

Initial Investigation Results of Surface Preparation Techniques of OFHC Copper for SRF Applications

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Introduction

As part of efforts to improve the performance of thin film coated accelerating cavities, improvement of the topography of the surface of copper is being pursued. This is known to strongly affect the properties of the deposited superconducting thin film. This study focuses on determining the optimal procedure to enhance the homogeneity and smoothness of the copper surface.

OFHC copper substrates have been processed using mechanical polishing (MP), chemical polishing (CP) and electrochemical polishing (EP) procedures as well as a combination thereof, leading to an optimised surface preparation process. The aim was to replace the presently used nitric acid etching process which results in a rough and non-homogeneous surface, as displayed in Figure 2.

The resulting samples have been analysed using a scanning electron microscope (SEM), a laser profilometer (LP) and a confocal laser scanning microscope (CLSM).

Experimental

OFHC copper samples were subjected to a series of different surface treatments, either in isolation or as a combination.

- Mechanical polishing with different grades of polishing fineness
- Chemical polishing with SUBU5
- Electropolishing with phosphoric acid and n-butanol in a ratio of 3:2

Surface roughness measurements were taken following each process and samples were weighed after each process to determine the thickness reduction and polishing rate

Electrodynamic sweep measurements of the anode-cathode-electrolyte combination were completed using a saturated calomel electrode (SCE) as reference

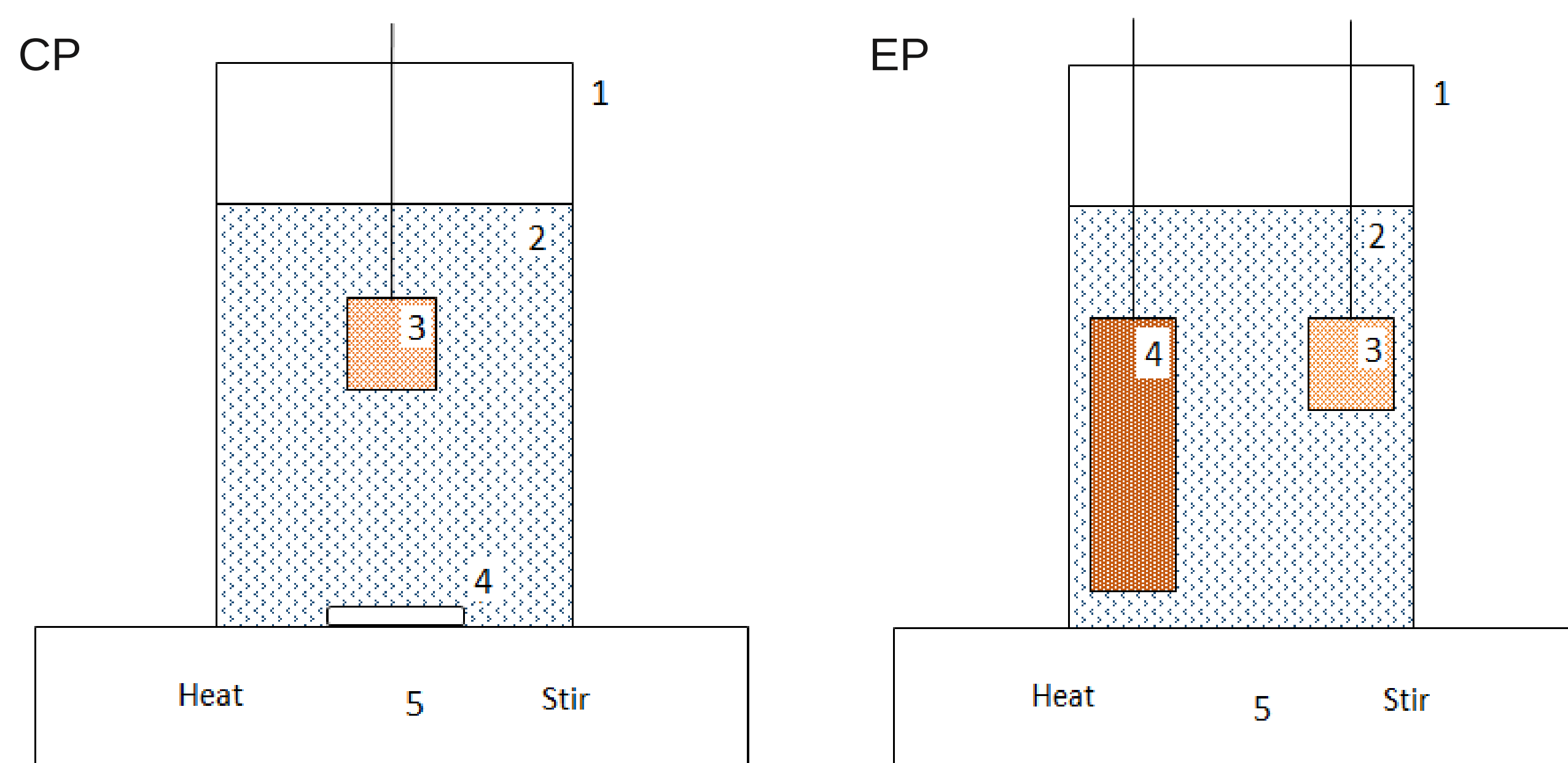


Figure 1: Schematic diagrams of the CP and EP setups. (1) Glass beaker (2) Polishing fluid, (3) Copper sample to be polished (Anode for EP), (4) CP = Magnetic stirrer, EP = Copper Cathode, (5) Heating and Stirring platform

Results and Discussion

Previous samples treated with a nitric acid etch prior to thin film deposition resulted in the surface displayed in Figure 2 (Left) with surface roughness $S_q = 526 \pm 90$ nm (uncoated) and $S_q = 844 \pm 14$ nm (coated). This is not conducive to superior superconducting cavities due to greater nucleation rates on defect sites leading to surface decoration.

