

A STUDY OF THE SURFACE QUALITY OF HIGH PURITY COPPER AFTER HEAT TREATMENT

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Abstract

The manufacturing flow of accelerating structures for the compact linear collider, based on diamond-machined high purity copper components, include several thermal cycles (diffusion bonding, brazing of cooling circuits, baking in vacuum, etc.). The high temperature cycles may be carried out following different schedules and environments (vacuum, reducing hydrogen atmosphere, argon, etc.) and develop peculiar surface topographies which have been the object of extended observations. This study presents and discusses the results of scanning electron microscopy (SEM) and optical microscopy investigations.

INTRODUCTION

The current design of the future compact linear collider (CLIC) features significant physical and technological challenges [1]. The CLIC Accelerating Structures (AS) currently designed in high purity copper, need to withstand extremely high surface magnetic and electric fields in order to achieve a maximum accelerating gradient of 100 MV/m. Several different design and manufacturing strategies for these AS have been considered which must all proof their performance in prototype high-power tests.

The individual designs foresee different joining methods in order to build a complete AS, including all the necessary components (e.g. vacuum manifolds, cooling circuits, radio frequency (RF) waveguides, damping material, etc.). These joining methods involve very extreme heat cycles that ultimately affect the materials and their respective surfaces.

This paper presents the outcome of an extensive parameter study initiated and conducted by the CLIC RF structures production team. The influence of the main furnace parameters on the surface topography and microstructure of high purity copper is investigated:

- 1) Treatment temperature: 820°C and 1040°C.
- 2) Protective atmosphere: vacuum, argon, hydrogen at 20 hPa and hydrogen at 1000 hPa.

The treatments were conducted in cooperation with selected industrial companies as well as one collaborating institute. Not all suppliers are equipped to fully satisfy the experimental programme. However, some duplicated treatments enabled the comparison of effects of the different

furnaces of the suppliers (operating nominally under the same conditions).

EXPERIMENTAL PROCEDURE

Material and Surface Finishing

All the experiments were conducted on samples of high conductivity Oxygen-Free Electronic copper OFE min 99.99% (Cu-OFE REF. UNS C10100 Grade 1) with a grain size of approximately 100 μm complying with ASTM F68 and CERN specifications [2]. The samples are machined to a diameter of 12 mm with one side finished by diamond fly cutting resulting in roughness values as small as $R_a = 20$ nm. This specific surface finishing is representative of the CLIC-AS surface state. Therefore, the effects of surface modification arising from heat treatments can be studied.

Prior to the treatments in the furnace the samples were cleaned according to three different cleaning procedures:

- 1) Solvent cleaning [3].
- 2) SLAC etching [4] (hereafter referred to as “etching”).
- 3) Passivation with chromic acid [3].

Temperatures

Two fundamentally different joining processes are currently considered for producing the CLIC-AS:

- 1) Brazing for 2 h at a temperature of 820°C.
- 2) Diffusion bonding for 1.5 h at a temperature of 1040°C.

Both processes aim at leak tight and sound joints of all the components involved. Nevertheless, the resulting modifications to the diamond finished surfaces of the components might degrade the high power performance of the final AS.

Atmospheres

The samples are protected from oxidation during the treatments using four alternative atmospheres: vacuum (10^{-6} mbar), argon at 1000 hPa, hydrogen at 20 hPa (absolute), and hydrogen at 1000 hPa. These four atmospheres have been selected in order to cover all joining procedures envisaged for the production process of the CLIC-AS.

Test Matrix

The combination of the two temperatures and the four atmospheres leads to a test matrix of eight possible heat treatment combinations. Six suppliers provided samples treated according to their available equipment (Table 1). Note that for each test performed three pairs of samples cleaned following the three different cleaning procedures were in the furnace at the same time. Non-treated reference samples were kept for comparison.

