A study on brazing of Glidcop® to OFE Cu for application in Photon Absorbers of Indus-2

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Abstract: The paper describes an experimental study aimed at standardizing brazing procedure for joining Glidcop to OFE Cu for its application in upgraded photon absorbers of 2.5 GeV synchrotron radiation source, Indus-2. Two different brazing routes, involving brazing with silver base (BVAg-8) and gold base (50Au/50Cu) alloys, were studied to join Glidcop to OFE Cu. Brazing with both alloys yielded helium leak tight and bakeable joints with acceptable shear strengths.

1. Introduction
Indus-2, a 2.5 GeV synchrotron radiation source emits 187 kW of SR power from bending magnet at 300 mA [1]. Only 15% is channelled in beam lines while the rest is absorbed by 64 water-cooled photon absorbers [2-4]. In the upgraded design of photon absorber, the bottom part facing SR is made of low oxygen (LOX) grade Glidcop AL-15 while the intermediate part is made of OFE Cu which is to be joined to the bottom part. The dissimilar joint between Glidcop and OFE Cu is required to be leak tight (helium leak rate < 2 x 10^-10 mbar.lit/s), bakeable upto 150°C and reasonably strong to withstand high pressure cooling water. The present study was undertaken to standardize brazing process to obtain helium leak tight, bakeable and strong brazed joint between Glidcop® to OFE Cu.

Glidcop represents an oxide dispersion strengthened (ODS) copper with uniform dispersion of sub-micron size Al₂O₃ particles in a fine-grained high purity copper matrix. Dispersion of hard and thermally stable Al₂O₃ particles, with low solubility in copper, provides effective barrier to the motion of dislocations and grain boundaries, thus preventing re-crystallization of the matrix [5-8]. The material offers a unique combination of high strength, high electrical and thermal conductivities, coupled with an ability to resist softening and retain a large portion of these properties even after exposure to elevated temperatures [9].

Fine grained of Glidcop is a disadvantage in the brazing operation as large number of grain boundaries

![Figure 1. Details of specimen used for brazing experiments. The figure also provides schematic illustration of shear testing of the brazed joint.](image)
permit rapid diffusion of constituents of the braze alloy, particularly Ag, along the grain boundaries [9-11]. The effect would leave the joint with less than adequate amount of braze alloy. The problem is addressed by electroplating Glidcop® part with Ni or Cu (particularly for Ag-bearing brazing alloy) prior to brazing. The plated layer acts as a barrier to the diffusion of Ag into Glidcop [10].

2. Experimental Procedure

The study involved brazing of Glidcop ring to OFE Cu button, as shown in figure 1. The geometry of specimen facilitated leak and shear testing on the same specimen. The study was carried out in two sets viz. (i) Set 1: vacuum brazing with BVAg-8 alloy and (ii) Set2: brazing with 50 Au/50 Cu alloy in hydrogen furnace. Brazing with BVAg-8 alloy was performed in an in-house developed vacuum furnace [12]. For brazing with BVAg-8 alloy, Glidcop ring of the assembly (figure 1) was electroplated with three different types of coatings viz. Ni, Cu and Ni + Cu before loading the assembly in the furnace. Different electroplating routes were evaluated before brazing with BVAg-8 alloy. The methodology adopted for evaluating electroplating process involved metallographic examination of electroplated Glidcop specimens (10 mm x 10 mm x 3 mm) in as-plated and as-fired (at brazing temperature of 830°C for 3 mins.) conditions. The procedure adopted for electroplating Glidcop ring involved (i) preliminary cleaning, (ii) cleaning, (iii) activation and (iv) plating in the electrolytic bath. Four different cleaning treatments (step ii) viz. “A”, “B”, “C” and “D” were evaluated for Glidcop rings. On the other hand, different routes studied for electroplating Glidcop part were: (i) Ni electroplating in Watts’ bath, (ii) Cu strike in cyanide bath followed by thick Ni plating in Watts’ bath, (iii) Cu plating in cyanide/acid baths, (iv) Ni strike in Watts’ bath followed by Cu plating in acid/cyanide baths. Table 1 presents experimental electroplating parameters.