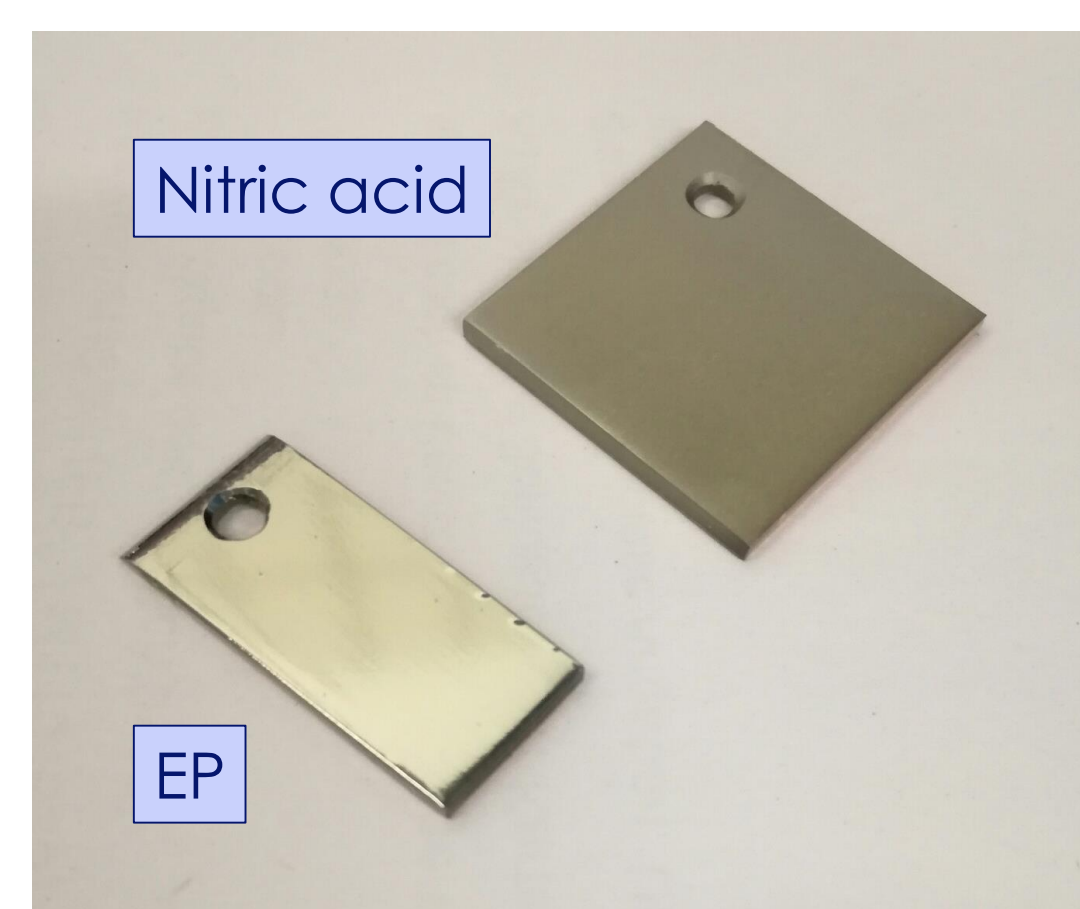
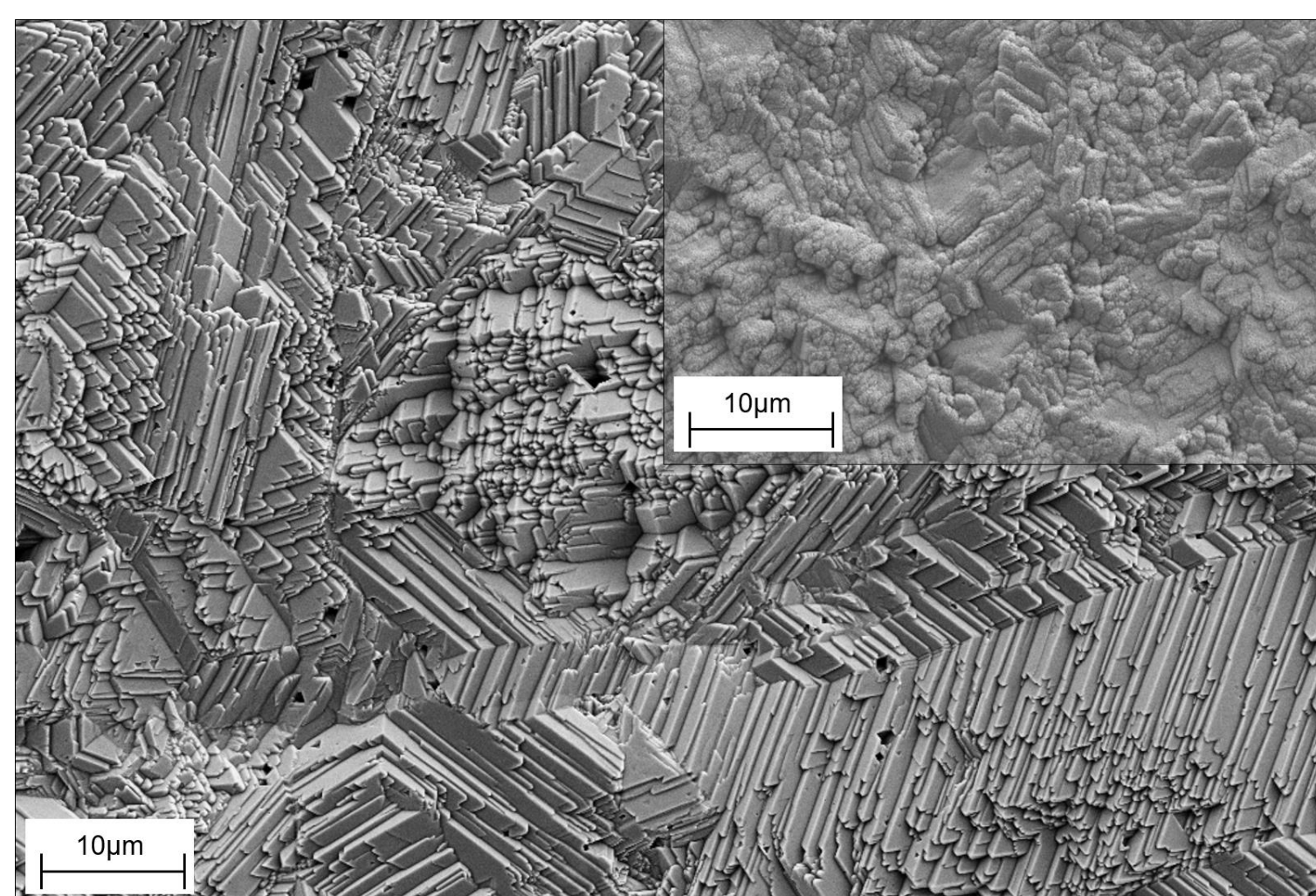


