COPPER-TO-SILICON-CARBIDE JOINTS DEVELOPMENT FOR FUTURE CLIC HOM DAMPERS

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Abstract

Ceramic-to-metal joints have been of paramount importance for the nuclear and aeronautic industry since the last century. In this document, two different approaches to the Cu-to-SiC joining are briefly described and discussed. The first approach consists of an intermediate piece of lower Coefficient of Thermal Expansion than copper aiming to reduce the expansion mismatch with the ceramic during the brazing cycle. Soldering is selected as a second attempt, whose lower joining temperature reduces the absolute expansion difference between Cu and SiC. In addition, four SiC metallization processes are proposed and some of them have been also tested and discussed.
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KEY WORDS

Brazing, CLIC, HOM damper, SAS, Cu-SiC joints, Vacuum Furnace
1. INTRODUCTION & STATE OF THE ART

In the frame of Compact Linear Collider’s (CLIC) copper radio frequency structures are used for acceleration at 12 GeV (1) (2). In order to extract safely the High Order Modes (HOM) travelling along with the main electromagnetic field, Silicon Carbide (SiC) is commonly used as an absorber (3). The joint between SiC and copper is currently mechanically done. As a long-term development copper-to-SiC brazed joints need to be further developed. As result of this strategy, several Cu-SiC joining tests have been performed and their results have been carefully analysed.

Previous CLIC tests on direct brazed joints were unsuccessful (4). The expansion mismatch between the copper piece and the SiC piece produced excessive mechanical stress in the interface which led to a brittle fracture on the SiC side. Also, brazing tests performed in the past on large surfaces (200x50mm) for the CLIC test Facility 3 (CTF3) resulted in broken ceramics (5). However, soldering at temperatures around 230°C produced sound joints, but it required a previous metallization of the ceramic part (5).

Along with this, SiC active brazed joints to Molybdenum (Mo) parts have been successfully obtained at CERN during the past year (6). This can be partially explained by a lower difference in the Coefficient of Thermal Expansion (CTE).

In this report the possible process changes proposed to reduce the stress of the joint are explained. Two different approaches have been studied (see Figure 1). The first approach is based on a reduction of the CTE mismatch, obtained through an intermediate piece whose expansion is similar to that of SiC which reduces the stress on the ceramic. The second approach consists of reducing the process temperature (soldering instead of brazing) and therefore reducing the maximum strain. For this second solution, a metallization of the ceramic is imperative and will be studied.

![Figure 1 – Schema of the test definition](image-url)
2. EXPERIMENTS & RESULTS

The description of these joints and the followed joining steps for the different procedures are explained in this section. All pieces have an internal diameter of $\Phi_i=58\text{mm}$ and an external diameter of $\Phi_e=64\text{mm}$.

2.1 BRAZING

The brazing test was performed including an intermediate piece made of Molybdenum (Mo), whose CTE is close to that of the ceramic. It is expected that using Mo will reduce the stress on SiC. For this brazing, a so-called Active Brazing Alloy (ABA) has been used, since non-active brazing alloys do not properly wet the surfaces of the ceramic and the Mo (7).

An active brazing test was done at the Brazing workshop (EN-MME-FW) at CERN. ABA-Cusil® (8) (See Table 2) was the selected brazing alloy. Once at the workshop, the surfaces of the Cu and Mo pieces were prepared with metallurgical discs to obtain the proper surface roughness, with a $R_a$ of 0.8 µm as the value needed for an optimum brazing.

<table>
<thead>
<tr>
<th>Name</th>
<th>Nominal Composition Percent</th>
<th>Liquidus</th>
<th>Solidus</th>
<th>Form</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>°C</td>
<td>°F</td>
<td>°C</td>
</tr>
<tr>
<td>Cusil-ABA®</td>
<td>Ag – 63.0</td>
<td>815</td>
<td>1500</td>
<td>780</td>
</tr>
<tr>
<td></td>
<td>Cu – 35.25</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ti – 1.75</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BrazeTec®-CB10</td>
<td>Ag – 64.8</td>
<td>805</td>
<td>1481</td>
<td>780</td>
</tr>
<tr>
<td></td>
<td>Cu – 25.2</td>
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<td></td>
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</tr>
<tr>
<td></td>
<td>Ti – 10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sn3.5Ag</td>
<td>Sn – 96.5</td>
<td>221</td>
<td>1795</td>
<td>221</td>
</tr>
<tr>
<td></td>
<td>Ag – 3.5</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 1 – Brazing and soldering alloys used in this document (8) (9)

After that, a degreasing of the ceramic followed by a firing at 1000°C for 2 hours was done to eliminate the remaining gasses trapped in the bulk. Also, a degreasing plus pickling of the copper and molybdenum pieces was performed to eliminate all traces of grease and oxides on the brazing surfaces, leaving clean surfaces that will be correctly wetted by the brazing alloy.