Table 1: Complete Test Matrix of the Study. “A” stands for treatment at 820°C and “B” for treatment at 1040°C.

	vacuum	argon	hydrogen 20 hPa	hydrogen 1000 hPa
Supplier 1	A+B	A+B	A+B	-
Supplier 2	A+B	-	-	-
Supplier 3	-	-	-	B
Supplier 4	-	A	B	-
Supplier 5	-	-	-	A+B
Supplier 6	-	-	-	A+B

Characterization and Procedure

Samples were examined using Scanning Electron Microscopy (SEM) and Optical Microscopy (OM). Special attention was paid to the three microstructural features developing during exposure to elevated temperature on the sample surface:

- 1) Grain size as a result of grain growth.
- 2) Grooving of the grain boundary (GB) as a result of higher GB diffusivity.
- 3) Faceting in the regions of GB as a result of “selective evaporation” of atoms in non-energetically favourable crystal position.

For the characterization of the final grain size mentioned in point 1) OM was used. Thanks to the diamond fly cut surface finishing and the protective atmospheres during the thermal cycles no metallographic preparation was necessary to reveal the grain structure. In some cases the final grain size was estimated based on a few grains only, due to the limited sample area available and large grain size. All the additional characterizations considered in the current study were conducted with SEM. Peculiar attention was paid to GB triple points. Five triple points were found to give a representative picture of the state of GBs on the observed samples.

EXPERIMENTAL RESULTS AND DISCUSSION

Influence of Temperature

As expected, all the samples submitted to the higher temperature (1040°C) exhibit larger grain size than samples submitted to lower temperature (820°C) treatments,

starting from the same initial grain size of approximately 100 μm . Two examples of the resulting microstructure of samples submitted to both temperatures can be seen in Fig. 1. The grain size for the sample treated at 820°C

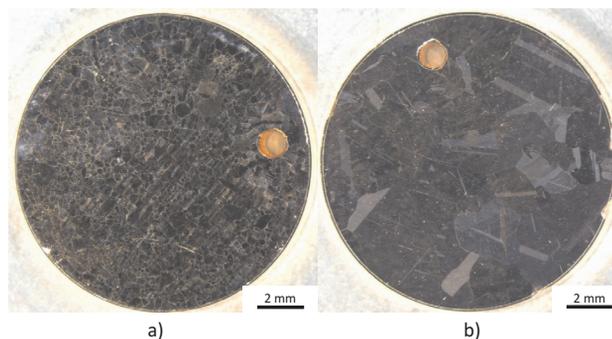


Figure 1: Comparison of OM pictures of etched samples treated under hydrogen at 20 hPa at a) 820°C and b) 1040°C.

in Fig. 1a (approximately $400 \mu\text{m} \pm 100 \mu\text{m}$) is significantly finer as the one treated 1040°C in Fig. 1b (approximately $2000 \mu\text{m} \pm 250 \mu\text{m}$).

Thanks to the diamond finishing of the samples the initial surface is extremely smooth and GB cannot be distinguished. Figure 2 shows that, in terms of faceting and GB

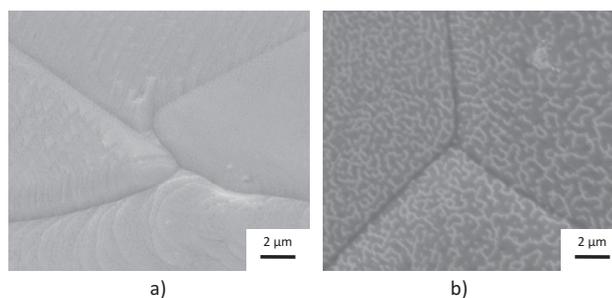


Figure 2: Comparison of SEM pictures of representative GB triple points of solvent cleaned samples treated under argon at a) 820°C and b) 1040°C. Note: stains in b) are artifacts of the treatment furnace.

grooving under argon atmosphere, all samples submitted to 820°C display similar signs of significantly higher activity than the samples submitted to 1040°C. In Fig. 2a the GB grooving and faceting is very pronounced compared to Fig. 2b.

Faceting occurs mainly on samples submitted to an 820°C cycle and argon atmosphere. The significance of the argon atmosphere will be discussed in the following section.