Figure 2: (Left) (Main) Nitric acid etched copper surface. (Inset) NbN film deposited onto nitric acid etched surface showing surface decoration (Right) NbN thin film deposited on EP is more glossy

CP and MP + CP were explored. Results showed significant improvements over nitric acid etched copper:

- Deterioration of copper surface after 60 min CP
- Increased etching at grain boundaries (Figure 3-Right)
- MP+CP displays similar results. CP overrides MP ($R_q \sim 0.11\mu\text{m}$)

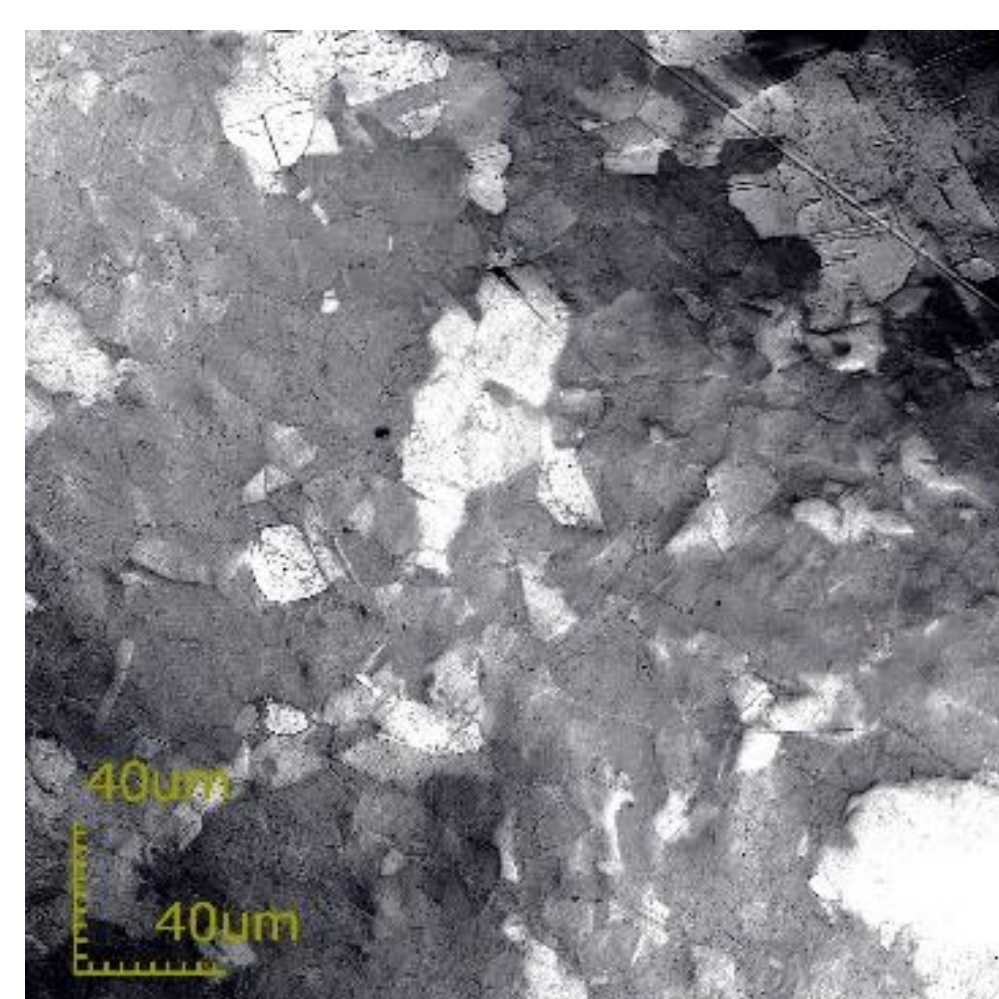
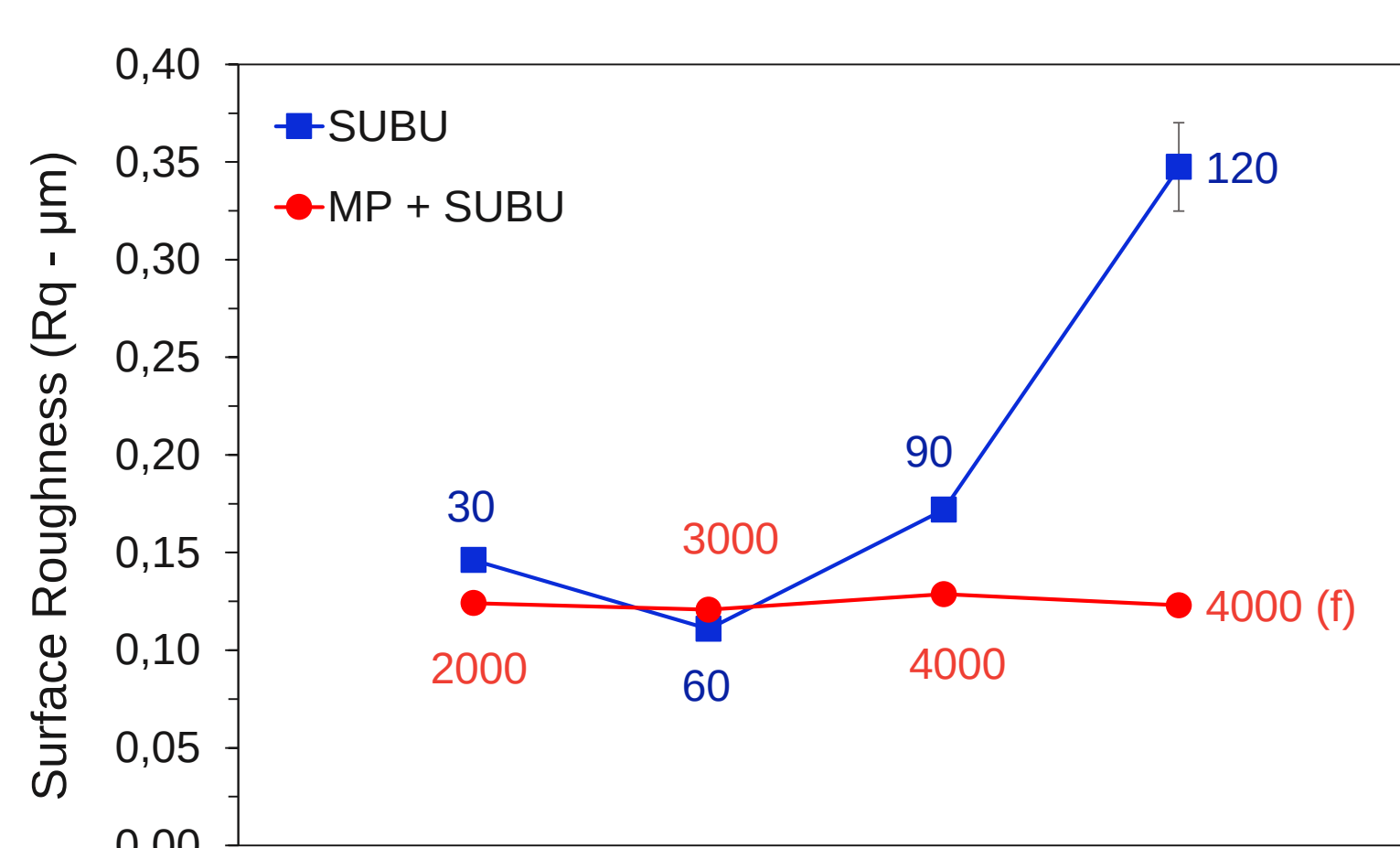


Figure 3: (Left) CP result overview. Blue data indicates results for CP only while red data is for MP + CP. The (f) specifies the use of $1\mu\text{m}$ polishing fluid after 4000 grade MP. (Right) CLSM image of the polished copper surface following 30 mins of CP. Enhanced grain boundary etching is visible.