The electro-plated Glidcop ring was subjected to vacuum firing at brazing temperature before its brazing with mating OFE Cu part with BVAg-8 alloy in vacuum furnace. On the other hand, no coating was provided on Glidcop part while brazing with 50Au/50Cu alloy in hydrogen furnace. Figure 2 presents thermal cycles used for brazing Glidcop/OFE Cu assemblies with BVAg-8 and 50Au/50Cu alloys. Brazed specimens were subsequently characterized by helium leak test (HLT), thermal cycling test to evaluate its bakeability, shear test and metallographic examination under optical and scanning electron microscope with energy dispersive spectroscopy (EDS). Thermal cycling test involved subjecting the brazed specimen to multiple thermal cycles. Each cycle involved heating to 150 °C, soaking for 8 hours, followed by cooling to room temperature. After each thermal cycle, the brazed specimen was subjected to HLT to determine hermeticity of the brazed joint.

Figure 2. Thermal cycles used for brazing with BVAg-8 and 50Au/50Cu alloys.

Figure 3. Cross-sections of Ni-plated Glidcop® specimen in (i) as-plated and (ii) as fired (at 830°C) conditions. Note blister formation at Ni/Glidcop interface after firing.

Figure 4. A row of gas porosities on the cross-section of Cu strike + Ni-plated Glidcop specimen after firing at 830 °C.
Table 1. Details of different steps involved in electroplating of Glidcop specimens

| Step 1 - Preliminary cleaning: |
|-------------------------------|
| (i) Ultrasonic cleaning in trichloroethylene for 15 mins. |
| (ii) Immersion in 50% v/v HCl (37%) for 2 mins. + water rinsing |
| (iii) Immersion in a soln. of chromic acid (50 gm/l) & H₂SO₄ (5 ml/l) for 2 mins. + water rinsing |
| (iv) Immersion in 50% v/v HCl for 2 mins. + water rinsing |

| Step 2 – Cleaning: |
|---------------------|
| A: 1. Hot immersion cleaning in 10% w/v NaOH for 10 mins. |
| 2. Cleaning in conc. HCl (37% w/w) for 40 sec. |
| B: 1. A1; 2. A2; 3. Bright dip in a soln. of conc. HNO₃ (1 part) & conc. H₂SO₄ (2 parts) for 1 min. |
| C: 1. A1; 2. B3 |
| D: 1. A1; |
| 2. Dip in a soln. of 75% HNO₃ (69% w/w) & 25% ortho-H₃PO₄ (85% w/w) by volume for 20 sec at room temperature |
| 3. Bright dip in a soln. of 55% ortho-H₃PO₄ (85% w/w), 20% HNO₃ (69% w/w) & 25% CH₃COOH by volume for 2 mins. |

| Step 3 - Activation: |
|----------------------|
| Immersion in 10% v/v H₂SO₄ for about 30 sec. |

| Step 4 – Electroplating: |
|--------------------------|
| 1. Nickel plating: Watts’ bath - NiSO₄·7H₂O (250 g/l) + NiCl₂·6H₂O (65 g/l) + Boric acid (45 g/l) |
| pH: 3.8 - 4.2; Temperature: 55 - 65 °C; Current density: 3 A/dm² |
| 2. Copper plating: |
| 2.1 Cyanide Copper bath - NaCN (48 g/l) + CuCN (40 g/l) + Na₂CO₃ (30 g/l) + Rochelle salt (60 g/l) |
| pH: 10 - 10.5; Temperature: 55 - 55 °C; Current density: 2.4 A/dm² (2 mins.) + 1.2 A/dm² (10 mins.) |
| 2.2 Acid Copper bath - CuSO₄·5H₂O (200 g/l) + H₂SO₄ (38 ml/l) + chloride (30 - 40 mg/l) |
| Temperature: room temperature; Current density: 2 A/dm² |

Plating was performed with periodic reversal - forward cycle: 20 sec.; reversed cycle: 4 sec.

3. Results and Discussion

3.1 Set 1: Vacuum brazing with BVAg-8 brazing alloy

3.1.1 Evaluation of electro-plated coatings through metallographic examination

Metallographic examination of Ni and Cu plated Glidcop specimens, prepared under different conditions, exhibited dense coatings with sound bonding with underlying substrate. All four cleaning procedures were found to be effective for successful electroplating. However, it was noticed that

Figure 5. Cross-section of as-fired Cu-plated (cyanide bath) Glidcop specimen.

Figure 6. Cross-section of Ni-strike + Cu plated (acid bath) Glidcop specimen after firing at 830°C.

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nickel plated specimens, with or without Cu-strike, when fired at 830°C, developed blisters/gas porosities at plating/substrate interface, as shown in figures 3 and 4. It is believed that these blisters are formed due to hydrogen charging during electroplating [13-15]. During firing at brazing temperature, atomic hydrogen dissolved in the electroplated deposits, diffuse to substrate/plating interface to form molecular hydrogen and resultant gas pressure led to the formation of blisters [15]. On the other hand, in Cu-plated Glidcop specimens, involving (i) plating in cyanide bath and (ii) Ni strike followed by Cu plating in acid bath, no porosities/blisters were seen in as fired condition (figures 5 and 6).