Influence of Atmosphere

Different kinds of atmospheres, excluding high pressure inert atmospheres such as 100 MPa of argon applied during

Hot Isostatic Pressing (HIP) (ongoing study), do not influence bulk microstructural processes such as grain growth or recrystallization behaviour. Nevertheless, they are known to influence the thermal behaviour of the first atomic layers [5] and hence differences in surface topography can be expected. Fig. 3 shows the result of a treatment at 820°C under the four considered atmospheres. Under argon in

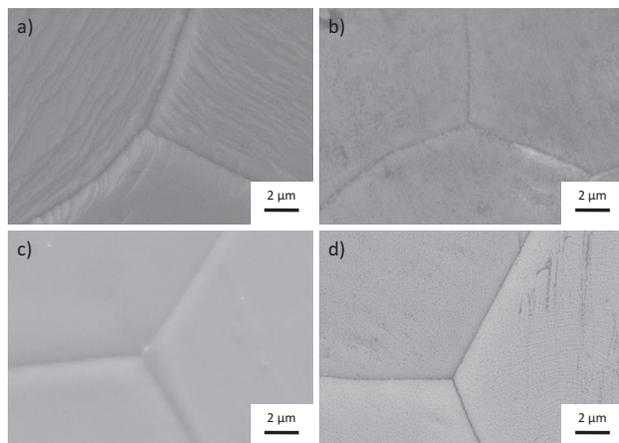


Figure 3: Comparison of SEM pictures of representative GB triple points of solvent cleaned samples treated at 820°C under a) argon, b) vacuum, c) hydrogen at 20 hPa and d) hydrogen at 1000 hPa. Note: stains are artifacts of the treatment furnace.

Fig. 3a the GB is more grooved and faceting is more pronounced, whereas in the other atmospheres GB grooving is medium to moderate and faceting low. At 1040°C no difference in faceting (absent to low for all the samples) and only marginal difference in GB grooving is observed between the four considered atmospheres. The reason for enhanced GB activity at 820°C in argon still has to be investigated.

Influence of the Cleaning Procedure

Figures 4b-d show that the cleaning procedure does not significantly influence the activity at GB's. GB grooving (moderate) and faceting (not detectable) are similar throughout all the samples. Nevertheless, passivation with chromic acid [3] systematically resulted in numerous surface pits for all of the four considered atmospheres. Three of these pits are shown in Fig. 4a indicated by the arrows.

SUMMARY AND CONCLUSION

An extensive testing program was conducted in order to address different issues related to pure copper submitted to high temperature joining processes. As expected, the influence of the temperature is found to be clearly evident when comparing grain sizes after treatment. Samples submitted to 820°C show in average approximately 400 μm grain size whereas those submitted to 1040°C feature approximately 1800 μm. The different atmospheres show no influence

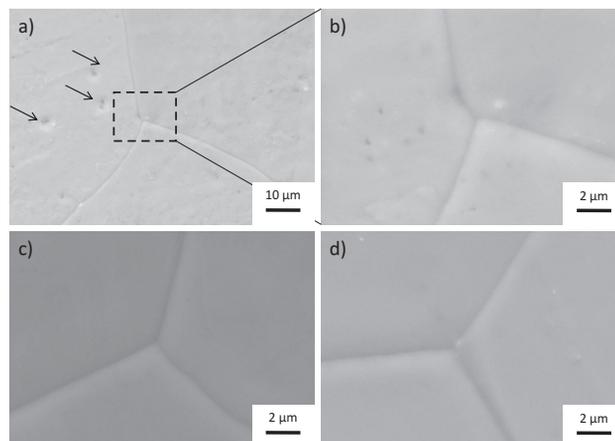


Figure 4: Comparison of SEM representative triple points pictures of samples treated at 820°C under hydrogen at 20 hPa a) overview of the surface of a passivated sample showing pits, b) passivated (detail of the GB triple point in a), c) etched, and d) solvent cleaned.

on any of the features studied with one exception: the low temperature treatment in argon showed enhanced GB activity in form of faceting. This effect is smaller at the high temperature treatment where only very small difference to the other atmospheres was detected. Cleaning of the samples was performed following three different routes: solvent cleaning, etching and passivation; yielding all three the same results within the frame of observed phenomena and samples. Nevertheless, passivation was found to introduce surface pits, distributed almost homogeneously.

The wide variety of different treatment combinations resulted ultimately in very comparable surface states with some exceptions. Additional tests are required in order to assess the relevance of the effects arising from the mentioned heat cycles for the RF-Performance of the final AS, such as: DC-Spark testing and high power RF prototype structure testing.

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