Results and Discussion cont.

EP and MP+EP were explored also showing significant improvements over nitric acid etched copper as well as CP samples:

- The material removal rate is found to drastically change with electrolyte temperature, increasing from $0.5\mu\text{m/min}$ at 30°C to $2.1\mu\text{m/min}$ at 50°C
- The smoothest surface was obtained using a combination of MP, with 4000 grade polishing paper + $1\mu\text{m}$ diamond suspension fluid, and EP at 40°C for 40 min, with a resultant $S_q = 43 \pm 1$ nm
- The polishing rate (rate of thickness reduction) showed a marked reliance on the current density, increasing steadily with increasing current density.

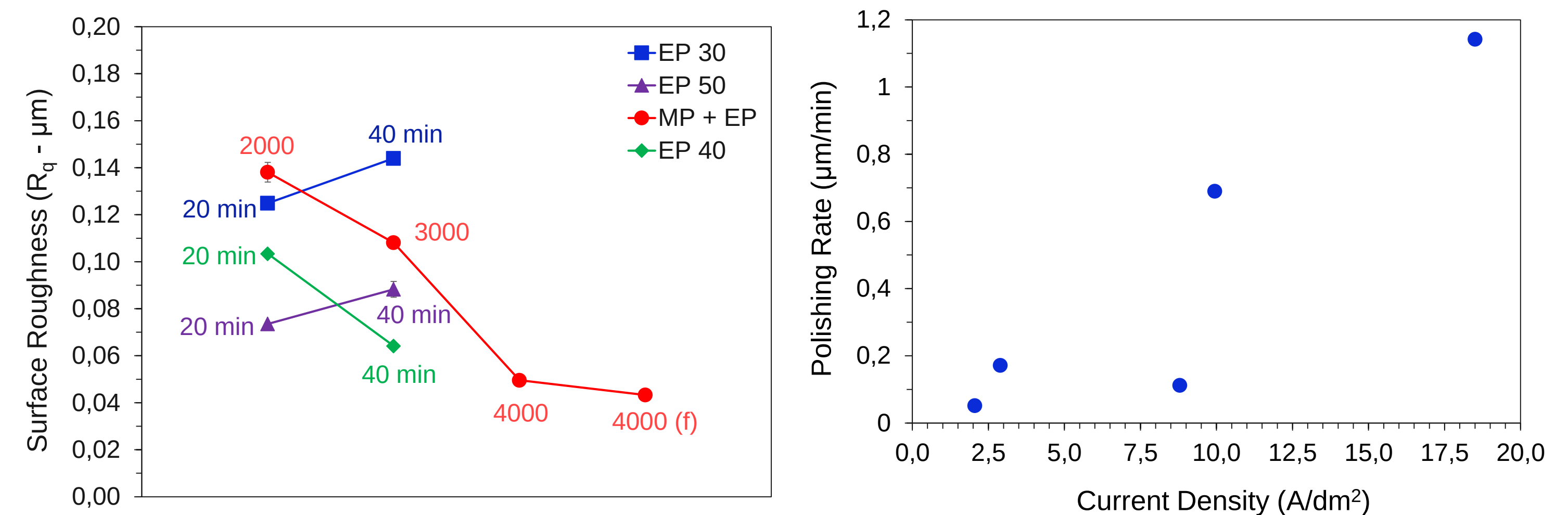


Figure 4: (Left) EP result overview. Blue data details 30°C electrolyte EP results. Green data details 40°C electrolyte EP and purple data details 50°C electrolyte EP. The red data points detail MP + 40°C electrolyte (40 min) EP. The (f) indicates the use of polishing fluid after 4000 grade MP. (Right) Electropolishing experimental results detailing the effect of current density on the polishing rate

Electrodynamic sweep completed to find the optimum polishing setting (Figure 5):

- From this, the optimum current density was determined to be equal to 18.5 A/dm^2 .
- The samples polished at this setting displayed a surface roughness of $S_q = 59 \pm 2$ nm
- Used electrolyte also shows a marked decrease in performance