3.1.2 Evaluation of brazed specimens

The specimens were prepared by vacuum brazing OFE Cu button with (i) Ni-plated and (ii) Cu-plated (Ni-strike + Cu-plating in acid /cyanide bath) Glidcop® rings, (refer figure 1). Vacuum brazed Ni-plated Glidcop /OFE Cu specimens exhibited helium leak rate of $10^{-4}$ - $10^{-5}$ mbar.l/sec. Metallographic examination of one of these specimens showed regions of decohesion at Ni plating/Glidcop interface, as shown in figure 7. The specimen exhibited intergranular penetration of brazing alloy into OFE Cu (refer figure 8) as well as into Glidcop. About 200 µm thick region of inter - granular penetration of brazing alloy into Glidcop is identified as “X” in figure 8. The figure also presents EDS concentration profiles of Ag, Cu and Ni across the brazed joint. Frequent peaks in Ag concentration profile represent Ag-rich grain boundary regions (in region “X”) in Glidcop. Shear testing of two brazed specimens exhibited failure strength of 135-151 MPa. Cross-sectional metallographic examination of the shear tested specimens confirmed that the site of shear fracture was Glidcop/Ni plating interface, as shown in figure 9. On the other hand, vacuum brazed Cu-plated Glidcop (Ni strike + Cu plated)/OFE Cu specimens exhibited leak tightness better than $2 \times 10^{-10}$ mbar.lit/s. The brazements remained leak tight even after undergoing 6 thermal cycles and thus established their bakeability for UHV application. Vacuum brazed specimen exhibited a sound brazed joint with about 30 µm thick layer of electro-plated Cu on Glidcop part (figure 10). The brazed specimens displayed shear strength of 136-144 MPa. Joint shear strength remained unaltered even after thermal cycling at 150°C. Cross-sectional metallography of the shear tested brazed specimen exhibited fracture at Cu-plating/Glidcop interface, as shown in figure 11.
3.2 Set2: Brazing with 50 Au/50 Cu alloy in hydrogen furnace

Visual examination of the brazed specimen exhibited brazing alloy flowing down to the bottom of the specimen, indicating full penetration of the brazing alloy across the length of the joint. Helium leak testing of the brazed specimens exhibited a leak rate < 2 x 10^{-10} mbar.l/s. Leak tightness of the brazed joint remained intact after undergoing 6 numbers of thermal cycles at 150°C. This ensured bakeability of the brazed joint for UHV application. Metallographic examination of the cross-section of brazed specimen exhibited full penetration of the brazing alloy, as shown in figure 12. Due to brazing at a temperature close to melting temperature of copper, OFE Cu part of the brazed assembly exhibited significant grain coarsening while no such effect was noticed in Glidcop ring. The brazed joint, although associated with isolated pinhole porosities, exhibited good bonding with both Glidcop and OFE Cu parts, as shown in figure 13. No inter-granular penetration of braze alloy was observed in the two substrates. Final composition (in wt%) of brazing alloy (as determined by EDS) - Cu: 79.3/84.4; Au:20.8/15.3, is indicative of extensive dilution of brazing alloy by the two substrates. Figure 14 presents concentration profiles of Cu and Au across the brazed joint. Shear strength of the brazed joint was found to be 163 MPa and fracture of the specimen took place in the brazing alloy (figure 15).
indicating that the two associated interfaces (Glidcop/brazing alloy & OFE Cu/brazing alloy) were stronger than the brazing alloy.

4. Conclusions
In the light of the results of this investigation, two brazing routes were standardized with BVAg-8 and 50Au/50Cu alloys for obtaining leak tight, bakeable and strong brazed joint between OFE Cu and Glidcop. Due to blister formation in Ni-plated Glidcop part during its exposure to brazing temperature, Cu plating of Glidcop was preferred for brazing with BVAg-8 alloy. Standardized brazing procedure for brazing with BVAg-8 alloy involved (i) electroplating Glidcop part - Ni strike in Watts’ bath followed by Cu plating in acid bath, (ii) pre-brazing firing of electroplated Glidcop part in vacuum furnace at 830 °C and (iii) vacuum brazing of Cu-plated Glidcop part with OFE part. In contrast, single step brazing with 50Au/50Cu alloy in hydrogen furnace produced satisfactory quality joint between OFE Cu and Glidcop.

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