Once the pieces were clean and ready for brazing, the assembly was prepared. Two ABA-Cusil foils of thickness 0.05mm were placed on the interfaces between Cu-Mo and Mo-SiC. Then, the assembly was introduced in the furnace and tungsten blocks were put on top of the assembly to assure that a good pressure of about 300g/cm² was obtained.
Once a vacuum of an order of $10^{-6}$ mbar was obtained, the cycle was launched. Brazing of ABA-Cusil takes place at 830°C for 5 minutes. The thermal cycle is shown in Figure 2. The brazing test resulted on a brittle fracture on the SiC side (Figure 3) due to an excess of the internal stress.

Figure 2 – Curve of the vacuum brazing cycle.

Figure 3 – Active brazing test of Cu-SiC with a Mo intermediate piece ($\Phi_i=58\text{mm}/\Phi_e=64\text{mm}$). Crack is on the ceramic part.
2.2 SOLDERING

Soldering was performed at around 220°C in order to reduce the global expansion mismatch and therefore the stress of the joint. On the other hand, at this temperature the use of an active brazing alloy is not possible and the wetting of the ceramic by normal soldering alloys is not enough. Therefore a metallization of the SiC piece was needed in order to improve the wettability of the base material. Four different options were considered for this metallization prior to the soldering test:

- Metallization by an active brazing alloy
- Ti-Cu coating (sputtering)
- Non-Evaporative-Getter (NEG) coating (sputtering)
- Cr-Ti-Cu coating (10)

A wetting test was performed on the second and third metallization methods (section 2.2.2), resulting on an insufficient wetting of the NEG coating. Therefore this metallization was not used for the soldering test. Soldering tests were performed only on the active brazing metallization (section 2.2.1) and the Ti-Cu coating (section 2.2.3). The last option (Cr-Ti-Cu coating) is yet to be tested, since CERN facilities do not have the capability to perform such a deposition and the coating in an external supplier would take more time.

2.2.1 ACTIVE BRAZING METALLIZATION SOLDERING TEST

First of all, an active brazing metallization test was done in the brazing workshop (EN-MME-FW) at CERN. Prior to the soldering test with the final pieces, a rough feasibility test was done on a broken piece from the active brazing test (see section 2.1). The idea of this rough test was to prove that this method was a good option for Cu-SiC joints without damaging any final piece. BrazeTec®-CB10 active brazing paste (9) (see Table 1) was selected due to availability in the workshop. It was applied on the soldering surface of the SiC piece with a small brush, left to dry in atmosphere for the night and introduced in the furnace. A full brazing cycle at 850°C was launched to produce the metallization of the ceramic. The metallization was caused by the melting of the brazing paste and the reaction of the Ti contained in it with the surface of the ceramic allowed the wetting. The result is shown in Figure 4.

After the metallization of the broken SiC piece, it was soldered to a small copper plate. The plate was grinded with polishing discs and cleaned with ultrasounds in an ethanol bath. After that the assembly was soldered. As a result of this rough test a sound joint was obtained in a first instance, but after several stress tests the assembly had a failure on the joint (see Figure 4).

Figure 4 – CB10 metallization (a) and soldering test (b). The failure occurred in the interface.
As a second step a Cu-SiC soldering test on the final pieces was performed. First, the cleaning of SiC and Cu pieces was done with the same procedure as in section 2.1. In addition, the copper piece was annealed at 600°C for 2 hours to reduce its mechanical properties, i.e. the yield strength. Then, the soldering surface of the ceramic was metallized with the ABA CB10 at 850°C, as explained before. In addition, metallized SiC and Cu samples were soldered using a 0.1mm thick foil of the Sn3.5Ag eutectic (see Table 1) as the soldering alloy. Again, the soldering alloy was selected due to workshop availability. The soldering cycle done is shown in Figure 5.