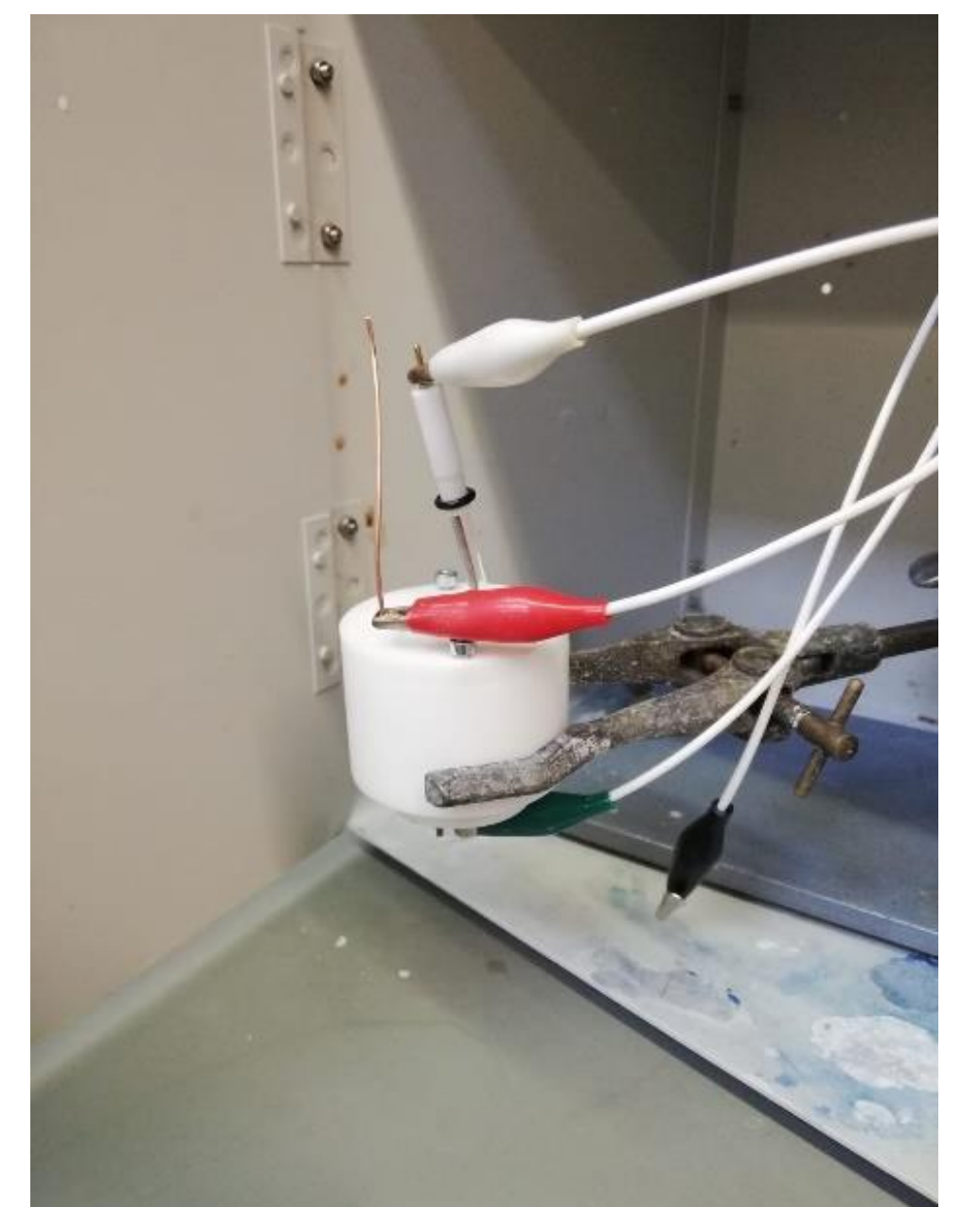
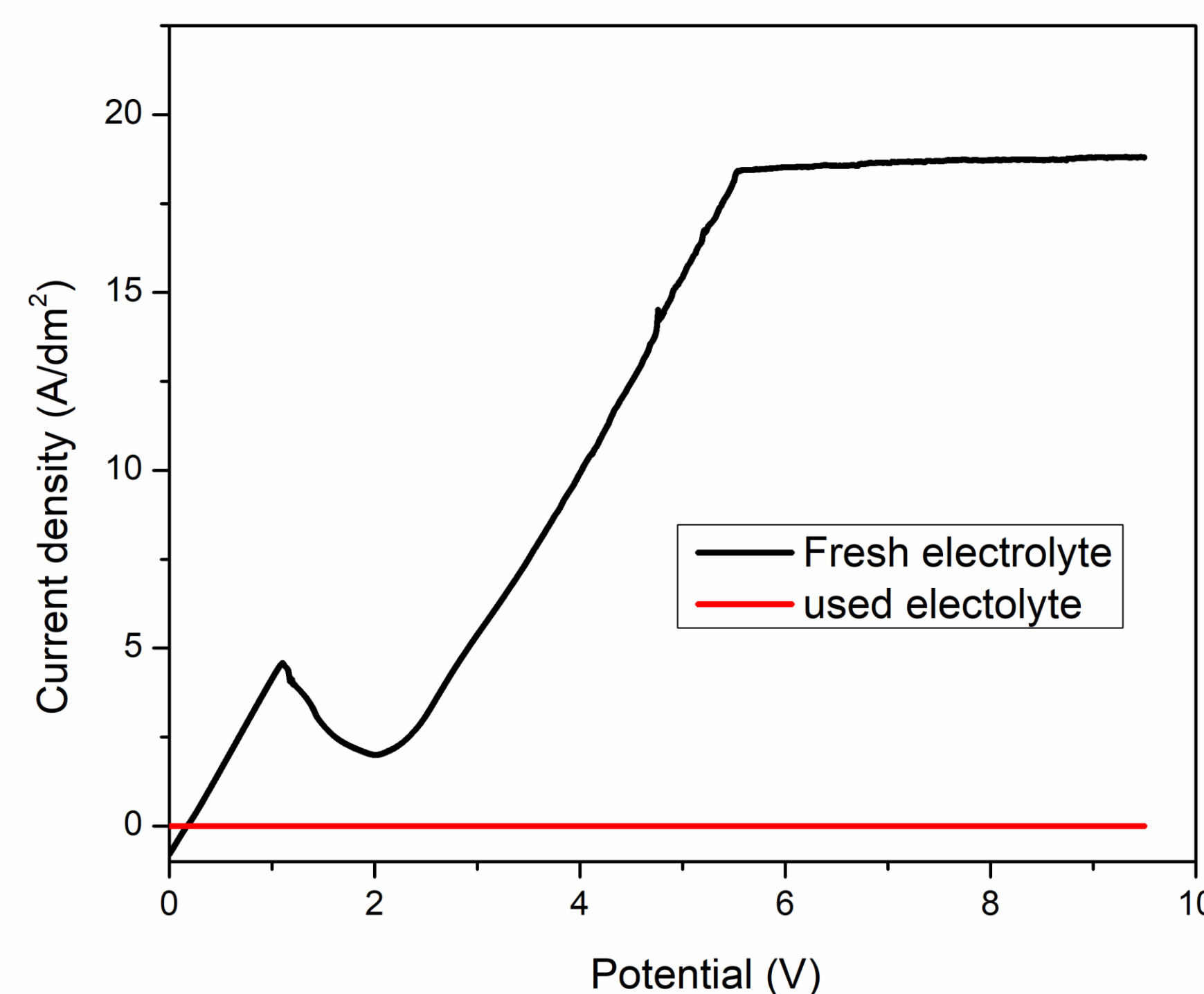


