pipe to nickel-plated 316LSS flange with BVAg8 filler resulted in sound transition joint with satisfactory shear strength of about 50-60 MPa and uniform joint thickness of about 50 +/- 10 µm. Vacuum brazed component displayed helium leak tightness better than 5 x 10-10mbar.lit/s and also withstood six numbers of thermal cycles between 300 K and 77 K without any degradation in joint's hermeticity; thereby establishing its suitability to withstand service-induced low cyclefatigue conditions.

Important contributing factors for establishment of sound titanium-316L SS transition joint with uniform joint thickness were (i) use of BVAg8 BFM with low brazing temperature of 1073 K which is below Beta trans temperature of Titanium (ii)

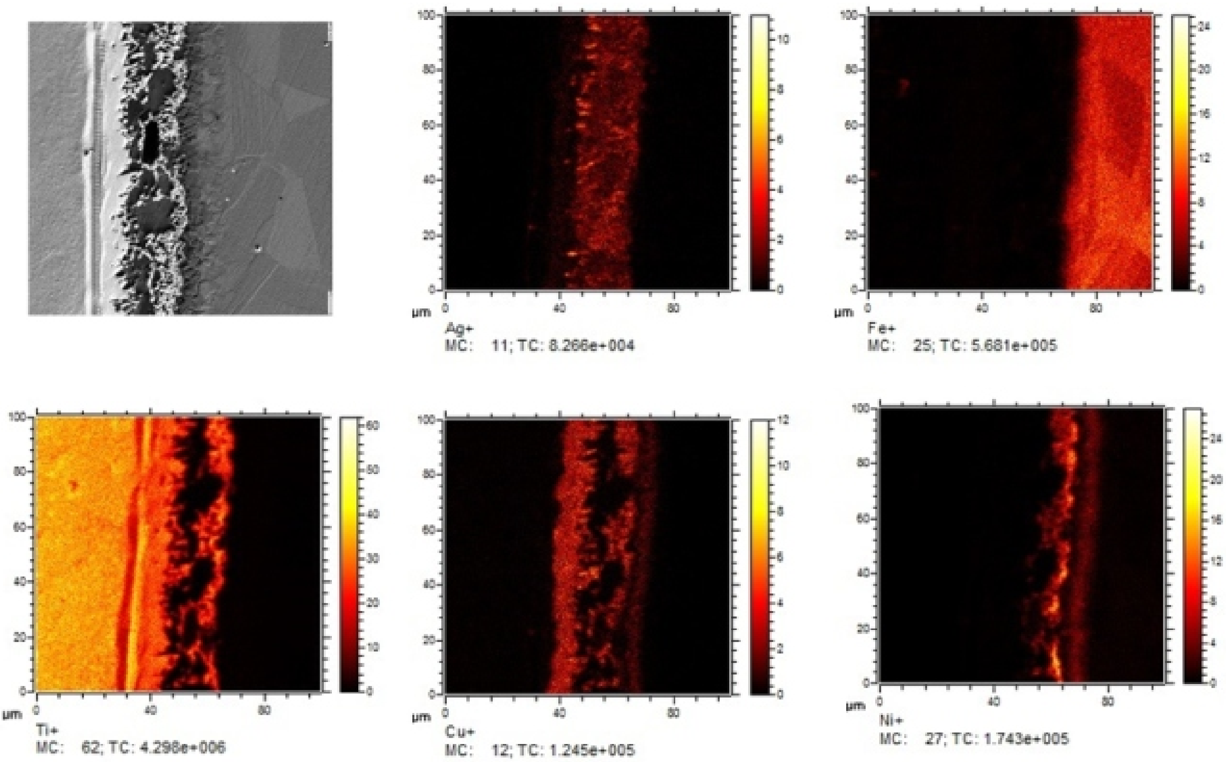

Fig. 6 : Micrograph of braze cross-section and associated EDS compositional mapping of Ag, Fe, Ti, Cu and Ni across braze joint.

use of Ni-platingon SS part for improved wetting and as adiffusion barrier to suppress iron migration towards braze metal(iii) Use of SS 304 plug with interference fit inside the Ti tube at the machining stage to precisely machine the Ti-tube with desired circularity and cylindricity, stress reliving heat treatment to prevent distortion (iv) conical seating arrangement to ensure uniform joint thickness and (v) retaining the SS 304 plug inside the Ti tube till end of brazing to control the joint thickness at the brazing temperature.

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