![Figure 5](image-url) - Curve of the vacuum soldering cycle.

The test resulted on an apparently sound joint (see Figure 6) that could not be broken through manual stress. However, mechanical tests should be done to properly study the joint strength.

![Figure 6](image-url) – Active metallization of SiC (a) and SiC-Cu soldered prototype (b). (Φi=58mm/Φe=64mm)
2.2.2 WETTING TESTS OF NEG & TI-CU COATINGS

In order to check the wettability of the sputtered Ti-Cu and NEG coatings, a wettability test was performed on the SiC broken piece from the active brazing test (see section 2.1). The broken cylinder was cut into pieces that were afterwards coated. The result is shown in Figure 7. Ti-Cu coating was properly wetted by the soldering alloy, while NEG coating is not wetted.

![Figure 7 - Wetting test. Pictures are before (a) and after (b) the thermal treatment. Ti-Cu coated sample (left) is wetted, while NEG coated one (right) is not.](image)

2.2.3 TI-CU COATING SOLDERING TEST

First, final geometry pieces were cleaned with the same procedure as in section 2.1. As for the previous soldering test, the copper piece was annealed at 600°C for 2 hours prior to the soldering process. The ceramic was first Ti-Cu coated with 1 µm of sputtered Ti followed by a 5 µm film of Cu which was sputtered on top of the Ti layer. Then, soldering was done at the brazing workshop with a 0.1mm thick foil of Sn3.5Ag as the soldering alloy following the same cycle that was done for the CB10 coated sample in section 2.2.1. It resulted in a sound joint that resisted to manual stresses. Figure 8 shows the assembly after the vacuum soldering cycle.

![Figure 8 - SiC and Cu samples before joining (a) and SiC-Cu soldered prototype (b). (Φi=58mm/Φe=64mm)](image)
3. DISCUSSION OF RESULTS

PRE-EVALUATION OF JOINT FEASIBILITY

Active brazing of SiC-to-Copper with a Mo intermediate from section 2.1 results in ceramic failure due to the mechanical stress, which is explained by an increase in the total strain difference between the ceramic and the metal due to the higher temperature with respect to soldering.

This result shows that even with a reduced CTE, the molybdenum interlayer transmitted too high stress to SiC. This can be explained by the high mechanical properties of molybdenum, even at the brazing temperatures. The rigidity of the molybdenum intermediate piece reduces strain accommodation through plastic deformation, which produces high stress transmission to the ceramic.

Thus, if only CTE is considered, properties of the materials that have a paramount importance on joint quality could be unconsidered in the pre-evaluation of the brazed and soldered joints. Amongst other factors we find the so-known Thermo-mechanical Compatibility Factor (TCF), defined by Norman C. Anderson (11) in 1996 as:

\[
TCF \equiv \frac{\epsilon_t}{\epsilon_y} \equiv \frac{1}{\epsilon} \sigma_y
\]

Equation 1 – TFC for \( \epsilon_t < \epsilon_y \) (11)

where \( \sigma_y \) is the yield strength of the metal, \( \epsilon_y \) is the yield strain of the metal and \( \epsilon_t \) is the total strain difference between the metal and the ceramic at brazing temperature.

This factor takes into account not only the CTE but also the mechanical properties of the metal that is joined to the ceramic, giving a qualitative idea of the compatibility of the assembly. Values in Table 2 were calculated by Anderson for Alumina-to-metal joints:

<table>
<thead>
<tr>
<th>Metal</th>
<th>TCF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Niobium</td>
<td>88</td>
</tr>
<tr>
<td>Platinum</td>
<td>33</td>
</tr>
<tr>
<td>Tantalum</td>
<td>28</td>
</tr>
<tr>
<td>Copper</td>
<td>20</td>
</tr>
<tr>
<td>Titanium</td>
<td>8.8</td>
</tr>
<tr>
<td>Titanium</td>
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<tr>
<td>Kovar</td>
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<tr>
<td>Nickel</td>
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<tr>
<td>Fe-42Ni</td>
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<td>Monel</td>
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<td>Invar</td>
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<td>Molybdenum</td>
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<tr>
<td>Stainless Steel 304</td>
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<tr>
<td>Inconel 600</td>
<td>2.1</td>
</tr>
<tr>
<td>Tungsten</td>
<td>2.0</td>
</tr>
</tbody>
</table>

Table 2 – TCF calculated values for Al\(_2\)O\(_3\) brazing at 780°C (11)
It can be seen that in the case of alumina-to-metal joints, Molybdenum has lower compatibility factor than copper although the CTE difference is much higher. The TCF is hence a promising parameter showing potential to be used on a qualitative study of compatibility of dissimilar materials for assemblies in terms of brazability. Only using the CTE mismatch could lead to underestimation of the internal stresses in the brazed joints.