Figure 5: (Left) Polarization curve showing current density vs. voltage of the new and used (3 cycles) phosphoric acid + n-butanol electrolyte. (Right) three electrode cell used to complete electrodynamic sweep measurements

A NbN thin film was deposited onto an EP copper sample. The thin film is once more seen to mimic the surface onto which it was deposited. Great improvement over Figure 2.

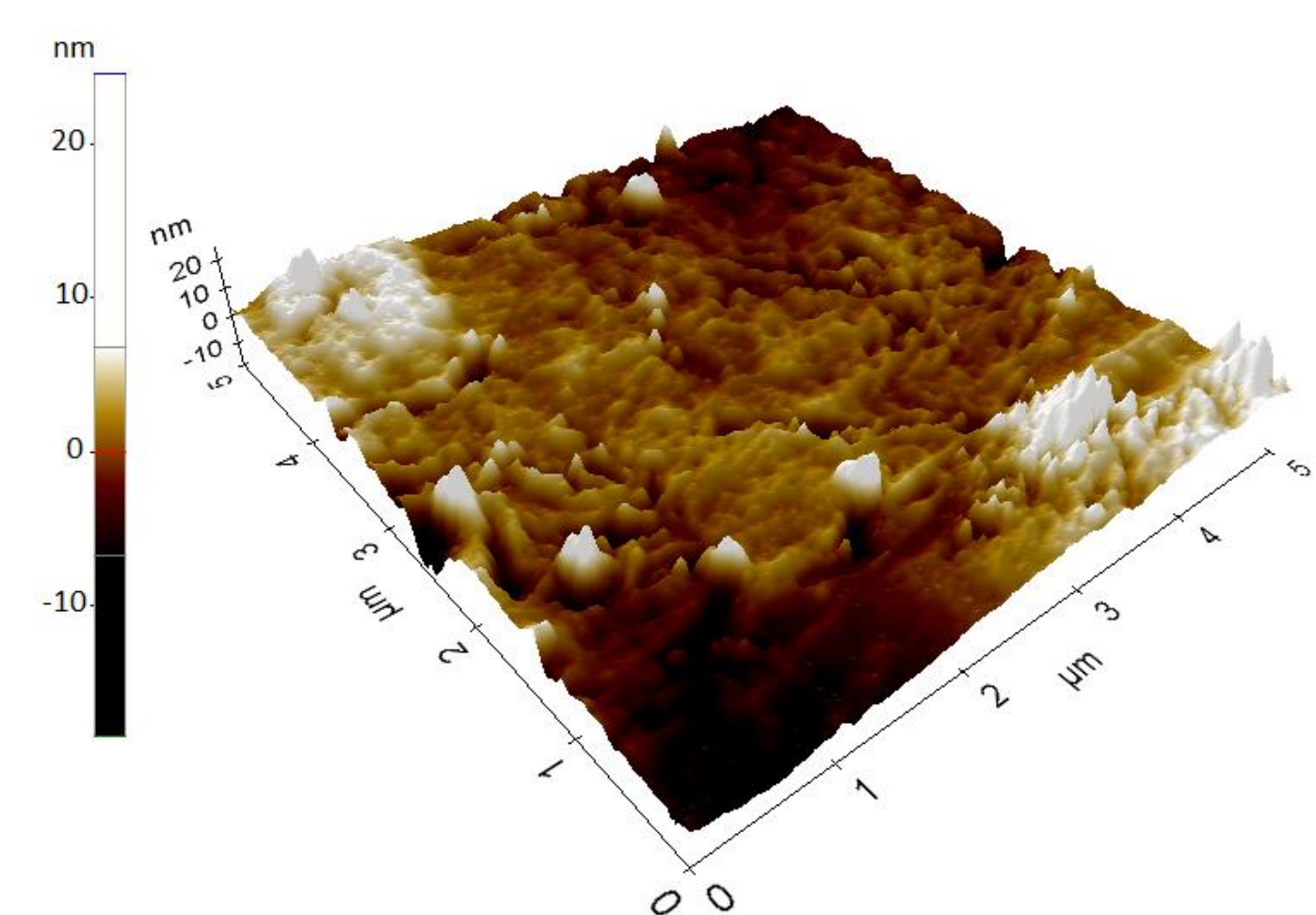
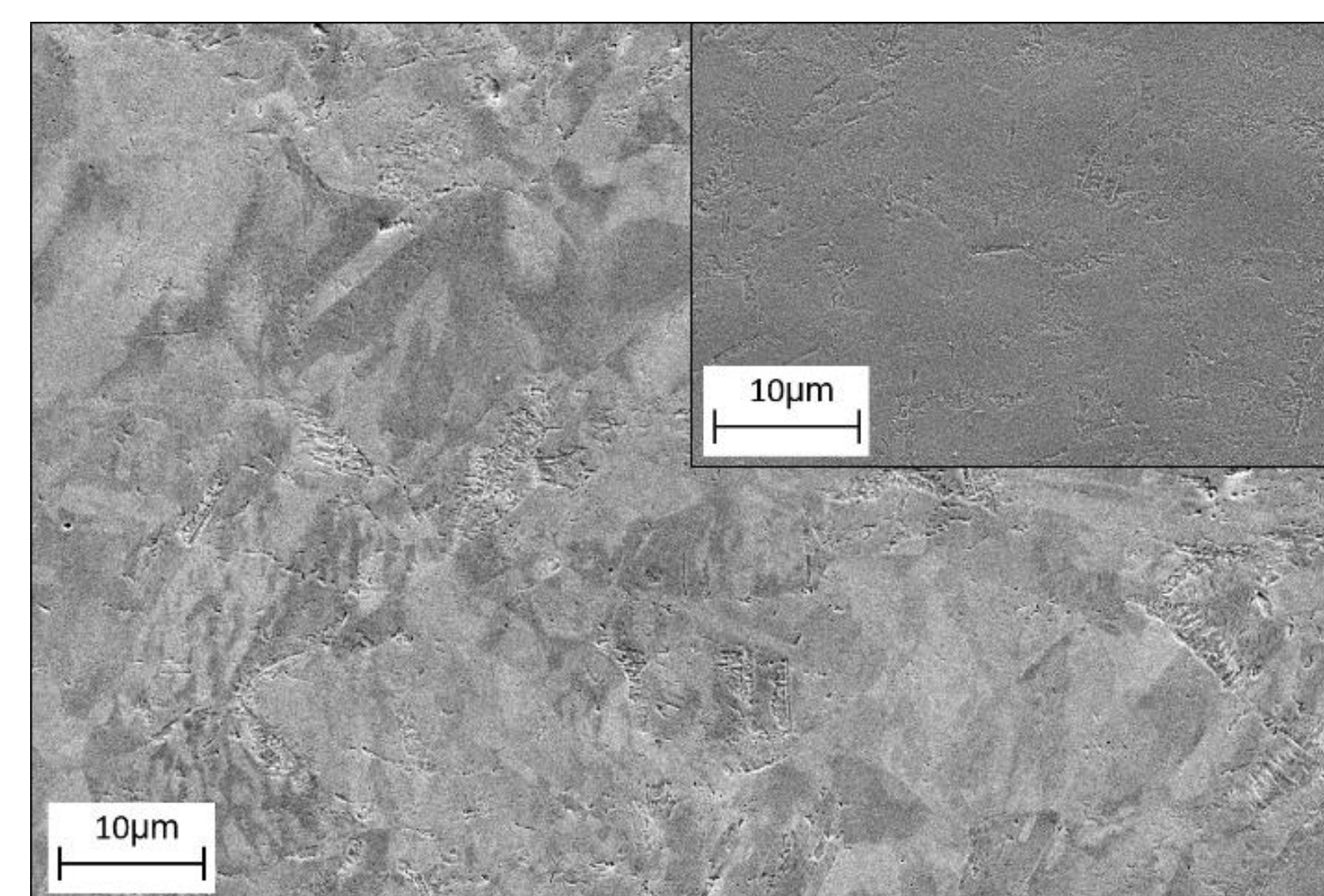


Figure 6: (Left) (Main) EP copper surface $S_q = 59 \pm 3$ nm. (Inset) NbN thin film deposited onto Electro polished surface $S_q = 63 \pm 7$ nm. (Right) AFM image of EP sample indicating more homogeneous surface

Conclusions

- The use of a combination of MP and EP results in the most homogeneous and smooth surface.
- Electrodynamic sweep results in optimum current density during polishing and best surface finish.
- The introduction of CP post EP is found to enhance the presence of grain boundaries which could lead to these being present in the thin film as well. If CP is to be used it should be for a short duration ($<5\text{min}$) to avoid possible pitting of the surface.

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