THERMAL FATIGUE OF POLYCRYSTALLINE COPPER IN CLIC ACCELERATING STRUCTURES: SURFACE ROUGHENING AND HARDENING AS A FUNCTION OF GRAIN ORIENTATION

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Abstract

The accelerating structures of the Compact Linear Collider (CLIC) will be submitted to 2 x 10^{10} thermomechanical fatigue cycles, arising from radio frequency induced eddy currents, causing local superficial cyclic heating. In order to assess the effects of superficial fatigue, high temperature annealed Oxygen-Free-Electronic copper samples were thermally cycled by pulsed laser irradiation. Post mortem Electron Backscattered Diffraction measurements, roughness characterisation and micro hardness observations have been performed. The results confirm previous findings: surface roughening depends on the orientation of surface grains. It is clearly observed that, through thermal cycling, the increase of hardness for a crystallographic direction is related to the amount of surface roughening induced by fatigue. Surface grains, oriented [100] with respect to the surface, exhibiting very low surface roughening, show limited hardening whereas grains oriented in [1 1 0], exhibiting severe surface roughening, show the most hardening. Consistently, surface roughening and hardening measured on [1 1 1] direction lie between the values measured for the other directions mentioned.

INTRODUCTION

The need for high accelerating gradients imposes considerable constraints on the materials of the Accelerating Structures (AS) of the future Compact Linear Collider (CLIC) under study at CERN. The design accelerating fields (100 MV/m) will be the highest ever achieved in an operating accelerator [1]. Prototype structure tests have shown that such gradients are feasible [2, 3]. Surfaces in CLIC AS are exposed to high pulsed Radio Frequency (RF) fields. For a structure equipped with waveguide damping the corresponding temperature rise due to surface magnetic fields is 47 K [2]. The resulting cyclic thermal stresses may cause surface roughening and degradation by fatigue after the 2×10^{10} cycles foreseen during 20 years of operation. The negative influence of increased surface roughness on RF-Performance and electrical breakdown resistance must be considered as a limiting factor. As Oxygen-Free-Electronic (OFE) copper is traditionally the standard material for RF applications, it is considered in the present study as the basic structural material for the AS.

Copper features highly anisotropic elastic properties [4]. Surface degradation through thermal and mechanical cycling is observed to depend strongly on local grain orientation [5, 6]. Early stages of fatigue damage of surfaces exposed to conditions comparable to the CLIC–AS can be simulated by RF fatigue [7] or laser fatigue [8]. These techniques help to understand the basic mechanisms ongoing in the material during thermal cycling and to allow predictions for material fatigue performance.

EXPERIMENTAL

All samples in this study are made of high conductivity OFE copper min 99.99 % (Cu–OFE REF. UNS C10100 Grade 1) annealed under vacuum at 1000 °C for 2 h. This is close to the baseline preparation precedure used for prototype CLIC AS [9]. The resulting temper is called "dead–soft" annealed. Before laser irradiation the sample surface was electropolished in which a layer between 80 μ m and 100 μ m thickness was removed. The resulting surface roughness Ra was between 1 nm and 4 nm.

The laser fatigue device uses a pulsed excimer–laser $(\lambda = 248 \text{ nm})$. The experimental setup is described in detail in [8]. The irradiated area is a rectangle of 1 mm x 0.6 mm. The absorption of ultraviolet light is known to be a thermal process in metals (excitation of electrons and phonons [10]) and therefore independent of the crystallographic orientation of the grains. The isotropic thermal expansion [5, 10] due to irradiation is constrained by the surrounding material in the lateral directions which results in a equi–biaxial strain load. A repetition frequency of 200 Hz, a pulse length of 40 ns and an energy density 0.3 J/cm² (which in-



Figure 1: SEM micrograph of irradiated and non irradiated area showing micro hardness indents in large [1 1 0] grain and a [1 0 0] twin. Grain orientation and boundaries are highlightened as identified by EBSD.



Figure 2: SEM micrographs with same magnification of thermally cycled areas within grains of the directions a) [1 0 0], b) [1 1 1] and c) [1 1 0].

duces a calculated temperature rise of $\Delta T = 280$ K [8]) ensure that in between each cycle the irradiated area can cool down completely to room temperature. The thermally–induced cyclic maximum compressive strain load ϵ_{th-max} is 7 x 10⁻³. Each examination site on the sample surface was irradiated repetitively 5 x 10⁴ times.

The developing roughness was characterised with the help of a 3D VeecoNT3300 in Vertical Scanning Interferometry (VSI). The roughness parameter Rz was chosen because of its high sensitivity to single local features and the surface index SI - 1 value was chosen because of its independence on feature orientation (perfect smooth surface SI - 1 = 0 [5]). Micro hardness measurements were performed with a Vickers diamond pyramid applying a load of 1 g. Grain boundaries and orientations are identified by Orientation Imaging Scanning Electron Microscopy (OIM-SEM). Fig. 1 shows a SEM micrograph of irradiated (left) and non irradiated (right) area. Hardness indents are visible in the two grains with different orientation within the fatigued area. In order to quantify the hardening and roughening due to cycling, hardness and roughness measurements are performed also in the non irradiated area (reference). More then 100 irradiation areas on 7 individual samples were examined in the present study.