METALLIZATION

The metallization tests of the SiC exposed in section 2.2 showed a bad wetting of the NEG coating and a proper wetting of the Ti-Cu coating. This difference can be partially explained by the absorption of oxygen on the soldering surface of the NEG piece. NEG coatings are thin film coatings used for Ultra High Vacuum (UHV) applications. Once activated, NEG tends to pump any residual gas in the chamber. Thus, in our particular case, the non-wetting behaviour of the NEG coated SiC piece might have been produced by an activation of the coating at the soldering temperature. This activation could have attracted the residual oxygen in the furnace saturating the coating with this element, which highly reduces wettability. Therefore, only the Ti-Cu coating was tested with final pieces.

SOLDERING

The sound joints obtained in section 2.2 through a vacuum soldering process prove that this method is promising when joining SiC-to-Copper pieces. However, further tests should be performed in order to obtain the real value of the mechanical strength of the joints. Also, different parameters should be analysed in future tests, such as the soldering alloy thickness, the soldering alloy type and the active metallization alloy type used, and their influence in joint strength. Also, metallurgical analysis of the joints should be foreseen in order to completely characterize the joints.

The unknown properties of the OFE-copper used in the first rough soldering test from section 2.2.1, as well as its rough preparation might be the reason of the failure of this test. To reduce the stress in the joint as much as possible, a thermal annealing of the copper pieces was added to the final soldering procedure in order to reduce the yield strength of the material, hence enhancing the absorption of stress due to plastic deformation.

JOINING CONTRAINTS IN APPLICATIONS

Even though the brazing test was unsuccessful, other configurations might be tempted since the reduced temperature of the soldering procedures could be a design constraint for a vacuum system for CLIC, the LHC and other accelerators. Temperatures used for baking and NEG activation thresholds are cyclical on the accelerator run and must be lower than the soldering temperature to avoid re-melting of the soldering alloy. Brazing temperatures are however well above those limits.

Fatigue due to heating cycles and mechanical stress transmitted to the assembly must also be considered for any joint of dissimilar materials.
4. **CONCLUSIONS**

Active brazing of SiC-to-Copper with a Mo intermediate piece is not a valid solution for the presented configuration. High yield stress and elastic strain can be detrimental for the resistance of a joint of dissimilar materials, mainly on a metal-ceramic combination. Thus, ductility and loss of mechanical properties with temperature are preferably compared to rigidity and high stability of mechanical properties at high temperatures in terms of internal stresses.

TCF should be taken into account as a qualitative measure of the compatibility of couples of dissimilar materials and not only the CTE mismatch of the base materials.

Active brazing metallization and Ti-Cu coating of SiC improved wettability of the base metal. On the other hand, NEG is not wettable at 220 degrees in the conditions of the high-vacuum furnace used.

Metallization of SiC combined with a soldering process have been tested and produce sound joints. Results show that it is a valid option for joining copper-to-SiC for the tested geometry, dimensions and tolerances. Yet, mechanical tests and metallurgic analysis of the joints need to be performed to fully characterize this procedure.

Reduced temperature of the soldering procedures could be a design constraint for systems in the LHC or other accelerators due to higher temperatures used in baking and NEG activation. Also thermal fatigue of the joints due to these cyclic heat treatments must be considered.

5. **FUTURE STEPS**

- Mechanical tests and metallurgical examinations of different pieces to check best parameters.
- Future campaigns of SiC-Cu samples with different soldering alloys and at different temperatures to increase the applications of this kind of joint.
- Try other active brazing metallization and soldering filler metals for the joint.
- Find better, more promising material combinations for active brazing of Cu-to-SiC joints.
- Further study TCF influence and look for new compatibility parameters.
6. REFERENCES

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