🔲 Rz in μm Si-1 1.0E+00 2.50 2.25 1.0E-01 2.00 1.75 1.0E-02 1.50 1.25 1 0F-03 1 00 0.75 0 50 1.0E-04 0.25 Ι Ι 1.0E-05 0.00 unfatigued [100][111][110](ref.) fatigued fatigued fatigued

Figure 3: Roughness increase after 5 x 10^4 thermal cycles for the three main crystallographic directions [1 0 0], [1 1 0] and [1 1 1].

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RESULTS AND DISCUSSION

It can be observed from Fig. 2 that grains oriented in [1 0 0], [1 1 1] and [1 1 0] direction exhibit extremely different surface evolution during cycling. Fig. 3 shows the results of the roughness measurements performed in the mentioned areas. The [1 0 0] grain in Fig. 2a remains very smooth after cycling. Rz and SI - 1 are comparable to the reference values. During the cyclic irradiation the [1 1 1] grain in Fig. 2b develops regular protrusions spaced by less than 1 μ m. Both roughness parameters Rz and SI - 1 increase significantly. The [1 1 0] grain in Fig. 2c develops even larger regular protrusions with higher spacing (4 μ m – 5 μ m) compared to the [1 1 1] grain. The roughness values are the highest observed among the three main directions. Two reasons for this orientation dependent roughening behaviour are discussed in [5]:

(1) The elastic anisotropy of copper results in different cyclic shear stresses when cycled with same maximum strain: the $[1\ 1\ 0]$ grains experience higher shear stress than the $[1\ 1\ 1]$ ones followed by the $[1\ 0\ 0]$ ones.

(2) The distribution of the Schmid factors (defined as the ratio of resolved shear stress on specific slip system to applied external stress) for an equi–biaxial strain load of the considered orientations differs significantly: [1 1 0] grains



Figure 4: Hardness increase after 5×10^4 thermal cycles for the three main crystallographic directions [1 0 0], [1 1 0] and [1 1 1].



Figure 5: Roughness vs. hardness before and after 5 x 10^4 thermal cycles for the three main crystallographic directions [1 0 0], [1 1 0] and [1 1 1].

possess 4 active slip systems whereas [1 1 1] grains 6 and [1 0 0] grains 8. The total strain is therefore distributed over a higher number of slip systems in the latter orientations, whereby the individual strain load per slip system is lower.

Both explanations suggest not only a higher irreversible dislocation motion in the subsurface area of the [1 1 0] grains as compared to the [1 0 0] grains. Qualitatively a higher shear stress or a higher strain load on an individual slip system also enhances the dislocation emittance of Frank-Read sources resulting in a higher dislocation density, hence hardness [11].

Fig. 4 shows a plot of hardness increase after cycling. The hardness of the [1 0 0] orientation was measured independently twice in two similar laser spots in order to confirm the reproducibility of laser irradiation and of the hardness measurements. All orientations show an increase in hardness of different amount after cycling: The hardness increase of the [1 0 0] grain (49 HV \rightarrow 58 HV \cong + 17 %) is the smallest one, followed by the one of the [1 1 1] direction (49 HV \rightarrow 65 HV \cong + 32 %) and the highest one in



Figure 6: Roughness increase vs. hardness increase after 5 x 10^4 thermal cycles for the three main crystallographic directions [1 0 0], [1 1 0] and [1 1 1].

the [1 1 0] orientation (47 HV \rightarrow 68 HV \cong + 44 %).

Fig. 5 plots the roughness versus the hardness of the three main orientations before and after cycling. Starting from an initially equally smooth surface with slightly different hardness, the [1 1 0] direction shows the highest roughening/hardening followed by the [1 1 1] direction and the [1 0 0] direction. Fig. 6 shows the direct comparison of roughness increase versus hardness increase of the three main orientations. The data points indicate a linear dependency of roughening and hardening. This confirms the explanations of different roughening behaviour and the predictions about different hardening characteristics given above.

SUMMARY AND CONCLUSION

OFE copper samples have been "dead-soft" annealed and repetitively irradiated with a pulsed excimer laser inducing a cyclic thermal equi-biaxial strain load. After testing, micro hardness and roughness measurements have been performed in grains of specific orientation identified by EBSD: [1 0 0], [1 1 1] and [1 1 0]. It was found that the [1 1 0] direction shows the most severe roughening followed by the [1 1 1] and the [1 0 0] direction. The same trend was observed for hardness where the [1 1 0] direction shows the most significant hardening, followed by [1 1 1] and the [1 0 0] direction. Additionally, the relation between roughness increase and hardness increase for the three crystallographic directions observed in the present paper was found to be linear. This experimental finding can be qualitatively explained by two consistent approaches:

(1) The elastic anisotropy of copper results in different cyclic shear stresses when cycled with same maximum strain.

(2) The distribution of Schmid factors for equi-biaxial strain loading of the considered orientations differs significantly resulting in different number of active slip systems combined with different Schmid factors.

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